CREEP STRENGTH EVALUATION OF SERVICED AND REJUVENATED T91 USING THE STRESS RELAXATION METHOD

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Abstract

High precision stress relaxation tests (SRT) at temperatures between 550C and 700C were performed on serviced and reheat treated T91, 9%Cr steel. The service exposure was 116,000 hours at steam temperatures to 550C. Constant displacement rate (CDR) tests were also run at 600C on notched specimens for the two conditions. Specimens heat treated after service were stronger at the lower test temperatures in terms of both tensile strength and creep strength. This difference was reflected in the CDR results, which also suggested a lower fracture resistance in the heat treated condition. Thus service exposure appears to have softened the alloy and enhanced its resistance to fracture, with no evidence of embrittling reactions. Based on the analysis of the SRT tests, projections were made of the times to 1% creep and the times to rupture as well as direct comparisons with minimum creep rate data. When plotted on the basis of a Larson-Miller parameter (C=30), the calculated values compared well with actual long time rupture testing for exposed and re-heat treated specimens, and generally showed higher precision. The longest test time was about eighteen months for the stress rupture data compared with the use of one machine for a few weeks for the SRT data. The latter actually covered a far greater range of creep rates and projected lives. The SRT test is especially consistent at higher parameter values, i.e., higher temperatures and/or lower stresses. This method of accelerated testing is now being applied to a wide range of alloys for fossil power plants for composition and process optimization, design analysis, and life assessment.

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Introduction

Design for Performance is a recently developed methodology for evaluating the creep strength and fracture resistance of high temperature materials. Whereas the traditional approach to creep design involves long time testing, and attempts to incorporate microstructural evolution in the test measurements, the new approach aims to exclude these changes in a short time high precision test. However, the test may be used unambiguously to evaluate the consequences of such changes in service-exposed samples. It may also be used to evaluate the effect of process changes on initial creep strength.

The traditional approach uses a single test to evaluate both creep strength (e.g. minimum creep rate) and fracture resistance (the time to rupture). In practice, because there is generally a good inverse relationship between minimum creep rate and time to rupture (1) they are, in fact, both measures of the same property -- creep strength. The new methodology recognizes that separate tests are necessary to measure creep strength and fracture resistance. For creep strength, a stress vs. creep rate response is determined from a stress relaxation test (SRT), and for fracture resistance a constant displacement rate test (CDR) of a notched tensile specimen is performed at a temperature where the part is most vulnerable to fracture (2).

Although the deformation histories are quite different, for many high temperature materials the two approaches give closely similar results for creep strength. In some cases, however, there are significant differences because very specific microstructural evolution occurs during long time testing. However, such changes may not be consistent with those occurring in service, unless the service application involves a similar thermal/mechanical history.

The *Design for Performance* methodology has been used successfully for metals, ceramics and polymers (3-5). It has been applied to accelerate and optimize materials development, provide a basis for design analysis, and offer a framework for remaining life evaluation of operating components.

The approach is used in this report to compare the creep strength of 9Cr-1Mo-V (T91) steel superheater tubing, which was exposed in service for 116,000 hours at steam temperatures to 550C, with that for the same tubing after re-heat treatment. Since extensive long-term creep rupture data for service exposed, unexposed and laboratory aged material were available (6), the study offered a basis for comparison of the evaluation methods.

Creep Strength Evaluation

Creep rate data may be determined directly and comprehensively from a stress relaxation test (SRT). Basically, the total strain on the specimen is held constant in a closed loop machine, and the stress relaxes as the elastic strain is replaced by inelastic creep strain. At constant strain, therefore, the creep rate is equal to minus the elastic strain rate. The stress vs. time response during relaxation may thus be differentiated and divided by the elastic modulus to give the creep rate, which is then plotted against the stress (2).

For a test run lasting 20 hours, approximately five decades in creep rate are covered. This is, in fact, a self-programmed variable stress creep test that involves very little inelastic deformation so that multiple runs may often be made on a single specimen. The data represent a characterization of the material's current creep strength. Thus, changes in state induced by heat treatments or service exposures may be readily evaluated. Further discussion and comparison of this approach with traditional long-term creep testing may be found in references 7 and 8. These also cover the use of the CDR test to provide a separate measurement of fracture resistance.

Fracture Resistance Evaluation

Many failures in engineering alloys occur at intermediate temperatures where there is a ductility minimum (9). This reduced ductility may be accentuated for notched tensile tests because of the triaxial stress state at the notch root. A constant displacement rate (CDR) notch tensile test was originally proposed as a means to accelerate the development of notch sensitivity which may occur in long time notch rupture tests (10). It has since been used in tests at MPa as a basis for fracture resistance evaluation in short time tests at a temperature and strain rate where the alloy is most vulnerable to fracture. The constant displacement rate across the notch ensures that once a crack initiates it will grow under control until a critical crack length for fast fracture is exceeded. Thus, the displacement at failure and the extent of unloading at failure provide measures of the fracture resistance of the alloy.

Experimental Procedure

A length of serviced T91 tubing was sectioned perpendicular to the length. One half was heat treated and one half was tested as-serviced. The heat treatment was:
One hour austenitize at 1040C, remove from furnace and air cool (normalize), then 2 hours temper at 760C air cool.

Standard specimens with a 25.4mm gage section and 4.06mm gage diameter were machined from the tube material for SRT tests of both conditions. Additionally, notches were machined in two specimens to give a stress concentration factor of 3.15 in CDR tests. The complete test matrix for serviced S specimens and heat treated HT specimens was:

Specimen S1	SRT tests at two strain levels of 0.4% and 1.3% at 550C
Specimen S2	SRT tests at two strain levels of 0.4% and 1.3% at 600C
Specimen S3	SRT tests at two strain levels of 0.4% and 1.3% at 650C
Specimen S4	SRT tests at two strain levels of 0.4% and 1.3% at 700C
Specimen HT1	SRT tests at two strain levels of 0.4% and 1.3% at 550C
Specimen HT2	SRT tests at two strain levels of 0.4% and 1.3% at 600C

Specimen HT3	SRT tests at two strain levels of 0.4% and 1.3% at 650C
Specimen HT5	SRT tests at two strain levels of 0.4% and 1.3% at 700C

Specimen S4	CDR test to failure at 600C
Specimen HT4	CDR test to failure at 600C

For the SRT tests the procedure involved loading to the prescribed total strain levels of 0.4% and 1.3% in a servo-hydraulic machine, then holding the strain constant for twenty hours during which the stress relaxed as elastic strain was replaced with inelastic creep strain. After unloading the specimen was held at zero stress for two hours to monitor any anelastic strain recovery. Using the measured elastic modulus on loading, the stress vs. time response was converted to a stress vs. creep rate curve covering approximately five decades in creep rate. This curve is the primary product of the analysis and provides a comprehensive measure of the current creep strength of the material. The strain of 0.4% was selected to just exceed the elastic limit and provide a measure of the current creep strength. However, to conform to traditional analysis and design approaches, the data were further reduced to produce stress vs. projected time to 1% creep. The specimen prestrained to 1.3% corresponding to about 1% inelastic strain was used for this analysis. Since the projection is for several thousand hours, based on the initial strain and the very low creep rates obtained during relaxation in a twenty-hour test, it is appropriate to use the term pseudo time. Again, we emphasize that the SRT test does not contain information on time-dependent microstructural changes.

The CDR tests were run to failure under extensometer displacement rate control at a rate of 0.25mm/hour.

Results

SRT Tests

Figures 1-4 show the stress-strain curves for the loading stage of the SRT tests from which modulus values were computed for the creep rate analysis. The modulus was calculated as the mean of the loading and unloading curves for each specimen at each temperature. No systematic variation was noted for the HT and S specimens so the following averages were used for all specimens as a function of test temperature.

Test Temperature C	Young's Modulus MPa
550	156,000
600	131,000
650	116,000
700	94,000

The precision of maintaining constant strain during the relaxation tests is clearly indicated. Actual control on strain was $\pm 2 \times 10^{-5}$ as shown in figure 5 for the example of specimen HT1-2 at 550C. The HT specimens were significantly stronger at 550C and, for

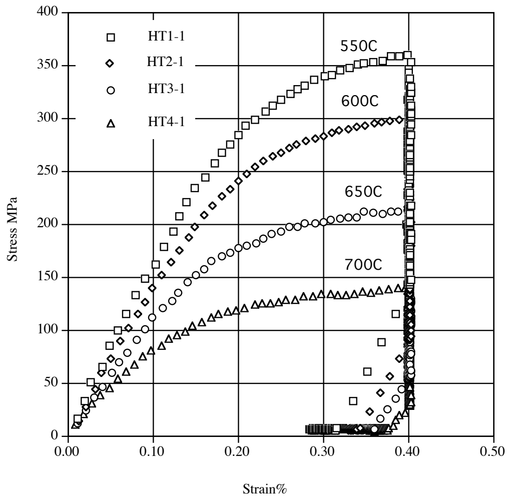


Figure 1 Stress-strain curves for heat treated condition to 0.4% strain

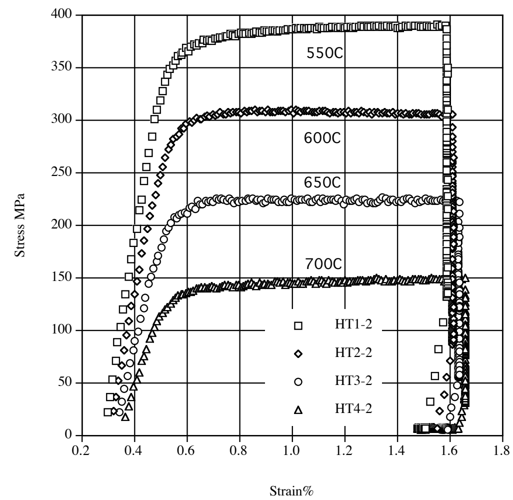


Figure 2 Stress-strain curves for heat treated condition to 1.3% strain

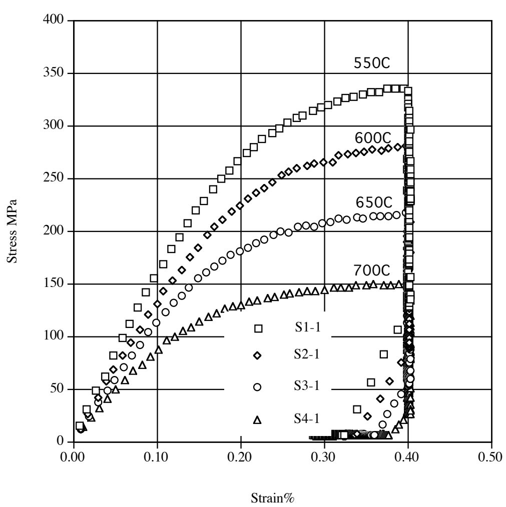


Figure 3 Stress-strain curves for serviced condition to 0.4% strain

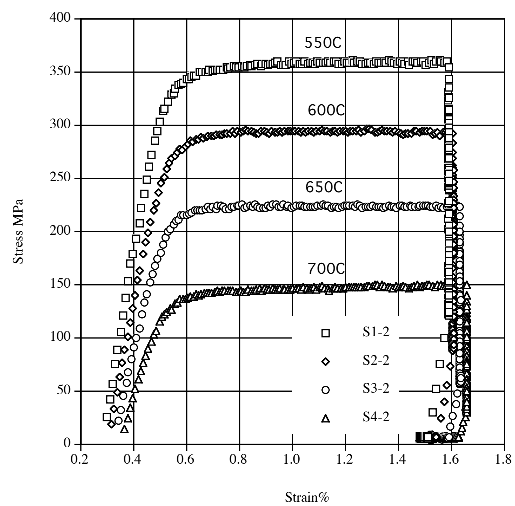


Figure 4 Stress-strain curves for serviced condition to 1.3% strain

low strains, at 600C. The anelastic recovery during holding at near zero stress was very small - less than 10% of the total inelastic strain.

Figures 6-9 show good consistency of the stress vs. ln time relaxation curves for all specimens. The curves were fitted with fourth order polynomials shown in the figures, which were then differentiated. The computed stress rates were converted to creep rates

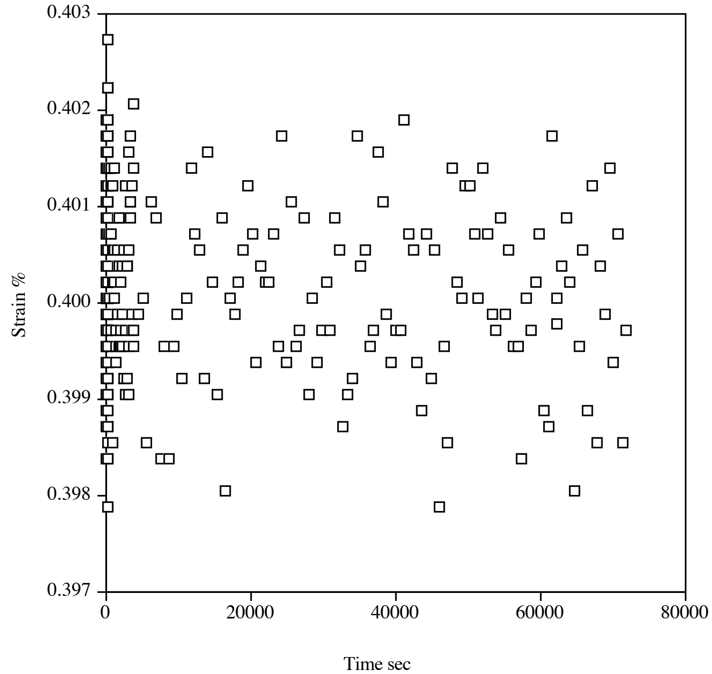
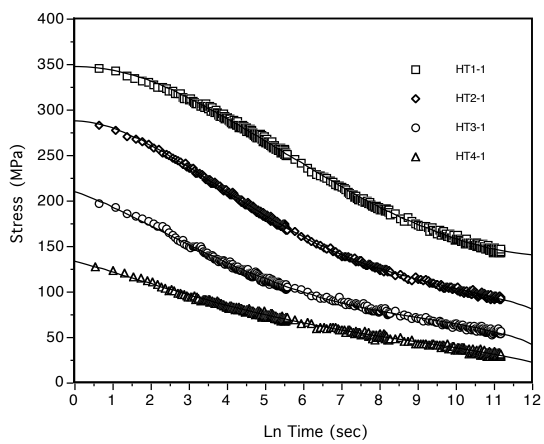


Figure 5 Example of strain control for specimen HT1-2



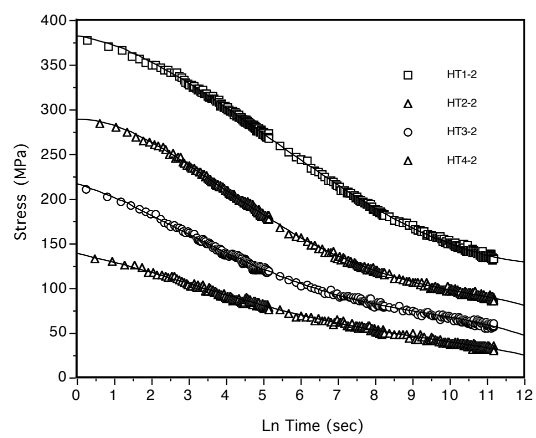
HT1-1 $y = 347.027 + 1.398x - 5.492x^2 + 0.434x^3 - 0.009x^4$ $r^2 = 0.999$

HT2-1 $y = 287.897 - 1.353x - 7.843x^2 + 0.964x^3 - 0.035x^4$ $r^2 = 0.999$

Ht3-1 $y = 209.603 - 14.146x - 3.145x^2 + 0.548x^3 - 0.024x^4$ $r^2 = 0.997$

HT4-1 $y = 133.828 - 12.285x - 0.435x^2 + 0.139x^3 - 0.007x^4$ $r^2 = 0.995$

Figure 6 Stress vs. ln time curves for heat treated condition to 0.4% strain



HT 1-2 $y = 382.104 - 6.311x - 4.857x^2 + 0.386x^3 - 0.007x^4$ $r^2 = 1.000$

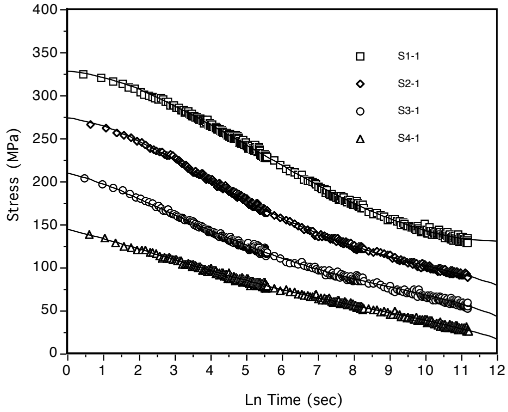
HT 2-2 $y = 289.584 - 0.226x - 8.361x^2 + 0.993x^3 - 0.035x^4$ $r^2 = 0.999$

HT3-2y $= 216.073 - 11.457x - 3.951x^2 + 0.613x^3 - 0.025x^4$ $r^2 = 0.998$

HT 4-2 $y = 138.243 - 7.844x - 1.790x^2 + 0.264x^3 - 0.010x^4$ $r^2 = 0.996$

Figure 7 Stress vs. ln time curves for heat treated condition to 1.3% strain

by dividing by the elastic modulus measured on loading. The derived log stress vs. log creep rate curves from both strain levels are shown in figures 10 and 11. For creep analysis 1.3% is chosen to ensure about 1% inelastic strain to compare with 1% creep strain in traditional tests. The actual amount is not critical since figures 2 and 4 show a near steady state stress after about 0.5% total strain.



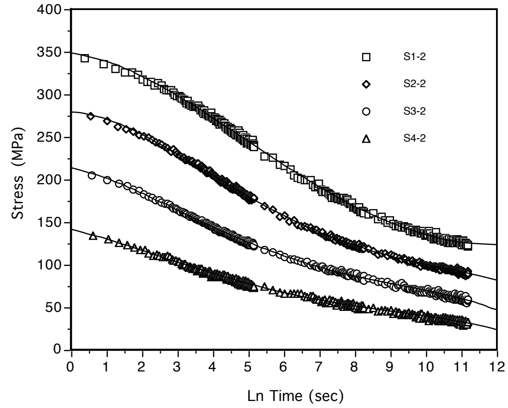
S1-1 $y = 328.443 - 3.865x - 4.081x^2 + 0.306x^3 - 0.005x^4$ $r^2 = 0.999$

S2-1 $y = 273.027 - 3.201x - 6.350x^2 + 0.782x^3 - 0.029x^4$ $r^2 = 0.999$

S3-1 $y = 210.068 - 10.736x - 3.070x^2 + 0.465x^3 - 0.019x^4$ $r^2 = 0.997$

S4-1 $y = 144.275 - 9.927x - 1.170x^2 + 0.201x^3 - 0.009x^4$ $r^2 = 0.997$

Figure 8 Stress vs. ln time curves for serviced condition to 0.4% strain



S1-2 $y = 347.984 - 4.749x - 5.159x^2 + 0.440x^3 - 0.009x^4$ $r^2 = 0.999$

S2-2 $y = 279.481 - 2.913x - 6.642x^2 + 0.788x^3 - 0.027x^4$ $r^2 = 0.999$

S3-2 $y = 213.335 - 8.469x - 4.260x^2 + 0.618x^3 - 0.025x^4$ $r^2 = 0.998$

S4.2 $y = 141.475 - 10.131x - 1.668x^2 + 0.291x^3 - 0.012x^4$ $r^2 = 0.996$

Figure 9 Stress vs. ln time curves for serviced condition to 1.3% strain

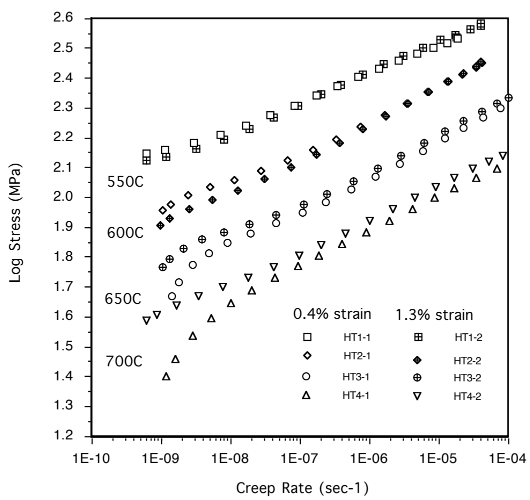


Figure 10 Log stress vs. creep rate for heat treated condition from both strain levels

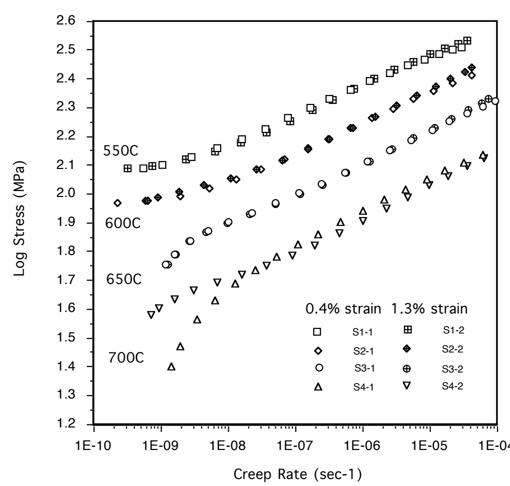


Figure 11 Log stress vs. creep rate for serviced condition from both strain levels

Figures 12 and 13 show comparisons for the calculated creep rates for 1.3% strain with minimum creep rate data measured in traditional creep tests. Figure 12 shows the comparison at 650C for the heat treated condition. Agreement is good at the lowest creep rate. Figure 13 shows the comparison at three temperatures for the serviced condition. Agreement is good at 650C and 700C. In general, consistent with previous studies on ferritic steels, agreement is best at higher temperatures and lower stresses.

Projected times to 1% creep were calculated from the 1.3% total strain curves of figures 12 and 13 at each stress by assuming that the specified creep strain is accumulated at the constant creep rate corresponding to that stress. Such projections have agreed well with

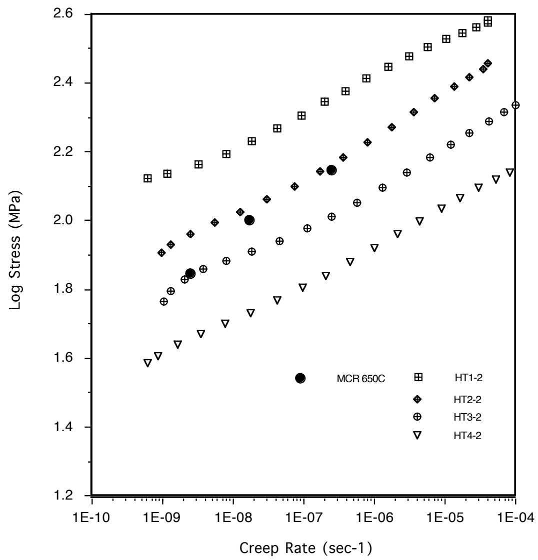


Figure 12 Log stress vs. creep rate for heat treated condition from 1.3% strain levels compared with mcr data at 650C

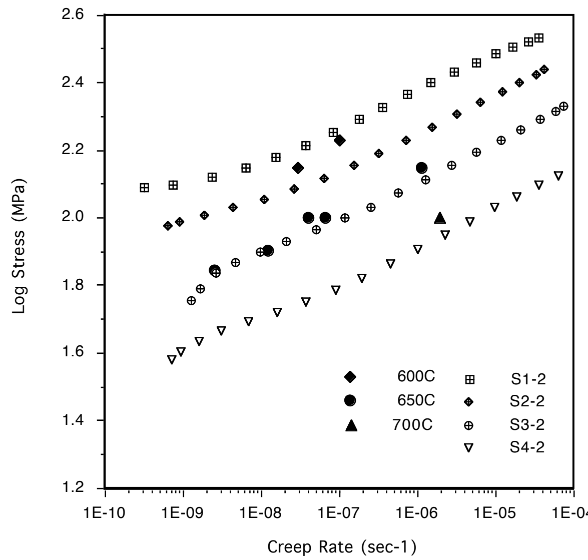


Figure 13 Log stress vs. creep rate for 116,000 hour serviced condition from 1.3% strain compared with mcr

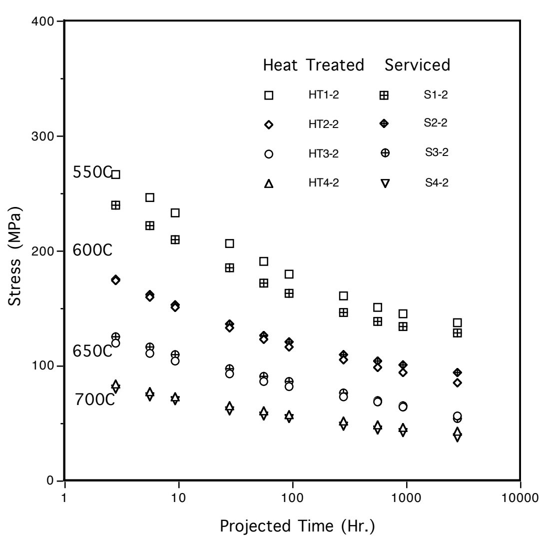


Figure 14 Stress vs. projected times to 1% creep strain

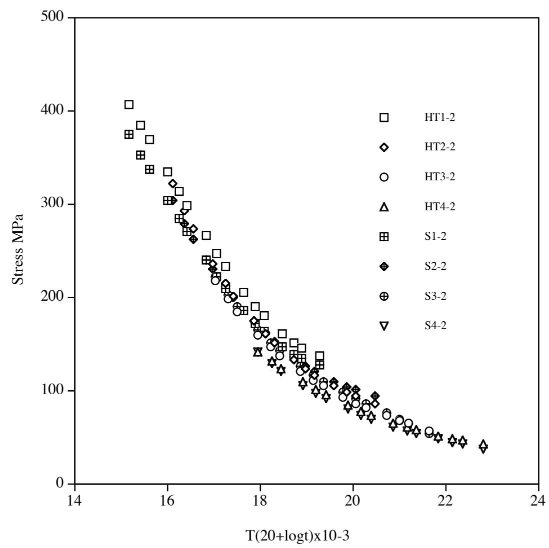


Figure 15 Stress vs. Larson-Miller Parameter with C=20 for projected time to 1% creep

traditional creep measurements for many polycrystalline alloys. Figure 14 shows the results for both conditions confirming higher creep strength at 550C after re-heat treatment. Finally, these computed points are combined in the Larson-Miller plot of figure 15, indicating a fairly good correlation using a standard constant of 20. Nevertheless, it is clear that some layering of the data occurs indicating that the correlation could be improved with refinement. This issue is addressed in the discussion.

CDR Tests

Only two CDR tests were run at one temperature, 600C. The results are plotted in figure

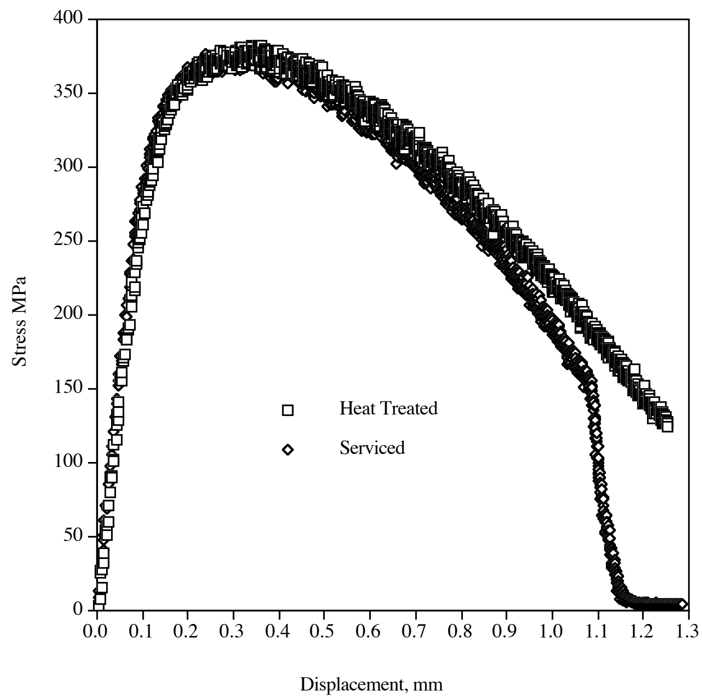


Figure 16 CDR tests at 600C on serviced and heat treated material

16. The stress levels are slightly higher for the heat treated condition, which is consistent with the smooth tensile data. However, an interesting difference was apparent in the fracture behavior. Much of the curve at stresses below the maximum relates to ductile crack propagation (10). This continued to failure in the serviced condition whereas brittle failure occurred at a critical crack length when the stress reached 124MPa in the heat treated condition. Thus the service condition results in reduced strength but enhanced fracture resistance relative to the heat treated condition, and by inference to the unexposed original condition. It may well be worth exploring this effect over a range of test temperature and relating the critical stress quantitatively to the prior history of processing and exposure. In any case, it is clear that long time service exposure does not result in any embrittling reactions.

Discussion

Some differences were noted in the tensile strengths for the two conditions based on figures 1-4. These differences are conveniently shown in figure 17, which plots the flow stresses at both strain levels. The heat treated specimens (re-normalized and tempered) are clearly stronger at 550C and to a lesser extent at 600C. At the highest temperature there is the possibility of a crossover although this could be due to test variability. The CDR notch bar test is also consistent with a strength increase at 600C and a fracture resistance decrease after heat treatment. Previous work on the tensile and creep strengths of serviced and aged material indicated a reduction in both properties compared with unexposed material for this alloy (6). However, in that work there was no indication of a

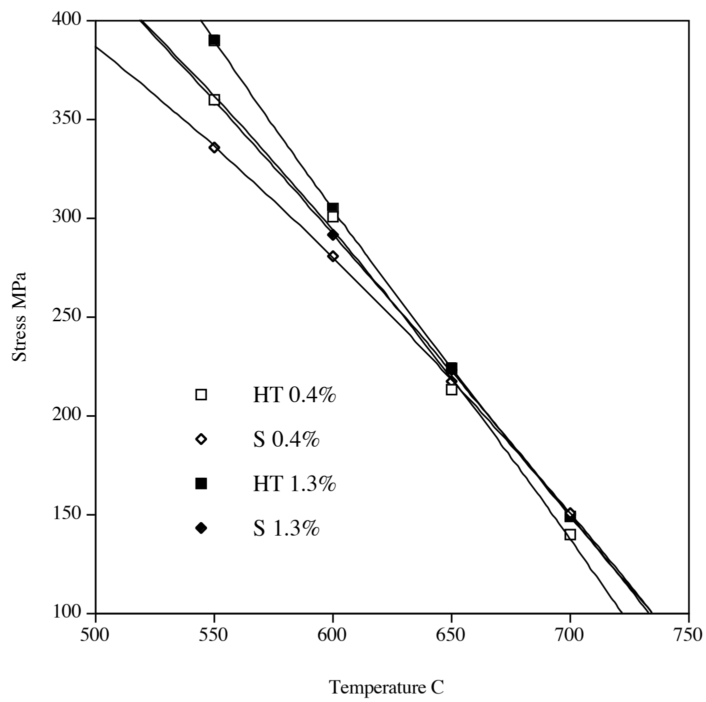


Figure 17 Flow stress at 0.4% and 1.3% total strain as a function of test temperature

specific influence of post exposure test temperature. In fact, if the primary service damage is a microstructural coarsening resulting in a general softening of the alloy it is to be expected that the effect would be noticeable only at lower temperatures and higher stress levels (in the case of creep strength) (11).

Traditional long-term creep testing and the Design for Performance approach have been compared in some detail recently (7). In this review the case was made for the new approach as an alternative rather than as a supplement. Nevertheless, there is interest in direct comparison of the two approaches. It should be anticipated that, since the deformation histories are different and that time-dependent microstructural evolution is minimized in the current approach, there would be differences. Where extensive primary creep is encountered in traditional testing, differences are expected. However, primary creep is very complex and not well understood. It may involve hardening and softening reactions as well as significant anelastic strain and is not generally included in detailed design analysis under nonsteady conditions.

When comparing with parametric representation of traditional creep rupture data it must also be recognized that time/temperature parameters are imprecise. For example, a recent analysis of data for the monocrystal CMSX-4 showed that the optimum value of the constant in the Larson-Miller parameter was stress dependent and ranged from 13.7 to 41.2 (12). By doing a similar iso-stress analysis on the data of figure 15, where there was

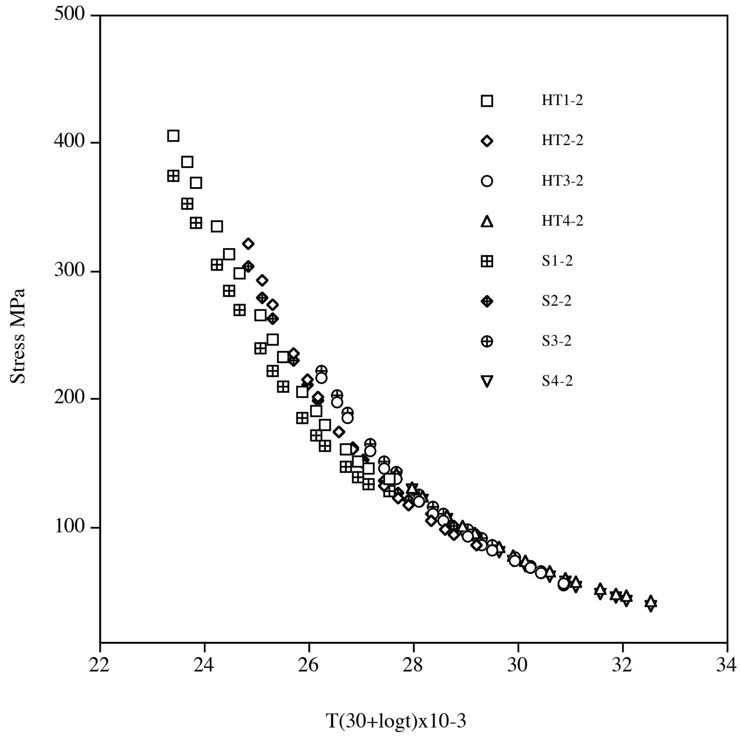


Figure 18 Stress vs. Larson-Miller Parameter with C=30 for projected times to 1% creep

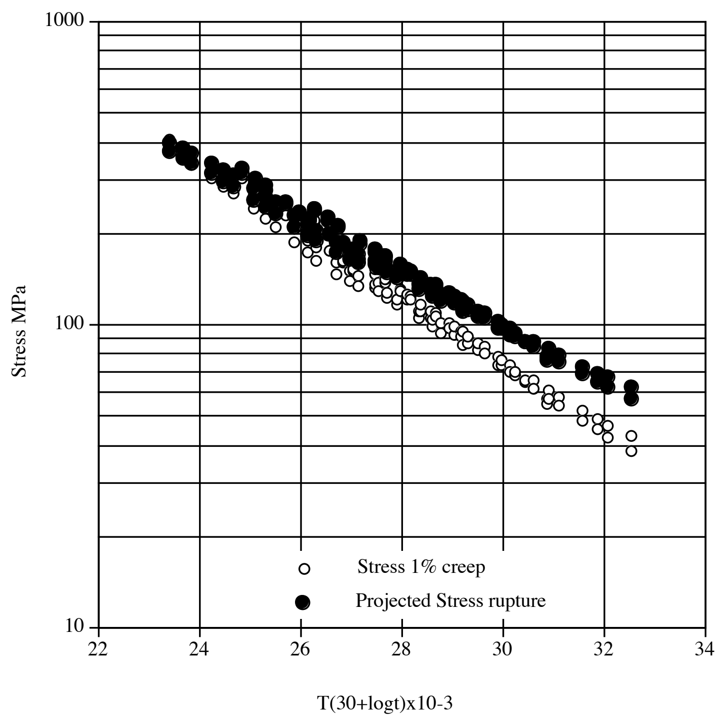


Figure 19 Stress vs. Parameter plot for projected time to 1% creep and computed time to rupture using the Gill-Goldhoff correlation

significant overlap at 125MPa, a value for the constant of 26 was calculated. However, since previous plotting has used a stress independent value of 30 for the constant (6) figure 18 shows this correlation. The correlation does not appear to be significantly improved compared with C=20 in figure 13, but it is useful as a basis for comparison with the traditional creep rupture data.

Since the SRT projections are for 1% creep we need a procedure to estimate rupture life curves. The Gill-Goldhoff correlation (13, 14) relates the stress for rupture to the stress for 1% creep in the same times. The equation developed in reference 13 was optimized for steel and expressed in ksi:

Log (rupture stress) = 0.3005 + 0.8266 x Log (1% creep stress)

The results of the analysis using this equation are shown in figure 19. For convenience the data for the two conditions are lumped together and the computed points plotted on a logarithmic stress plot.

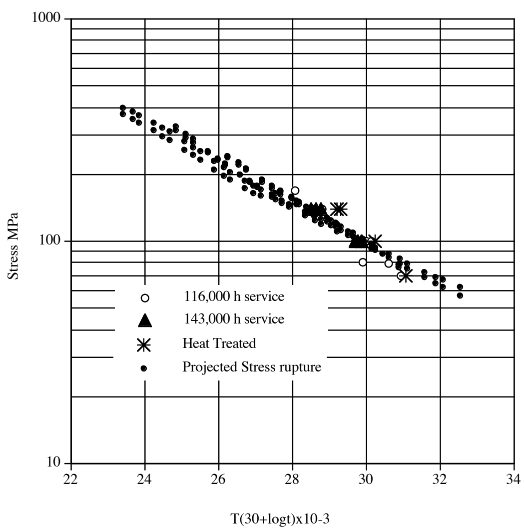


Figure 20a Stress vs. Parameter plot for projected time to rupture compared with actual long term data

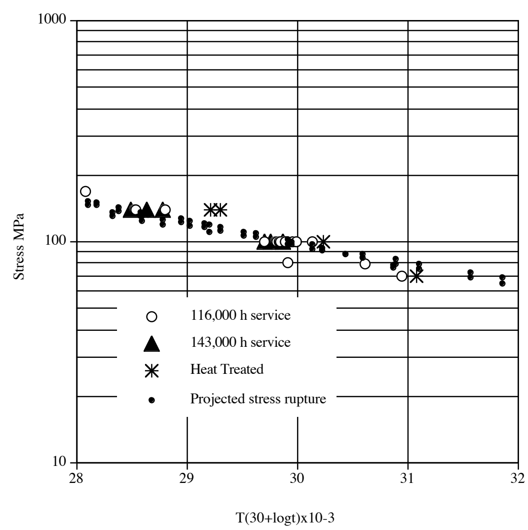


Figure 20b Stress vs. Parameter plot for projected time to rupture compared with actual long term data

Figure 20a shows the projected stress rupture values taken from figure 19 compared with ORNL results for rupture data up to 14,900 hours. These data include samples that had been exposed the same as the SRT tests for 116,000 hours, samples with 143,000 hours exposure, and some re-normalized and tempered material. The correlation appears good with a smaller spread and wider data coverage for the SRT tests. Finally, Figure 20b is an expanded plot of the data over the parameter range common to both types of test.

The excellent comparison (within the limits of the parametric correlation) of the SRT projections with the traditional stress rupture testing confirms the value of the accelerated testing. It should also be noted again that both tests have specific objectives and individual merits. The long term testing may incorporate time-dependent microstructural changes in the test, but this may be misleading unless the thermal mechanical service history is accurately simulated by the constant load creep rupture test. There is no *a priori* reason why one test should be used as the standard for judging the value of another. The only real criterion is whether a reliable and accurate framework for design can be based on the values measured by the test. The longest test time was about 18 months for the stress rupture data compared with the use of one machine for a few weeks for the SRT data. The latter actually covered a far greater range of creep rates and projected creep times.

Conclusions

1 Stress vs. creep rate data were readily obtained from stress relaxation tests on T91 at temperatures between 550C and 700C.

2 Increased tensile strength was measured in tests at 550C and 600C for the re-normalized and tempered condition compared to the serviced condition for 116,000 hours exposure.

3 Creep rate calculations and projected creep life curves also indicated increased creep strength at 550C after re-heat treatment compared with the service exposed condition.

4 Creep rate calculations, especially at higher temperatures and lower stresses, compared closely with minimum creep rate data for both the heat treated and serviced conditions

5 Constant displacement rate notched tests at 600C indicated that the serviced condition showed lower strength but increased fracture resistance.

6 The general property degradation in service suggested a softening due to microstructural coarsening with no evidence for embrittling reactions.

7 A simple Larson-Miller parameter correlation for projected times to 1% creep was generally applicable for both heat treatments.

8 Existing long-term rupture data correlated very well with projections of rupture lives based on the SRT tests.

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References

1 F. C. Monkman and N. J. Grant, Proc. ASTM, vol. 56, 1956, p. 595.

2 D. A. Woodford, “Test Methods for Accelerated Development, Design and Life Assessment of High-Temperature Materials,” Materials and Design, vol. 14, no. 4, 1993, p. 231.

3 D. A, Woodford, "Creep Analysis of Directionally Solidified GTD111 based on Stress Relaxation Testing," Materials at High Temperatures, vol. 14, no.4, 1997, p. 413.

4 D. A. Woodford, "Creep Design Analysis of Silicon Nitride using Stress Relaxation Data," Materials & Design, vol.17, no.3, 1996, p. 127-132.

5 S. K. Reif, K. J. Amberge and D. A. Woodford, Materials and Design, vol. 16, no. 1, 1995, p. 15.

6 R. W. Swindeman, P. J. Maziasz and C. R. Brinkman, “Aging Effects on the Creep-Rupture of 9Cr-1Mo-V Steel,” Proc. Int. Joint Power Generation Conference, Miami Beach, Florida, July, 2000.

7. D. A. Woodford, "Design for High Temperature Applications," Materials Selection and Design, ASM Handbook, Volume 20,1997, p.573-588.

8. “Accelerated Testing for High-Temperature Materials Performance and Remaining Life Assessment,” EPRI, Palo Alto, CA: 1999, TR-114045.

9 N. F. Fiore, “Mid-Range Ductility Minimum in Ni-Base Superalloys,” Reviews on High Temperature Materials, vol. 2(4), 1975, p. 373-408.

10 J. J. Pepe and D. C. Gonyea, “Constant Displacement Rate Testing at Elevated Temperatures,” Int. Conf. Fossil Power Plant Rehabilitation, ASM Int., 1989, p. 39.

11 D. A. Woodford, “Creep Damage and the Remaining Life Concept,” J. Eng. Mat. and Techn., vol. 101, 1979, p. 311.

12 D. A. Woodford,” Parametric Analysis of Monocrystalline CMSX-4 Creep and Rupture Data,” Met. and Mat. Trans., vol. 29A, 1998, p.2645-2647.

13 R. F. Gill and R. M. Goldhoff, “The Analysis of Long-Time Creep Data for Determining Long Time Strength, ASM publication, No. P9-101, 1969.

14. R. M. Goldhoff and R. F. Gill, “A Method for Predicting Creep Data for Commercial Alloys on a Correlation Between Creep Strength and Rupture Strength,” ASME paper no. 71-WA/Met-2, 1972.