

## ACCELERATED HIGH TEMPERATURE PERFORMANCE EVALUATION FOR ALLOY OPTIMIZATION, EMBRITTLEMENT, AND LIFE ASSESSMENT

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### ABSTRACT

Creep strength and fracture resistance are two properties that are critical in the selection and optimization of high temperature materials. These same properties may be progressively impaired by service exposure and are, therefore, key to the assessment of remaining life of high temperature components. In recent years, a methodology based on accelerated measurements of these properties has been developed. The two critical properties are decoupled in this approach, which allows a clearer separation of the effects of microstructural evolution, damage development and environmental attack. The evaluation of creep strength, based on a high-precision stress relaxation test (SRT), and fracture resistance, based on a constant displacement rate test (CDR), are illustrated using extensive data on a conventionally cast nickel based alloy (alloy 738) and also some recent work on a ferritic steel (T91). The well-known effect of section size on rupture life of conventionally cast alloys is shown to be an effect on fracture resistance rather than creep strength. By using miniature specimens the variation in creep strength in critical components can be quickly determined. The comparisons with long term creep rupture test data for both alloy classes show good agreement indicating that the accelerated testing is quite capable of yielding both comparative and design data. With the added advantage of a separate fracture resistance criterion the new methodology should be broadly useful.

## KEYWORDS

Creep strength, high temperature fracture, stress relaxation testing, nickel based alloy, ferritic steel, accelerated testing, life assessment, miniature specimens, design analysis.

## INTRODUCTION

The uniaxial creep rupture test provides the primary basis for the selection and design of materials for high temperature equipment. These tests may last for upwards of 10,000 hours, and it is generally believed that the longer the test time the more accurate may be the prediction of component failure<sup>(1)</sup>. However, modern approaches to repair, rejuvenation or replacement of critical components require rapid turnaround and seek accurate assessment of material performance capability. Hence, there is a strong interest in accelerated testing.

In addition to the need to generate suitable material data in a short time there are more fundamental objections to the creep rupture test. The test involves an arbitrary combination of deformation processes and fracture processes, both creep strength and fracture resistance are evaluated from the same basic test, and the properties that are being measured are changing during the course of the test. All these complexities would be acceptable if the test suitably reflected operating conditions. In reality, however, most components experience non-steady stresses and temperatures, multiaxial stresses, cyclic stresses, active and often aggressive gaseous environments, and synergistic interactions among these factors. These concerns are especially applicable to the combustion turbine. Moreover, long time data have little relevance to cyclic operations where the zero time origin for creep analysis should be reset for each cycle.

With these deficiencies in mind, an alternative approach to testing and evaluation has been developed<sup>(2,3)</sup>. The principal features of this Design for Performance methodology may be summarized as follows:

- Creep strength and fracture resistance are de-coupled and measured in separate tests or as distinctly different properties.
- Both properties are measured as current values so that time-dependent changes during the test are minimal.
- Creep strength is evaluated in terms of creep rate rather than time.
- Fracture resistance is evaluated in terms of crack extension or ductility rather than time to failure.
- State of damage is assessed in terms of current values of the critical properties.

### 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<sup>(2,3)</sup>.

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 reference 4.

These data can be used in a number of ways:

- They can be used for comparison to optimize chemistry, processing or heat treatment.
- They can be used to set design stresses for limiting creep rates.
- They can be used directly in finite element analysis where creep rate as a function of stress and temperature is required.
- They can be used to establish current creep strength of service exposed components as a basis for remaining life assessment decisions.
- The data may also be analyzed to give direct comparison with times to specific creep strains or rupture lives.

### Fracture Resistance Evaluation

Many failures in engineering alloys occur at intermediate temperatures where there is a ductility minimum<sup>(5)</sup>. This reduced ductility may be accentuated for notched tensile tests because of the triaxial stress state at the notch root. It may also be developed directly from gas phase embrittlement (GPE) resulting from intergranular penetration of aggressive gaseous species, especially oxygen<sup>(6)</sup>. 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<sup>(7)</sup>. 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. For miniature specimens the CDR approach has been used with unnotched specimens.

For both notched and smooth specimens the displacement at failure and the extent of unloading at failure provide measures of the fracture resistance of the alloy. Although these measures give only qualitative comparison, they fulfill the need for an accelerated test to describe the current fracture resistance. Ultimately they allow a lower bound fracture resistance to be specified for a given application. More sophisticated crack propagation tests in general require larger specimens and, because of the need to approach design stresses, much longer test times. As with the traditional creep strength evaluation methods, this approach is not expected to be able to duplicate operating thermal-mechanical conditions.

## MATERIALS AND METHODS

This paper summarizes recent results on the conventionally cast nickel based turbine blade alloy (alloy 738) and the 9%Cr-1Mo-V-Nb iron based boiler tube alloy (T91).

Standard tensile specimens 76 mm long with a gage section of 25.4 by 4.1mm diameter were used for both alloys. In addition, a miniature specimen 41.3mm long with a gage section of 25.4 by 2mm diameter was developed specifically for creep strength evaluation in thin sections of gas turbine blades. The SRT test procedure involved loading to the prescribed strain level, then holding the strain constant for twenty hours during which the stress relaxed as elastic strain was replaced with inelastic creep strain. 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, as described previously<sup>(2,3)</sup>.

For the equiaxed structure loading to 0.4% total strain (i.e. about 0.1% inelastic strain) minimized transient effects and provided a stress vs. creep rate response characteristic of the current creep strength. However, to conform with traditional analysis and design approaches, the data were further reduced to produce stress vs. projected time to 0.5% creep strain for the alloy 738. For this representation a total strain level of 0.8% (approximately 0.5% inelastic strain) was used. 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. It should be emphasized that the SRT test does not contain information on time-dependent microstructural changes beyond the test duration. Exact agreement with traditionally generated long time data is therefore unlikely because strain is not a state variable. However, this comparison serves as a legitimate calibration of the two methods. It is appropriate to compare the methods but not to judge their usefulness in terms of how closely either corresponds to the other. As discussed later, each test uses a different deformation path, neither of which conforms to the extremely complex thermomechanical history of gas turbine blades.

The same procedure was used for the SRT tests on the T91 steel except that a total strain of 1.3% was used to provide a basis for comparison with traditional creep data to 1% creep strain. The SRT tests were run on standard specimens taken from serviced (116,000 hours at steam temperatures up to 550°C) and re-heat treated material.

In the case of the CDR tests notches were machined in standard specimens to give a stress concentration factor of 3.15. However, for the blade alloy smooth miniature specimens were used because of the desire to sample from thin sections. The CDR tests were run to failure under extensometer displacement rate control at a rate of 0.25mm/hour.

## RESULTS ON IN738

### SRT Tests

Figure 1 shows an example of a relaxation sequence at 850°C from four strain levels of material taken from the root section of a service exposed blade<sup>(8)</sup>. It is normal practice to assume that this represents undamaged material because of the low operating temperature in this region. The four successive runs are plotted in terms of stress vs. ln time and fitted to fourth order polynomials. The form of these curves allowed easy differentiation, and the creep rate was then obtained by dividing the stress rate by the elastic modulus measured on loading according to the following:

$$e_e + e_i = e_t = \text{constant} \quad (1)$$

$$\frac{de_i}{dt} = \frac{-de_e}{dt} = -\frac{1}{E} \cdot \frac{d\sigma}{dt} \quad (2)$$

Where:  $e_e$  = elastic strain  
 $e_i$  = inelastic creep strain  
 $e_t$  = total strain  
 $\sigma$  = stress  
 $E$  = elastic modulus

A plot of log stress vs. creep rate at three temperatures from 0.4% total strain is shown in Figure 2. This includes duplicate testing at 900°C on the standard specimen, and results on a miniature specimen taken from an adjacent location at 850°C. The small differences for the two tests at 900°C may be real since there was a small but significant difference in measured modulus. The average loading values used for  $E$  were 145,000 (800°C), 140,000 (850°C), 140,000 (900°C) and 135,000 MPa (900°C repeat.). The values were typically repeatable to within 10%, which correspondingly leads to a variation in creep rate and projected time of 10%. Results for the miniature specimen at 850C indicate no specimen size effect on creep strength. The modulus was identical to that measured on the larger specimen (140,000 MPa).

Using an intercept at log stress=2.5, and an average creep rate for the duplicate tests at 900°C, it was possible to develop an Arrhenius type scaling law from Figure 2. This allowed a master plot to be constructed covering 10 decades in creep rate and extrapolation of the lower temperature data. Thus, Figure 3 shows possible design points at 800°C and 850°C based on creep rates of  $3 \times 10^{-11} \text{ sec}^{-1}$  (1% in 100,000hr.).

Using the 0.8% SRT tests, a pseudo time was calculated for various creep rates and 0.5% creep, and plotted as projected times in Figure 4 for the three temperatures. For example, the lowest creep rate of  $10^{-9} \text{ sec}^{-1}$  gives a pseudo time of 1,390 hours. The resulting curves are shown as linear plots on a semi-logarithmic scale. Also shown in the Figure are similar projections taken from a second heat of alloy 738, and actual times to 0.5% creep for a third heat of alloy 738 taken from the internal files of MPA, Inc. Although quantitative comparisons are not possible because three heats were used, the actual creep times are within the range of the two projected time curves based on the SRT tests at all temperatures.

An alternative correlation between traditional creep measurements and SRT projections is shown in a separate study in which equivalent rupture lives were estimated based on creep to specific strains of 5%, 10% or 15%<sup>(8)</sup>. An example of this analysis for alloy 738 in terms of a Larson-Miller parameter is shown in Figure 5. Similar analyses for other alloys indicated that rupture lives can be estimated within a factor of three using this approach. In all analyses the correlation improved with increasing parameter values, i.e. increasing temperature or decreasing stress.

A fairly extensive study of miniature specimens of IN738 taken from a serviced blade and vane in Figure 6 in terms of stress-creep rate at 850°C shows that the blade root and thicker vane sections have significantly higher creep strength than all the other specimens<sup>(9)</sup>. This may be a response to a larger grain size and dendrite arm spacing in these areas. However, it is possible that it is primarily an orientation effect. For example, of the vane specimens, the two higher

strength locations had measured elastic moduli of 133,000 and 138,000 compared with 125,000, and 124,000 MPa, for the others. The standard specimen result for the blade root confirm the absence of a specimen size effect. The two minimum creep rate points fall on the lower bound of the data.

### CDR Tests

Rupture life is primarily a measure of creep strength; fracture resistance should better be identified with a separate measure that reflects the concern with embrittlement phenomena, which may lead to component failure. Most engineering alloys lose ductility during high temperature service. At a fixed strain rate for example, ductility first decreases with increasing temperature. This is believed due to grain boundaries playing an increasing role in the deformation process leading to the nucleation of intergranular cracks. At still higher temperatures, processes of recovery and relaxation at local stress concentrations lead to an improvement in ductility. Maximum embrittlement generally occurs in a critical range of temperature and strain rate.

With cast superalloys gas phase embrittlement (GPE) resulting from intergranular penetration of aggressive gaseous species is of greatest concern. Such penetration is invariably a major factor in sustained load cracking (creep crack growth) and time-dependant fatigue crack growth. The embrittlement that occurs due to this intergranular penetration and GPE of oxygen is illustrated in the CDR data shown in Figure 7<sup>(9)</sup>. In this figure, the specimens were smooth miniature specimens suitable for removing from the thin sections of gas turbine blades. This type of embrittlement has been observed in specimens taken from thin sections of serviced blades.

## RESULTS ON T91

### SRT Tests

Figures 8 and 9 show comparisons for the calculated creep rates for 1.3% strain with minimum creep rate data measured in traditional creep tests. Figure 8 shows the comparison at 650°C for the heat treated condition. Agreement is good at the lowest creep rate. Figure 9 shows the comparison at three temperatures for the serviced condition. Agreement is good at 650°C 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 8 and 9 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 traditional creep measurements for many polycrystalline alloys. Figure 10 shows the results for both conditions confirming higher creep strength at 550°C after re-heat treatment.

When comparing with parametric representation of traditional creep rupture data it must be recognized that time/temperature parameters are imprecise. For example, a recent analysis of

data for the monocrystal CMSX-4<sup>1</sup> showed that the optimum value of the constant in the Larson-Miller parameter was stress dependent and ranged from 13.7 to 41.2<sup>(10)</sup>. By doing a similar iso-stress analysis on the data of figure 10, where there was significant overlap at 125MPa, a value for the constant of 26 was calculated. However, since previous plotting of traditional creep rupture data has used a stress independent value of 30 for the constant<sup>(11)</sup>, Figure 11 shows this correlation. The correlation was not significantly improved compared with C=20, but it is useful as a basis for comparison with the traditional creep rupture data. Nevertheless, it is clear that some layering of the data occurs indicating that the correlation could be improved with refinement.

Since the SRT projections are for 1% creep we need a procedure to estimate rupture life curves. The Gill-Goldhoff correlation<sup>(12, 13)</sup> relates the stress for rupture to the stress for 1% creep in the same times. The equation developed in reference 12 was optimized for steel and expressed in ksi:

$$\text{Log (rupture stress)} = 0.3005 + 0.8266 \times \text{Log (1\% creep stress)}$$

The results of the analysis using this equation are shown in Figure 12. For convenience the data for the two conditions are lumped together and the computed points plotted on a logarithmic stress plot. Figure 13 shows the projected stress rupture values taken from Figure 12 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.

### CDR Tests

Only two notched CDR tests were run at one temperature, 600°C. The results are plotted in Figure 14. 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<sup>(7)</sup>. 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 is clear that long time service exposure does not result in any embrittling reactions in this case.

## DISCUSSION

The results presented in this paper for a cast nickel based superalloy and a wrought ferritic steel are used to evaluate the SRT test as a basis for creep strength measurement and the CDR test as a basis for high temperature fracture resistance evaluation. As stated previously, the traditional creep rupture test incorporates changes in microstructure and damage evolution during the test. Such changes are relevant only if the application involves a similar deformation history. The complexities associated with combustion turbine operation, particularly cyclic operation, render

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<sup>1</sup> CMSX-4 is a trade name of the Cannon-Muskegon Corporation

the long time test and extrapolation of test data of little relevance. The most striking example of this may be the measurement of a finite rupture life in a failed part. The material of the failed part clearly has finite creep strength even though the part has failed. This paradox is a direct consequence of the fact that the creep rupture test does not measure the current state but rather a complex integration of all the changes that occur during the test. Although the service exposure of boiler components may see less cycling and thermal-mechanical complexity, there is still an incentive to develop accelerated testing, particularly to evaluate the current state, i.e. remaining life assessment.

The SRT test is designed to provide a comprehensive description of the current creep strength. The change in state during the evaluation is minimal so that the test gives a good indicator of the consequences of changes resulting from processing or service exposure. For example, the test results of Figures 2 and 3 provide a comprehensive characterization of a given heat of alloy 738. They may be used to evaluate the effect of heat treatment and exposures, and also for heat to heat comparison. Figure 3 may be used to provide design points such as those indicated at 800°C and 850°C for creep rates of 1% in 100,000 hours. Additionally, the parametric correlation of Figure 3 may be used as direct input in finite element creep analysis. If significant creep strains are anticipated then similar master curves may be made at different inelastic strain levels. The prestrain is the primary selection criterion for the SRT test. Whereas in the traditional creep test the strain is accumulating in an arbitrary manner under different test conditions and thus changing the state in an uncontrolled way, the SRT response may be controlled systematically by the choice of prestrain. On this basis a choice of 0.4% prestrain for equiaxed alloys minimizes transient effects and provides a stress vs. creep rate response characteristic of the current creep strength. However, to produce stress vs. projected time to 0.5% creep strain a total strain level of 0.8% (approximately 0.5% inelastic strain) was used. Similarly, for the 1% creep strain criterion used in the steel analysis, a total strain of 1.3% was used. In this case the strain is not critical since the tensile curves reach a near steady state stress after about 0.5% strain.

For high strength superalloys it has been found that time projections, such as those shown in Figure 4, generally agree quite well with long time creep data. This is because these alloys are often not very sensitive to microstructural changes. Since alloy 738 is a widely used blade alloy for industrial gas turbines the time projections may be used with traditional design criteria to assess remaining life as in the parametric representation of Figure 5<sup>(5)</sup>. For the purpose of the analysis the creep rate is assumed constant for a specific strain to failure. In terms of predicting remaining life of components these values are no less accurate than actual rupture time measurements. They should be viewed, however, only as an improved way of measuring the current creep strength. For a better evaluation of remaining life, separate measurements of fracture resistance should be made as described in detail elsewhere<sup>(6)</sup>.

Clearly, the SRT approach allows the detailed mapping of creep strength, using miniature specimens if necessary, for different locations in a blade in the unexposed condition. Such data would be invaluable for post-service condition assessment. Comparisons may also be rapidly established for orientation studies, chemistry variations and possible test specimen size effects. In addition to the effect of section thickness in influencing solidification rate and consequent microstructure, there may be an effect of test specimen size for specimens taken from the same region and microstructure.

Figure 2 indicates that for specimens taken from the same location, implicitly with a similar microstructure, there are actually no significant effects of test section size on creep strength. This is a particularly interesting and important observation since previous studies have demonstrated a strong effect of section size on rupture life (reduced life in smaller sections) in equiaxed castings<sup>(14)</sup> but not in longitudinally oriented DS castings or monocrystals of the same alloy<sup>(15)</sup>. One likely explanation is that the reduced rupture life in small specimens is associated with embrittlement due to oxygen penetration and environmentally enhanced intergranular cracking<sup>(6)</sup>. The effect is absent if there are no transverse grain boundaries. In the present work, fracture processes should not influence the results and they do in fact indicate that the creep strength in equiaxed castings is not affected by section size. We anticipate, therefore, that there would be section size effects on fracture resistance in equiaxed alloys. These results indicate that the creep strength of a particular microstructure (same grain size, dendrite spacing and modulus) should be independent of test specimen size to a good approximation. However, as indicated above, the creep strength of different sections within a component may vary considerably because of the influence of solidification rate on microstructure.

The excellent comparison (within the limits of the parametric correlation) of the SRT projections with the traditional stress rupture testing for the ferritic T91 steel 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.

#### ACKNOWLEDGEMENT

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#### CONCLUSIONS

- The standard creep rupture test is unsuited as a basis to evaluate the high temperature creep strength or life of a component because the critical properties are changing during the course of the test.
- A high-precision stress relaxation test (SRT) is capable of comprehensively characterizing the current creep strength in a short time in terms of the stress vs. creep rate with minimal change in state during the test.
- SRT testing of alloy 738 and T91 demonstrates the application to alloy selection and optimization, design analysis and life assessment.
- Specifically it is shown that creep strength defined in terms of stress vs. creep rate is unaffected by test specimen size for both equiaxed alloys.

- The SRT test is especially suitable for evaluating the variation in creep strength with location in components before and after service.
- Embrittling reactions that may occur during service exposure can be readily detected using a constant displacement rate test.
- It is shown how the new methodology may provide an approach to both design and life prediction that is faster, cheaper and better.

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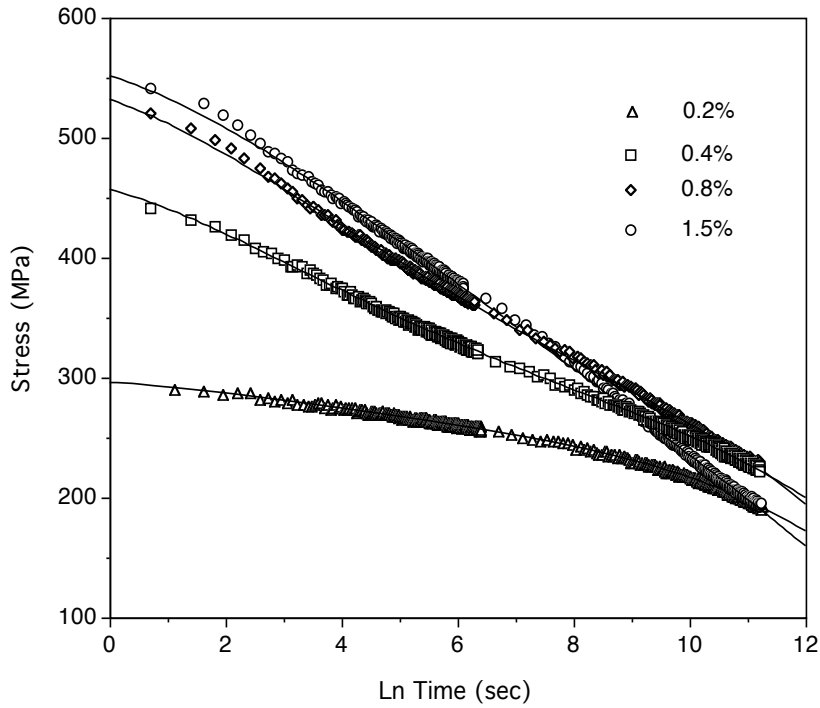


Figure 1 Stress vs. Ln time curves from various strain levels at 850°C for IN738

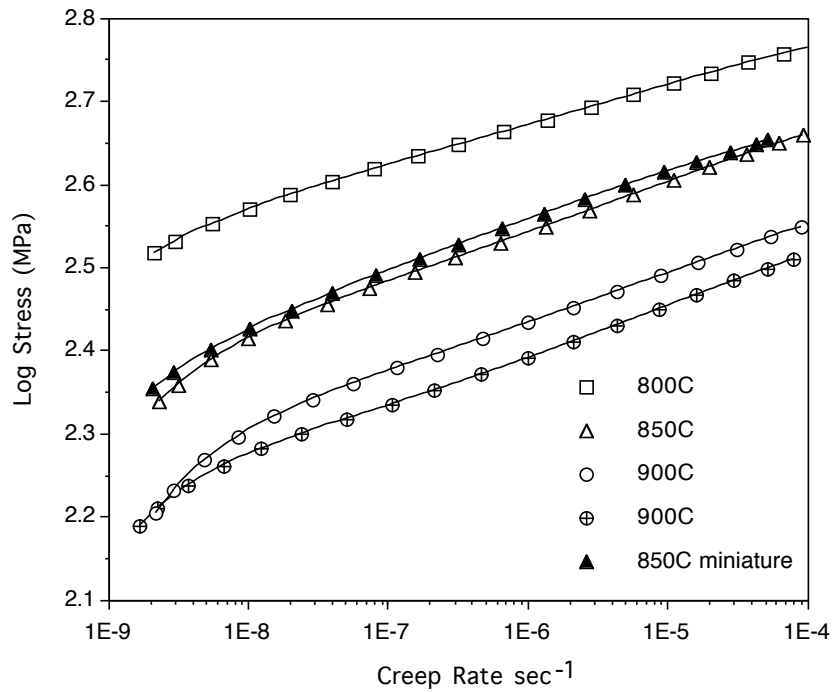


Figure 2 Stress vs. creep rate curves for alloy 738 from 0.4% strain as a function of test temperature

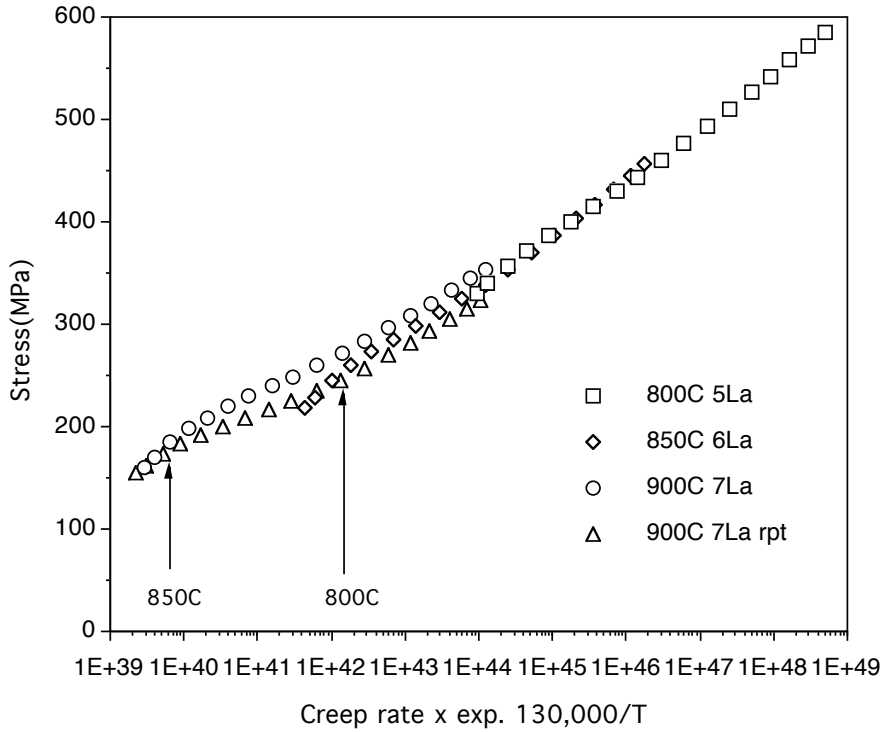


Figure 3 Stress vs. parameter plot for alloy 738 showing possible design points at 800°C and 850°C

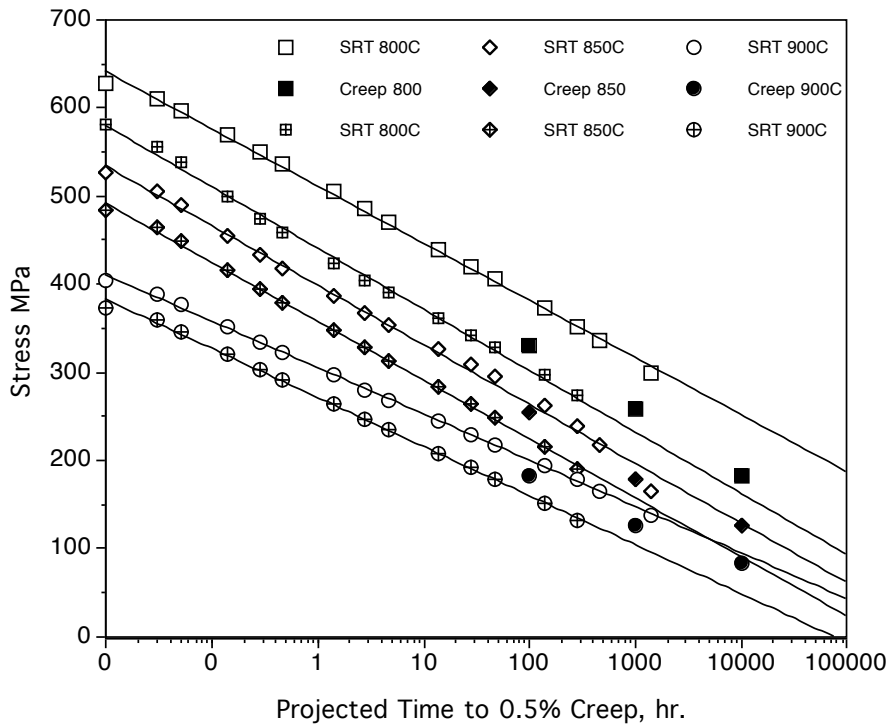


Figure 4 Stress vs. projected time to 0.5% creep in two heats of alloy 738 compared with actual long time creep data in a third heat

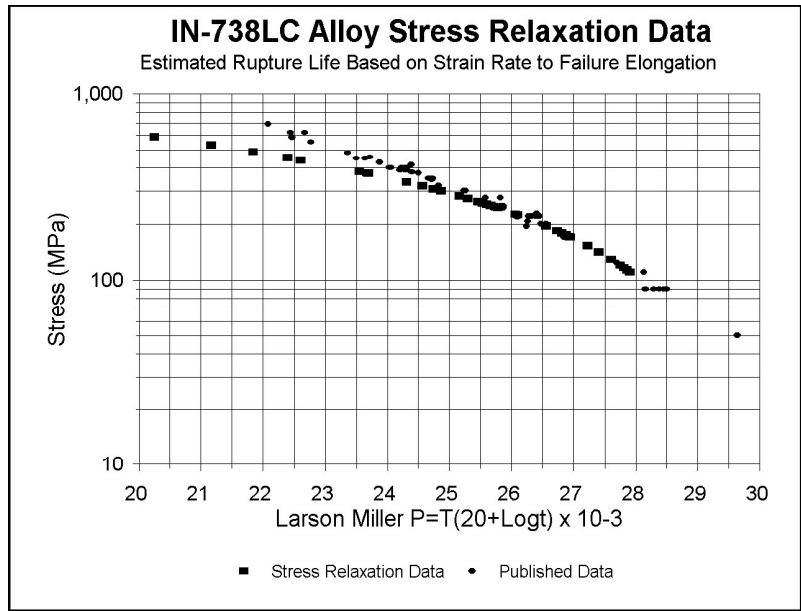


Figure 5 Comparison for alloy 738 of SRT projected rupture life from tests at 800°C, 850°C and 900°C with published data

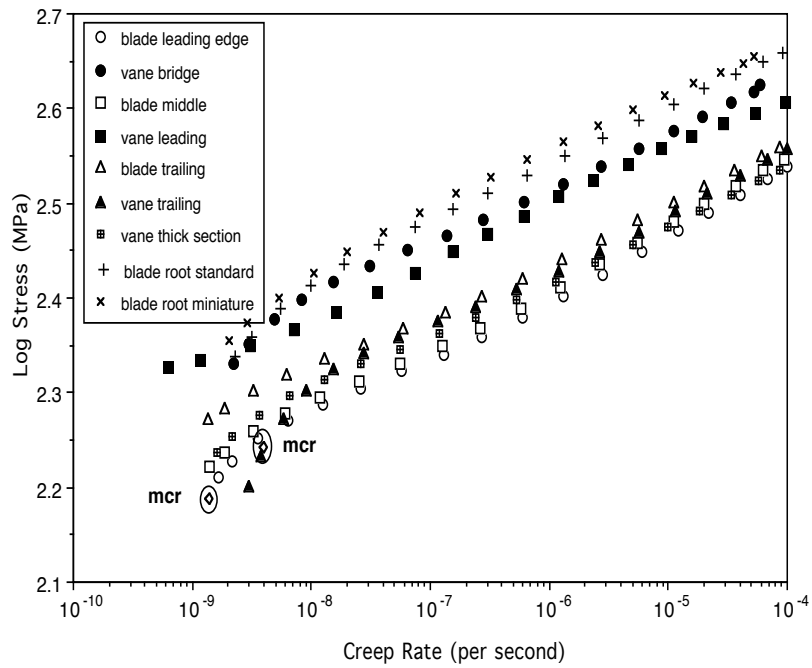


Figure 6 Miniature specimen data for alloy 738 at 850°C taken from serviced blade

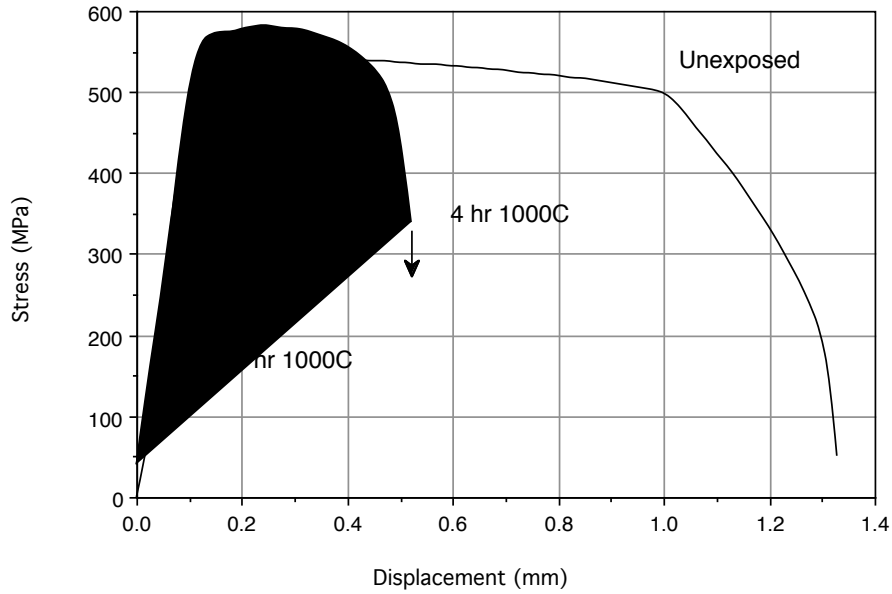


Figure 7 Effect of oxygen embrittlement for various exposures in air at 1000°C in tests at 800°C on CDR data for alloy 738

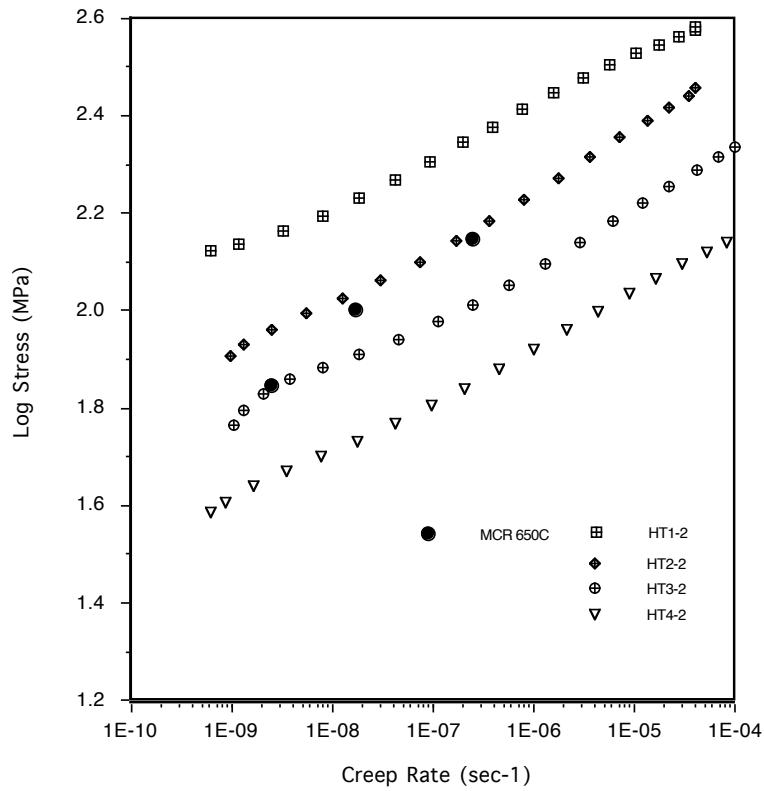


Figure 8 Log stress vs. creep rate for T91 heat treated condition from 1.3% strain levels compared with MCR data at 650°C

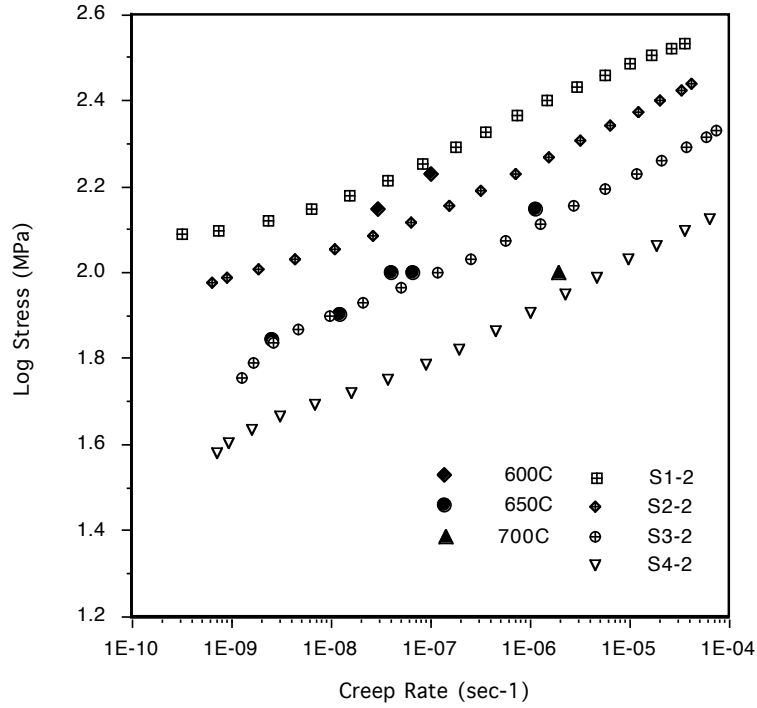


Figure 9 Log stress vs. creep rate for 116,000 hour serviced condition of T91 from 1.3% strain compared with MCR

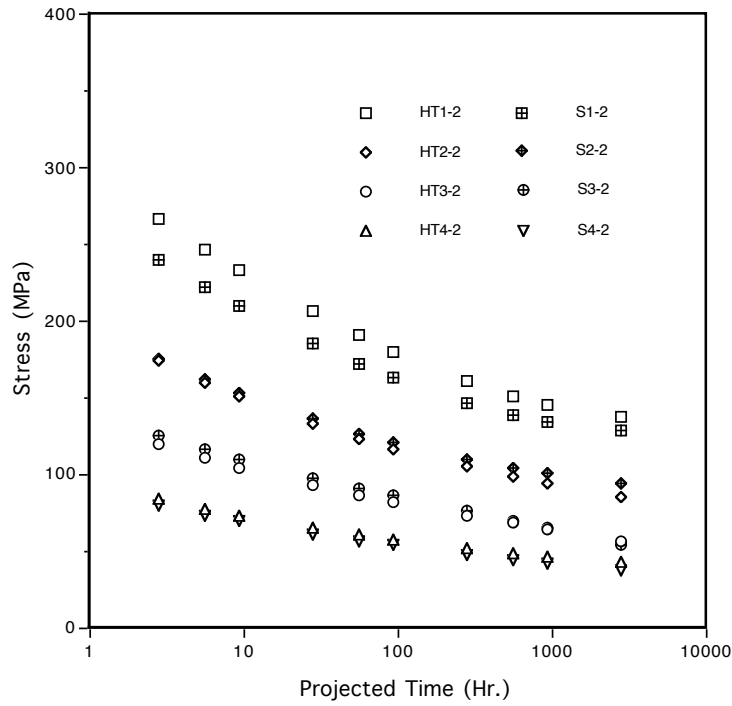


Figure 10 Stress vs. projected times to 1% creep for serviced and heat treated T91

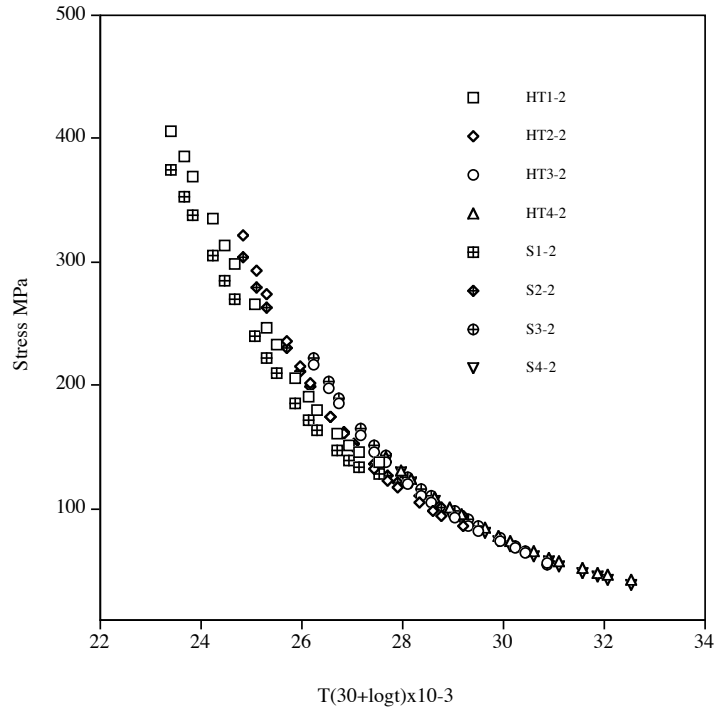


Figure 11 Stress vs. Larson-Miller Parameter with  $C=30$  for projected times to 1% creep

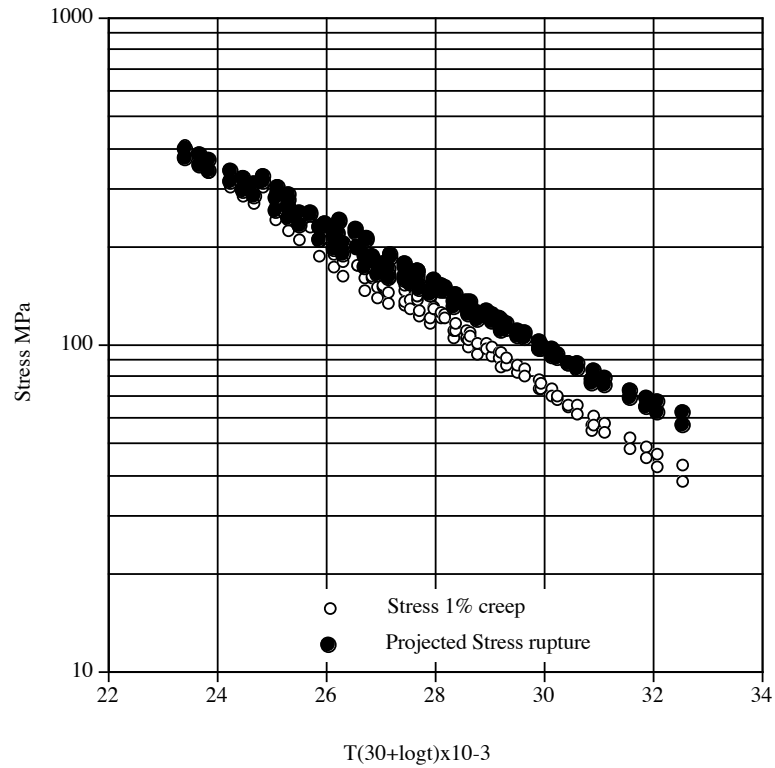


Figure 12 Stress vs. parameter plot for projected time to 1% creep and computed time to rupture using the Gill-Goldhoff correlation

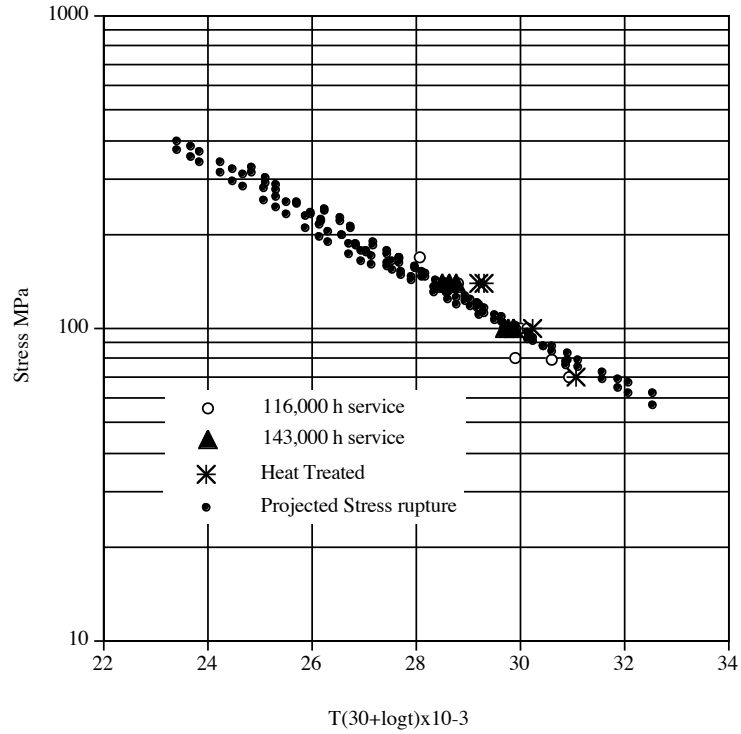


Figure 13 Stress vs. Parameter plot for projected time to rupture compared with actual long term data

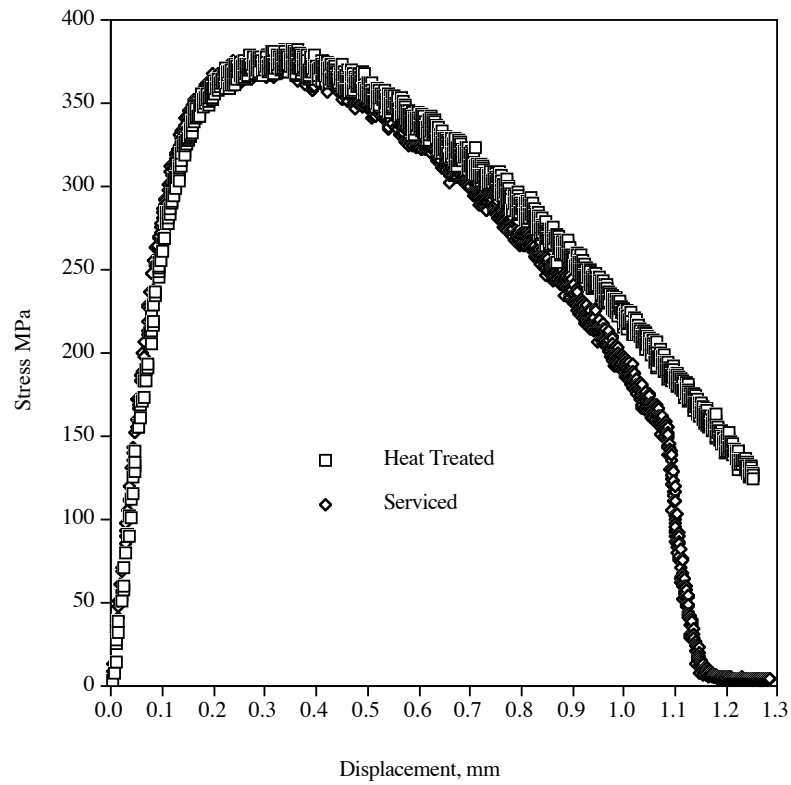


Figure 14 CDR tests at 600°C on serviced and heat treated T91