ISOCHRONOUS RELAXATION CURVES FOR TYPE 304 STAINLESS STEEL AFTER MONOTONIC AND CYCLIC STRAIN*

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ABSTRACT

Relaxation tests to 100 hr were performed on type 304 stainless steel in the temperature range 480 to 650°C and were used to develop isochronous relaxation curves. Behavior after monotonic and cyclic strain was compared. Relaxation differed only slightly as a consequence of the type of previous strain, provided that plastic flow preceded the relaxation period. We observed that the short-time relaxation behavior did not manifest strong heat-to-heat variation in creep strength.

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INTRODUCTION

Type 304 stainless steel is widely used as a structural material in fossil and nuclear power plants. The high thermal expansion coefficient, low thermal conductivity, and low yield strength of type 304 stainless steel, make it susceptible to thermal stresses which exceed its yield strength. At temperatures below 427°C (800°F), Section III of the ASME Boiler and Pressure Vessel Code provides criteria to protect structures from low-cycle fatigue and excessive ratchetting. Simplified elastic design methods are available for most applications, and relaxation data are not needed. Above 427°C relaxation of residual stresses by thermal creep is assumed possible. This relaxation produces creep strains which damage the material. For cyclic situations the ASME Code Case 1592 provides design criteria to protect structures from failure by creep-fatigue ratchetting mechanisms.¹ The designer has the option to perform a simple elastic analysis and use very conservative design limits or perform a more sophisticated inelastic analysis. If he chooses the latter option, the designer is allowed more generous design limits.² The ASME Code provides materials data which the designer can use to solve some problems requiring inelastic analyses. Isochronous stress-strain curves,³ for example, are

quite useful for problems which involve monotonic creep. However, no data are available for quick estimates of residual stresses when inelastic strain accumulates under relaxation conditions. For such problems isochronous relaxation curves, if they can be developed, could prove considerably valuable. The concept of isochronous relaxation curves is not a new one. Curves for 1/2 Cr-Mo-V steel were presented by Elder, Marriott, and Murphy in 1972⁴ and for 2 1/4 Cr-1 Mo steel by Ellis et al. in 1975.⁵ This report summarizes work on the subject at the Oak Ridge National Laboratory. Most data have been accumulated on a single heat of type 304 stainless steel used in a program sponsored by the Department of Energy. This program intends to develop high-temperature structural design methods and failure criteria.

MATERIAL AND TEST METHODS

Specimens were machined from a 25 mm plate of a reference heat of material⁶ (heat 9T2796) with the following ladle analysis: 0.048% C, 1.22% Mn, 0.028% P, 0.015% S, 0.048% Si, 18.6% Ni, 9.7% Cr, and 0.32% Mo. Most specimens were threaded-end bars with 6.35 mm gage section diameters. After machining specimens were reannealed for 0.5 hr at 1093°C in argon and rapidly cooled. In this condition the material had a 120 μ m average grain size, a Diamond Pyramid Hardness value near 125, and a room temperature yield near 176 MPa.

Monotonic tests were performed in the system shown schematically in Fig. 1. The specimen was placed in 44-kN electrohydraulic testing machine and heated in a resistance furnace. Temperatures were maintained to within $\pm 2^\circ\text{C}$

of the reported temperature throughout the test. For low stress tests, in which strains were small, an extensometer was attached to the center 25 mm of the reduced section. For high-stress tests, when strains exceeded 1%, the extensometer was attached to the shoulders. This produced an effective elastic gage length near 38 mm. The creep strain gage length was near 32 mm. The extensometer was an averaging type with Linear Variable Differential Transducers. The sensitivity was better than $\pm 10 \times 10^{-6}$ strain. All tests were performed in a servo-controlled mode using the extensometer feedback signal for control. Control was better than $\pm 25 \times 10^{-6}$ strain, although in several instances command signal fluctuations produced changes in the control strain, which exceeded 25×10^{-6} strain in the specimen.

Cyclic tests were performed in a system shown schematically in Fig. 2. Here the specimen was placed in a die-set gripping fixture mounted in a 110 kN electrohydraulic testing system. The specimen was heated by induction. Temperatures were maintained constant within $\pm 2^\circ\text{C}$ of the reported value. The extensometer was attached to one side of the specimen and had a sensitivity near 10×10^{-6} strain. Strains were controlled within $\pm 25 \times 10^{-6}$ strain.

RESULTS

Relaxation curves for a series of tests at different temperatures are compared in Fig. 3. All tests were started at nearly the same stress (approximately 210 MPa) and strain (approximately 7-9%), but the temperatures ranged from 482 to 649°C. Most of the relaxation occurred during the first ten hours of testing. Relaxation, however, increased dramatically as the temperature increased from 482 to 649°C. This, of course, implies that thermal activation is very important to the relaxation process in this temperature range. Another series of tests at different initial stresses are compared in Fig. 4. All tests were at 538°C, but the starting stresses ranged from near 80 to 275 MPa with corresponding starting strains from 0.5

to 15%. Tests with high starting stresses exhibited high initial relaxation rates. However, the curves converge very little after ten hours or so, possibly because of differences in the "hardness" of the material⁷ produced by the different plastic loading strains. Alternatively, tests with high starting stresses may have absorbed more creep strain while relaxing to a given stress and this creep strain produced creep hardening, manifested by low relaxation rates at lower stresses.

Isochronous relaxation curves conveniently represent the influence of the previous plastic strain on the relaxation response. Such curves are shown in Fig. 5 for the relaxation data at 538°C. To construct the curves the engineering stress data shown in Fig. 4 were converted to true stresses for 0, 1, 10, and 100 hr and plotted against true strain. The strain for each test was chosen so that the starting relaxation stress fell on a "reference" stress-strain curve (labelled 0 hr), which represented a monotonic tensile test at a strain rate near 6.7×10^{-5} /s. The relaxation strength clearly rises systematically with strain and the slope of each isochronous curve either equals or falls below that of the stress-strain curve. The slope decreases with increasing time or initial stress.

Isochronous curves for 482, 566, 593, and 649°C are shown in Figs. 6-9, respectively. As the temperature increases, relaxation increases, and the slope of the isochronous curve decreases. These isochronous relaxation curves complement the isochronous stress-strain curves derived from tensile tests and constant load creep data, and developed for use in design by Manjoine.⁸ The isochronous relaxation curves differ in several ways from the isochronous stress-strain curves. The most fundamental difference, of course, is that the isochronous stress-strain curves apply to load-controlled situations, whereas the isochronous relaxation curves apply to strain-controlled situations. For a given time and strain the curves also

differ in indicated stress level. As an example, in Fig. 5 the 100-hr isochronous curve based on creep data at 538°C can be compared to a curve based on relaxation data at the same temperature and time. The isochronous relaxation curve produces stresses substantially below the isochronous stress-strain curve, especially at high strains. Another feature unique to the isochronous relaxation curve is its relative insensitivity to heat-to-heat variations, at least for short-time applications. As an example, the relaxation strengths at 1, 10, and 100 hr for several heats are compared

• in Fig. 10 to the strengths predicted from the isochronous curves developed for heat 9T2796. Data sources include a compilation by Freeman and Voorhees⁹ (ASTM STP 187), Manjoine,¹⁰ and work at ORNL.¹¹ Most of the data fall within $\pm 10\%$ of the predictions. This scatter is small compared to the $\pm 20\%$ or more typical of the variation in creep strength from heat to heat.¹²

Isochronous relaxation curves also differ from isochronous creep curves in treatment of response after cyclic strains. No proven way exists to use the isochronous stress-strain curve to predict the relaxation of stress after a reversed plastic strain transient. However, such a prediction may easily be made from isochronous relaxation curves if one knows the peak stress in hysteresis loop. A method is shown schematically in Fig. 11. Here a hysteresis loop is centered about zero strain. The peak stress, σ_{max} , is translated parallel to the abscissa of the plot to the intersection with the zero time isochronous relaxation curve. The relaxed stress, σ_R , can then be estimated by reading downward from this point to the appropriate isochronous relaxation curve for the hold period in the cycle. This method assures that the relaxation curve for any hold period during repetitive strain-cycling is the same as the relaxation curve for the monotonic test. Data which support the validity of this assumption are shown in Figs. 12 and 13. Figure 12 shows data obtained from relaxation rests on cycled specimens of heat 9T2796 at 593°C. The relaxation strengths for 1, 10, and 100 hr are plotted against the

strain values characteristic of the monotonic tests for the same starting stress. Data show that cycling generally produces slightly lower strengths than do the monotonic tests, but the overall trend is similar. Figure 13 compares the relaxation strengths produced in cyclic tests on a number of heats to relaxation strengths predicted from monotonic tests on a single heat (9T2796). Data sources include Majumbar and Maiya,¹³ Conway et al.,¹⁴ Iida,¹⁵ and ORNL work.¹¹ In general the observed relaxation strengths fall within 90 to 100% of the predicted strengths. The observed and predicted values agree well, considering that heat-to-heat scatter in creep strength for type 304 stainless steel is about $\pm 20\%$.

Although the isochronous relaxation curves shown in this report represent both monotonic and cyclic loadings, they do not apply in a number of situations.

1. We know that very short-time relaxation behavior, say for the first ten minutes or so, is influenced by the plastic strain rate preceding the relaxation hold.^{11,12} This is shown schematically in Fig. 14. The lower plastic strain rates often produce lower relaxation rates even though the strain rate may not greatly affect stress-strain behavior. The isochronous curves shown in this paper generally reflect behavior of material strained at a rate near $6.7 \times 10^{-5}/s$. Some of the cyclic data, however, reflect the behavior of material strained at rates as high as $4.3 \times 10^{-3}/s$. Strain-rate differences may explain why short-time cyclic strengths fall below predictions based on monotonic data.

2. We have evidence that for reloading conditions which do not produce plastic strain before relaxation (i.e., elastic reloading), the relaxation rate decreases from one run to the next. Since the isochronous relaxation curves were constructed from test data from runs in which some plastic strain

had been introduced before each loading, the predicted relaxation may be greater than observed in situations in which only elastic reloading occurred.

3. The response in the stress transient "dip test" is not captured. In this loading condition, similar to that of the experiments of Solomon,¹⁶ plastic or creep strain is introduced at a high stress and the relaxation response is observed after a rapid load decrease. The behavior for this loading condition is shown schematically in Fig. 15 for several unloading increments. Generally, the short-time response shows less relaxation than predicted from isochronous relaxation curves. At some stress levels, in fact, the stress remains constant or even increases for a period which can last many hours. This, of course, reflects the influence of the "back stress" often associated with dislocation pile-ups or dislocation bowing and is related to the internal stress.¹⁷

DISCUSSION

In this report we have emphasized the features of an experimentally developed set of isochronous relaxation curves. These curves may be used in much the same way as the isochronous stress-strain curves now contained in the ASME Boiler and Pressure Vessel Code. However, we do not suggest that the isochronous relaxation curves substitute for properly formulated constitutive relationships and proper materials data. In fact, if the proper constitutive relationships are established, we should be able to predict relaxation behavior even if many of the complex histories for which the isochronous relaxation curves were developed do not apply. In this regard, a significant effort is underway in the U. S. and elsewhere to develop and verify unified constitutive equations, which can represent material behavior over a broad range of conditions. Examples are the work of Hart,¹⁸ Onat,¹⁹ Miller,²⁰ Swearingen and Rhode,²¹ and Gittus,²² to

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mention just a few. Many of these efforts center on the description of the creep rate as a function of stress, temperature, and one or more structure-dependent state variables. The determination of creep curves and relaxation curves from these models often requires the solution of two or more coupled differential equations which relate to flow rules and growth laws.

The data we report here have been processed into the type of information needed for constitutive equation development, for example, temperature, history, and strain rate as a function of stress. Results are currently being used to guide the development of constitutive equations needed for the U.S. breeder reactor program. We refer to the work of Robinson, Pugh, and Corum^{23,24} for more details. The scope of this paper, however, does not include a discussion of the implications of our data for constitutive equation development.

We do feel, however, that this paper should consider the use of isochronous relaxation curves to analyze creep-fatigue behavior. The bulk of the data on creep-fatigue are derived from tests which involve strain-controlled constant strain-rate cycling with relaxation hold periods at the hysteresis loop tips. Hold times are in the range 0.01 to 1 hr. Very few data are available for hold times beyond 1 hr. For design applications a need exists to predict relaxation for times in the range 10 to 1000 hr. From such data the creep "damage" is estimated and used to develop the conservative strain-fatigue curves needed for simplified elastic analyses.²⁵ Many investigators correlate their short-time relaxation data by means of an equation developed by Gittus²⁶ and later modified by Conway.²⁷ Examples may be seen in the work of Jaske and co-workers,²⁸ Iida and co-workers,¹⁵ Majumbar and Maiya,¹³ and Bui-Quoc et al.²⁹ Gittus developed his equation from a creep law of the form:

$$\dot{\epsilon} = A\sigma^n t^m,$$

where $\dot{\epsilon}$ is creep rate, σ is stress, t is time, and A , n , and m are material parameters which depend on temperature. Gittus showed that the relaxation behavior of Nimonic 80A springs could be best modelled by assuming that creep rate is linear with stress. That is, the stress exponent, n , is near unity. The original Gittus equation for relaxation was then developed into the form:

$$\frac{\sigma_0}{\sigma} = \exp(KEt^{m+1}) \quad (2)$$

Conway modified the equation by eliminating the starting stress and arrived at the equation:

$$\left| \frac{\dot{\sigma}}{\sigma} \right| = \frac{KEt^m}{n+1} \quad (3)$$

which plots linearly on log-log coordinates. Equation (3) can be easily integrated to produce either a relaxation curve or a measure of damage during the relaxation process. We have examined our data in terms of Eq. (3) and show some examples in Fig. 16. Generally we find that the equation provides a reasonably good fit. However, we point out that the equation assumes a stress exponent of unity, whereas if the relaxation results from dislocation creep or glide we would expect an exponential or power law ($n > 1$) dependency. If the Gittus-Conway model really applies, then we can only conclude that anelasticity, or some similar phenomenon, must play an important role in the relaxation process. Of course, other deformation processes, such as diffusion creep, also have a stress exponent of unity, but we think that the creep rates associated with these processes are too low to consider here. We expect that power law creep will eventually dominate the relaxation process, hence to extend Eq. (3) out of the range of available data is somewhat tenuous.

Relaxation behavior may also be predicted by the straightforward use of a creep law in conjunction with time-hardening or strain-hardening theory. Above 650°C power law creep in conjunction with time-hardening provides a good method to predict relaxation for type 304 stainless steel. This has been shown by Kennedy³⁰ and Manson and Zab.³¹ At 600°C and below, strain-hardening guides

relaxation predictions reasonably well for low starting stresses and for a single relaxation period. This has been shown by Pugh.²⁴ However, the fact that reloading into the plastic range, or reversal loading, produces relaxation behavior which differs little from relaxation behavior in the first loading implies that strain-hardening has only limited value in predicting relaxation behavior. We assume that behavior in cyclic relaxation reflects the influence of either anelasticity or dynamic recovery process.

The experimental isochronous relaxation curves constructed here make no assumptions about the deformation mechanisms or hardening laws and seem to apply, provided some plasticity precedes relaxation. We must point out, however, that the experimental data are limited to total testing times in the range 1 to 1000 hr. No one really knows whether the material will behave similarly in the 10,000 to 1,000,000-hr range. More work in this testing regime is necessary.

CONCLUSIONS

1. In the temperature range 480 to 650°C isochronous relaxation curves can be constructed from relaxation data by plotting the true stress versus the true strain. The strain is determined by locating the starting stress of the relaxation test on a reference stress-strain diagram established at high strain rates.

2. Isochronous relaxation curves should apply to strain-controlled situations while isochronous stress-strain diagrams should apply to stress-controlled conditions. Isochronous relaxation curves reflect lower stresses than do isochronous stress-strain curves at equivalent times.

3. Isochronous relaxation curves show less heat-to-heat variation than do isochronous stress-strain curves.

4. Isochronous relaxation curves may be used to approximately predict relaxation in cyclic-strain situations in which hold times are introduced at peak strains.

5. The use of isochronous relaxation curves should be limited to relatively short times, less than 1000 hr, and to situations in which relaxation is preceded by macroscopic plastic flow.

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