EFFECTS OF MICROSTRUCTURAL DEGRADATION ON CREEP LIFE PREDICTION OF 2-1/4 Cr - 1 Mo STEEL

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ABSTRACT

Accelerated aging at 630°C has been performed on 2-1/4 Cr - 1 Mo steel in order to simulate microstructures observed in boiler tubing after long-time service. Results of microscopy, creep tests and tensile tests on the simulated microstructures are reported.

KEYWORDS

Creep; high temperature fracture; 2-1/4 Cr - 1 Mo steel; microstructural degradation; accelerated aging; boiler tubing; life prediction.

INTRODUCTION

Due to the importance of component integrity in long-time, high-temperature service, particularly in the power generation industry, considerable work has been carried out on residual creep life studies and damage accumulation with the object of predicting creep lives and avoiding premature failures of components (Hart, 1976; Sidey, et al., 1979; Williams and Wilshire, 1977; Williams and Cane, 1979; Woodford, 1973).

Many empirical relationships have been developed to predict long-term creep behaviour by extrapolating short-term laboratory creep data (Conway, 1969). These parametric approaches implicitly assume that the microstructure of the material remains unchanged during the creep life and also they do not accommodate the changes which may result from fluctuations in the operating stress and temperature. It is known that the microstructure of low alloy ferritic steels continues to change throughout the service life (Murphy and Branch, 1971; Toft and Marsden, 1961; Williams and Wilshire, 1977) which invalidates the basic assumption of most of the parametric approaches to creep life prediction. Also, the recent development of fracture mechanism maps emphasizes the fact that the mechanisms of failure can change from one mode to another through changes in temperature, strain rate and stress (Ashby, et al., 1979). Clearly, a predictive approach should consider the fracture mechanism and the changes in the microstructure of the material during service in order to achieve reliable estimates of creep life.

Baker and Nutting (1959) have studied the changes in precipitate structure, morphology and distribution in 2-1/4 Cr - 1 Mo steel resulting from high temperature exposure. The process of microstructural degradation can be accelerated by overtemperature heat treatment which provides an effective way of investigating the effects of changes in the microstructure on the creep properties of materials operating in the creep range (Hale, 1973). This paper presents the results of accelerated aging experiments to produce simulated service microstructures.

MATERIALS AND EXPERIMENTAL PROCEDURE

The work was performed on commercial heats of annealed 2-1/4 Cr - 1 Mo steel whose compositions and properties conformed to ASTM A213-T22 alloy. Round tensile and creep specimens were machined of 3 mm diameter and 15 mm gauge length from stock or virgin material, as well as tangentially from a superheater tube with 85 000 hours of service exposure. The "stock" specimens were thermally aged at 630°C for various time periods to produce microstructures approximately simulating 25, 50, 75 and 100 x 10^3 hours of service exposure at 540°C (Table 1). These times were calculated on the basis of the activation energy for self-diffusion in iron.

TABLE 1 Accelerated Aging Time at 630°C

Time in Service at 540°C (h)	25 000	50 000	75 000	100 000
Equivalent Time at 630°C (h)	454	909	1 364	1 820

Tensile tests were performed at 625°C using a strain rate of 5 x $10^{-5}s^{-1}$. Creep tests were performed in air at 625°C using single-lever constant load creep machines. The creep data were collected at different nominal stresses between 50 MPa and 100 MPa. Creep deformation was measured using an "LVDT" transducer with a sensitivity of 2 µm. Carbon extraction replicas were prepared of the "service" and "simulated" microstructures to identify the carbide precipitates and monitor the changes in their structure, morphology and distribution. Chemical analysis of the carbides was carried out using energy spectroscopy analysis. The concentration of elements was calculated following the method of Cliff and Lorimer (1975).

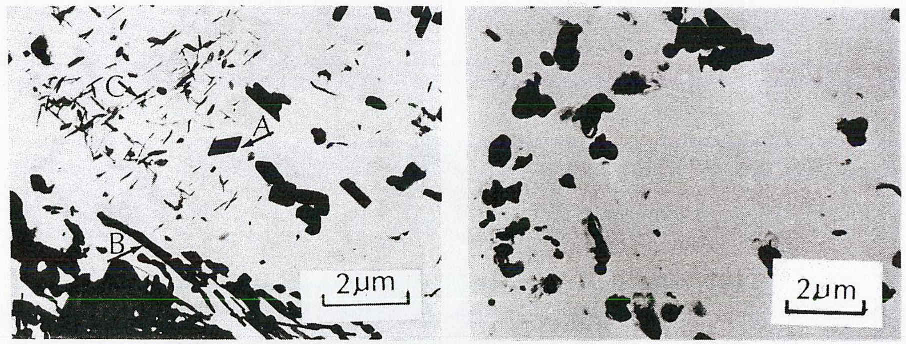
RESULTS

Optical Microscopy

Virgin material had a ferrite/pearlite microstructure, while the service material consisted mainly of spheroidized carbides in a ferrite matrix. Considerable spheroidization of the carbides could be detected during the early stages of the aging process in the "simulated" microstructures, but at the longer times any further changes were not resolvable.

Electron Microscopy

Figure la shows the general distribution of the precipitates in the "stock" material. The microstructure consists of "thick" precipitates in the form of plates "A" which occur near the pearlitic cluster "B". The same figure shows "acicular" precipitate morphology "C." Further observation of other areas of the specimen showed that the acicular carbides were predominant in the "stock" material. The service material (Fig. lb) possessed coarse precipitates with none of the fine acicular form. These micrographs indicate the considerable



(a) (b)

Fig. 1. Carbide precipitates in (a) stock material (b) service material after 85 000 hours.

microstructural degeneration resulting from long-term, high-temperature service.

Carbides in the stock and service materials were analyzed from selected area diffraction patterns. In the stock material, all particles analyzed had the cubic structures of M_6C and $M_{23}C_6$ and no trace of carbides with M_2C structure was found. In the service material, two carbide structures were found: M_6C cubic, and carbides containing >70 weight percent Mo. The latter could be either M_2C or MC.

Electron micrographs of the microstructures after 25, 50, 75 and 100 000 hours simulated service exposure are shown in Fig. 2. The 25 000 hour sample (Fig. 2a) shows the acicular carbide, although overall its volume fraction was less than in the stock alloy (Fig. la). Figures 2b through 2d show the general trend of continuous growth of carbides together with the disappearance of the acicular form with increasing aging time, the resulting effect being to increase the interparticle spacing. The carbides were mainly M_6C and $M_{23}C_6$, with a Mo rich carbide present only in the 100 x 10^3 hour microstructure. The electron microscopic investigation indicated that considerable changes had taken place in the size, distribution and morphology of the precipitates due to accelerated aging.

Composition of the Precipitates

Chemical analyses of the precipitates were determined by x-ray energy spectroscopy techniques (Fig. 3). It can be seen that all the service simulated microstructures had one stable carbide ($M_{23}C_6$) that showed only slight changes in the relative amounts of Cr to Fe. On the other hand, most of the carbides that were identified in the service material were typical of M_6C, with the exception of precipitates that contained more than 70 percent Mo by weight whose structure was not conclusively identified by selected area diffraction. The figure shows a general trend that as the aging time was increased, the carbides became enriched in molybdenum. The observations indicate that accelerated aging had produced significant changes in the chemical composition of the precipitates. The changes are mainly related to a reduction of Fe content of the carbides and an increase in Mo concentration, as observed in the service material.

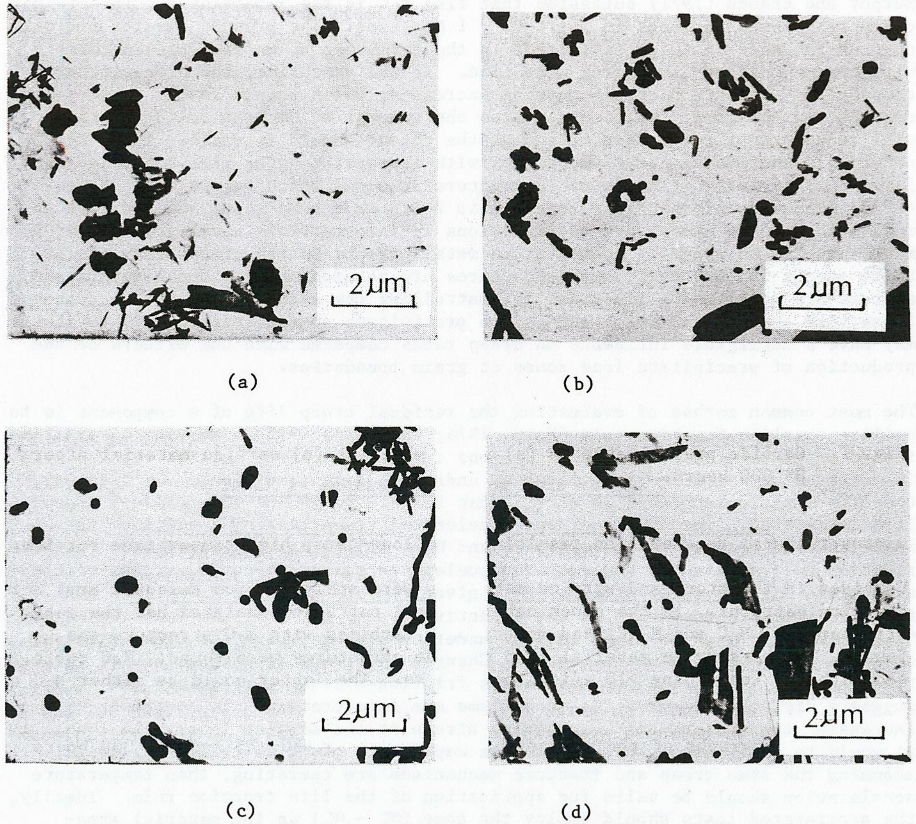


Fig. 2. Carbide precipitates in samples aged at 630°C to simulate service times of (a) 25 000 (b) 50 000 (c) 75 000 (d) 100 000 hours.

Creep and Hot Tensile Properties of Simulated Microstructures

Figure 4 shows that major reductions in yield and tensile strengths resulted from the initial stages of accelerated aging and minimum values were reached in the 50 000 hour samples. On the other hand, the ductility, as measured by the reduction of area, peaked at 50 000 hours then remained almost constant. From the figure, it can be seen that the data from the service material closely resembles that for a simulated microstructure of 100 000 hours. The ductility for the service material was 68 percent compared with 85 percent for the simulated microstructures. This difference may be a result of the creep damage which existed in the service material.

Accelerated creep tests were performed using the over-temperature approach recommended by Hart (1976). Figure 5 shows that the time to rupture decreased as the aging time was increased. Creep data from the service material were superimposed on the figure. It can be seen that the data resemble those from specimens with more than 75 000 hours of simulated service.

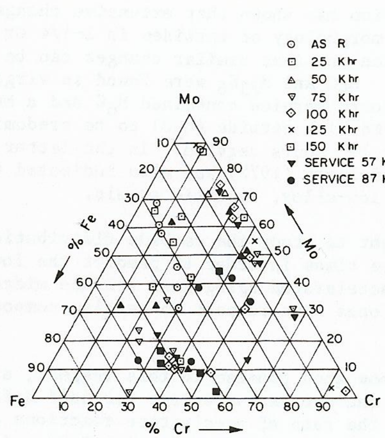


Fig. 3. Compositions of carbides in terms of Fe, Cr and Mo in service, stock and accelerated aged materials.

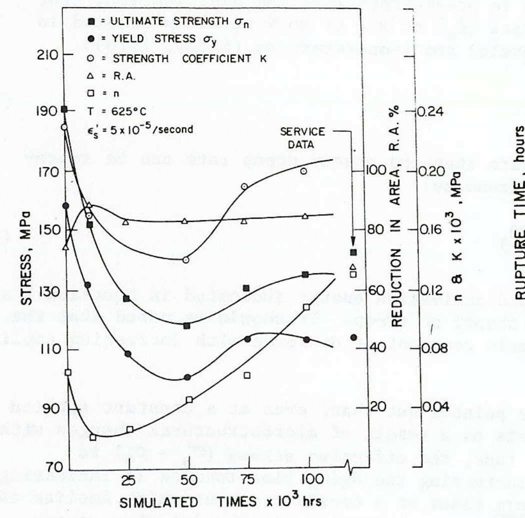


Fig. 4. Tensile properties at 625°C.

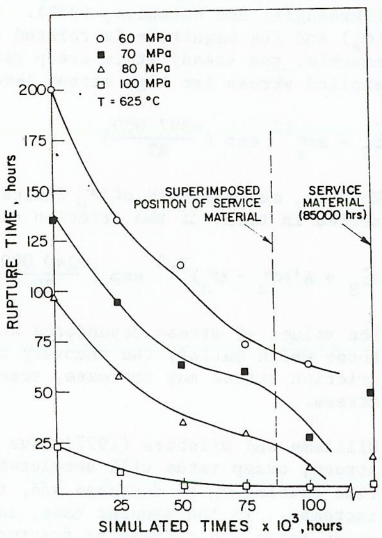


Fig. 5. Creep rupture properties at 625°C

DISCUSSION

The present investigation has shown that extensive changes take place in the composition, size and morphology of carbides in 2-1/4 Cr - 1 Mo steel during high-temperature service and that similar changes can be produced by accelerated aging of new material. M_6C and $M_{23}C_6$ were found in virgin material, whereas material with 85 000 hours service contained M_6C and a Mo rich carbide. Collins (1978) has reported a Mo rich carbide (M_2C) to be predominant in a 1/2 Cr - 1/2 Mo - 1/4 V steel after 10^5 hours service. In the latter, VC had been the major initial precipitate. Sellars (1972) has also indicated that Mo_2C is the most stable carbide in the low-alloy, ferritic steels.

Clearly, it is important to study the carbide distribution in specimens with different service exposure times in order to predict the long-term properties of ferritic steels, but accelerated aging must produce microstructures which are equivalent to service ones with respect to carbide composition, distribution and morphology.

The present results show good promise in this respect, although the accelerated aging times, based on the activation energy for self-diffusion in iron (Q_D), seem to underestimate the rate of precipitate reactions in service conditions. This could be due to inappropriateness of using Q_D for iron, or to service temperatures being higher than estimated, or to enhancement of reaction rates in service material due to the presence of the operating stress. However, the general trend towards the production of Mo rich precipitates suggests that accelerated aging treatments can be developed to simulate service microstructures.

The high stress dependencies and large activation energies associated with the creep behaviour of many precipitation-hardened alloys have been reconciled with the recovery theory of creep by introducing the concept of a friction stress (σ_o) (Threadgill and Wilshire, 1972). This friction stress opposes the applied stress (σ_a) and its magnitude is related to precipitate size and distribution. For example, the steady state creep rate ($\dot{\varepsilon}_s$) of a 1 Cr Mo V steel is related to applied stress (at high stress levels) and temperature by (Sidey, 1976):

$$\dot{\varepsilon}_s = A\sigma_a^{17} \exp\left(\frac{-397\ 000}{RT}\right) \quad (1)$$

However, measurements of σ_o indicate that the steady creep rate can be represented in terms of the friction stress by:

$$\dot{\varepsilon}_s = A'(\sigma_a - \sigma_o)^{3.5} \exp\left(\frac{-240\ 000}{RT}\right) \quad (2)$$

The values of stress dependence and activation energy indicated in Equation 2 are those which satisfy the recovery theory of creep. It should be noted that the friction stress may increase, remain constant or decrease with increasing applied stress.

Williams and Wilshire (1977) have pointed out that, even at a constant applied stress, creep rates will accelerate as a result of microstructural changes with time causing σ_o to decrease and, thus, the effective stress ($\sigma_a - \sigma_o$) to increase. In the present case, increasing the aging time results in increasing creep rates and decreasing fracture times at a constant stress which implies that the microstructural changes are leading to reductions in the friction stress. In terms of predicting long-term behaviour from short-term creep tests, it suggests that existing extrapolation techniques will overestimate lifetimes due to their inability to consider the progressive deterioration of creep resistance of these materials resulting from microstructural degradation.

Murphy and Branch (1971) suggested that fine M_2C is the important precipitate for creep resistance purposes in 2-1/4 Cr - 1 Mo steel. The present results indicate that an increase in aging time leads to the formation of Mo rich precipitates with the Mo content increasing with time. At the same time, the precipitates coarsen and the interparticle spacing increases, which should result in a decrease in the creep resistance. Also the removal of Mo from solid-solution will reduce creep resistance, although the effect should be small. Thus, the observed reduction in creep properties with increasing aging time can be correlated qualitatively with the microstructural changes which take place. Hale (1973) measured interparticle spacings in 2-1/4 Cr - 1 Mo steel and was able to relate changes in creep rate to variations in interparticle spacing. However a major problem in applying quantitative metallography to the creep behaviour of these steels is that their microstructures are heterogeneous. Local events and effects are going to be important in controlling the slow creep rates encountered in service and, for example, changes in precipitate characteristics within grains may have a negligible influence on creep rates compared with the effects of the production of precipitate free zones at grain boundaries.

The most common method of evaluating the residual creep life of a component is to utilize the life fraction rule (Hart, 1976; Woodford, 1973). Service material is tested under accelerated creep conditions in the laboratory and its rupture life is compared with that for new material under the same conditions. In this way, the life fraction expended in service can be determined and the residual creep life calculated. The choice of the accelerated creep testing conditions is important and work by Woodford (1973) and Hart (1976) has indicated that the life fraction rule applies to temperature-accelerated tests but not to stress-accelerated tests for low alloy steels. At a given temperature, the creep rate and fracture time are dependent on the effective stress ($\sigma_a - \sigma_o$) so that increasing σ_a is likely to lead to creep testing under conditions where the creep rate possesses a different dependence on σ_a. Changes in stress dependence can be related to changes in the applied stress versus friction-stress relationship and this suggests different deformation mechanisms are in operation. In contrast, by increasing temperature and maintaining stress at the service level, the change in σ_o would be small since the temperature dependence of σ_o is relatively small. Assuming the same creep and fracture mechanisms are operating, then temperature acceleration should be valid for application of the life fraction rule. Ideally, the accelerated tests should employ the same ($\sigma_a - \sigma_o$) as the material experiences in service. The laboratory determination of the friction stress is not feasible at the very low creep rates associated with service operation but it has recently been suggested (Evans and Harrison, 1979) that σ_o can be found empirically. By determining friction stress values for a range of service simulated microstructures it should be possible to evaluate residual creep lives of service material along the lines proposed by Williams and Wilshire (1977).

CONCLUSIONS

During long-time service under creep conditions, 2-1/4 Cr - 1 Mo steel undergoes continuous microstructural changes in terms of carbide compositions, sizes and morphologies.

By employing accelerated aging techniques, laboratory samples have been produced with reasonably similar microstructures to long-time service material.

Tensile and creep tests on service simulated microstructures at 625°C showed that these properties decreased with increasing aging time. Since in service the alloy undergoes similar continuous changes in microstructure, it is likely that the basic creep resistance of the material is also continuously decreasing. These effects can be related to the increase in interparticle spacing which is the net result of the complex, continuous carbide reactions taking place.

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