

EFFECT OF HEAT TREATMENT AT 1150°C ON CREEP-RUPTURE
PROPERTIES OF ALLOY FA-180

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ABSTRACT

The alloy FA-180, with a composition of Fe-28Al-5Cr-0.5Nb-0.8Mo-0.025Zr-0.05C-0.005B (at.%), is of interest because of its improved creep-rupture resistance when compared to alloy FA-129 (Fe-28Al-5Cr-0.5Nb-0.2C). At a temperature of 593°C and under a stress of 207 MPa, the creep-rupture life of FA-129 heat treated for 1 h at 750°C is about 20 h while the FA-180 alloy lasts approximately 100 h. Heat treatment at 1150°C has been shown to further improve the creep life of FA-180 and creep-rupture lives of approximately 2000 h have been attained. This strengthening was attributed to the presence of fine matrix and grain boundary Zr-rich MC precipitates that were produced by the heat treatment. The current study continues our investigation of the effect of heat treatment at 1150°C on the improvement of creep-rupture life in alloy FA-180. As part of our effort to understand the strengthening mechanisms involved with heat treatment at 1150°C, transmission electron microscopy was used to correlate the microstructure with the improved creep resistance. Results indicate that heat treatment at 1150°C for 1 h, followed by rapid quenching in water or mineral oil, produces even further improvements in the creep-rupture life of this alloy. A specimen being tested at 593°C and 207 MPa was stopped after over 6000 h of life, while another specimen lasted over 1600 h at 650°C and 241 MPa. The microstructure of the oil-quenched specimen contained many dislocation loops which were not present in the air-cooled specimens. These loops pinned dislocations during creep testing at temperatures of 593-700°C, resulting in a stabilized deformation microstructure and increased creep-rupture strength.

INTRODUCTION

Past studies have shown that binary Fe₃Al possesses low creep-rupture strength compared to many other alloys, with creep-rupture lives of less than 5 h being reported for tests conducted at 593°C and 207 MPa.^{1,2} The combination of poor creep resistance and low room-temperature tensile ductility due to a susceptibility to environmentally-induced dynamic hydrogen embrittlement³⁻⁵ has limited use of these alloys for structural applications despite their generally excellent corrosion behavior.⁶ With regard to the ductility problem, alloy development efforts have produced significant improvements, with ductilities of 10-20% and tensile yield strengths as high as 500 MPa being reported.^{7,8} Likewise, initial improvements in creep resistance have been realized through small additions of Mo, Nb, and Zr.^{1,9-13}

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In recent years, further creep strengthening has been produced by using heat treatments to control the microstructure.^{14,15} As shown in Fig. 1, for an Fe-28Al-5Cr (at.%) alloy containing additions of Mo, Nb, Zr, C, and B (designated alloy FA-180), a 1-h 1150°C heat treatment produced creep lives of over 2000 h for tests conducted at 593°C and 207 MPa. Especially interesting is the sharp dependence of creep strength on heat treating temperature, which is illustrated by the data in Fig. 1. In earlier studies, transmission electron microscopy revealed the presence of fine precipitates which appeared to pin dislocations.¹⁴ This, together with an activation energy for creep of approximately 150 kcal/mole (a value which is about twice that obtained earlier¹ for the binary alloy heat treated at 750°C) and high creep exponents of 7-12 (ref. 15), indicated that the observed strengthening was being produced by a precipitation mechanism. The general conclusion was that the 1150°C heat treatment resulted in the dissolution of coarse particles remaining from the melting and casting process, and then reprecipitation of finer Zr-based precipitates during cooling or the early stages of creep produced the strengthening.

The current research effort is focused on reaching a better understanding of the relationship between microstructure and strengthening mechanism(s) for this alloy and heat treatment. This paper summarizes those efforts.

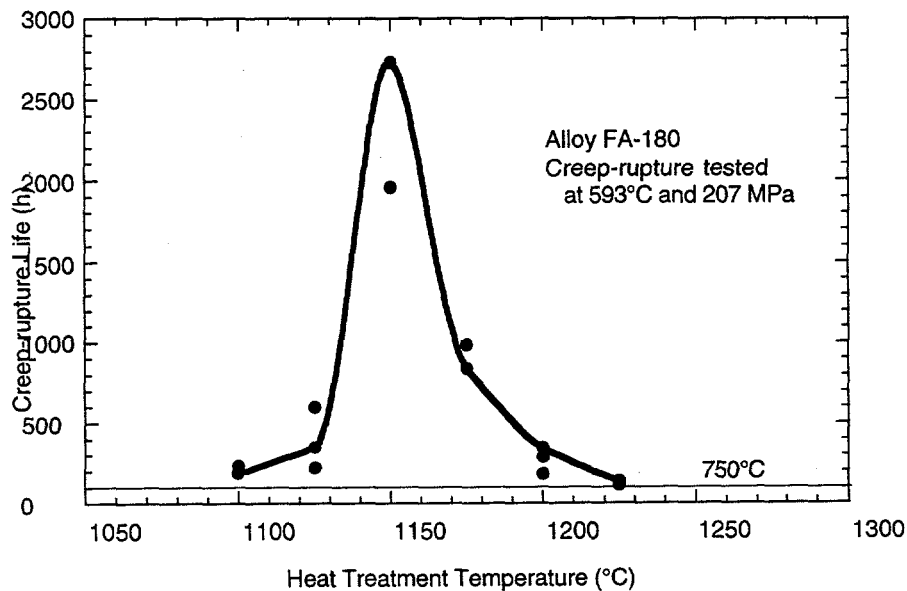


Fig. 1. Creep-rupture life as a function of heat treatment temperature (for 1 h) for tests conducted on alloy FA-180 at 593°C and 207 MPa.

EXPERIMENTAL PROCEDURES

The alloy composition used in this study was Fe-28Al-5Cr (at.%) with 0.5% Nb, 0.8% Mo, 0.025% Zr, 0.05% C, and 0.005% B (Oak Ridge National Laboratory designation FA-180). It was prepared by arc-melting and drop-casting into a chilled copper mold. Fabrication to 0.8-mm-thick sheet was accomplished by hot-rolling, beginning at 1000°C and finishing at 600-650°C. After a stress relief heat treatment of 1 h at 700°C, flat tensile specimens (0.8 x 3.18 x 12.7 mm) were mechanically punched from the rolled sheet. Before creep-rupture testing, specimens were further annealed in air for 1 h at 1150°C and then were either air cooled to room temperature or were quenched in oil or water.

Creep-rupture tests were performed in air at temperatures between 593 and 750°C under stresses of 138 to 276 MPa (20-40 ksi). In order to obtain creep exponents and activation energies, minimum creep rates (MCR) were measured as the slope of the linear portion of the test curve and the data were plotted to a power-law equation. The results for quenched specimens were compared to earlier published data on air-cooled specimens.¹⁵

Optical metallography and scanning electron microscopy (SEM) were used to study the microstructures and fracture modes. Analytical electron microscopy (AEM) using either a Philips CM30 (300 kV) or a CM12 [120 kV, with ultra-thin-window x-ray energy dispersive spectrometry (XEDS) detector] electron microscope was performed on samples cut from the gage portion of selected test specimens.

RESULTS AND DISCUSSION

Creep-rupture tests were conducted on specimens cooled by different methods from the 1150°C heat treatment temperature and the data are presented in Table I. The creep-rupture lives of the rapidly cooled specimens are compared in Fig. 2 with the life of a specimen that had been cooled in air from the 1150°C heat treatment temperature and then tested at 593°C. The more rapidly cooled specimens (oil or water quenched) exhibited the best resistance to creep. Even though the air-cooled specimen had exhibited a very good rupture life of approximately 1959 h when tested at 593°C and 207 MPa, a specimen that had been quenched in oil showed no signs of rupturing (no increase in the very low, steady-state creep rate) after 6480 h, at which time the test was stopped. Likewise, the water quenched specimen that was tested at 650°C under a stress of 241 MPa ruptured after 1637 h, while another specimen that had been cooled more slowly in air and tested at the same conditions lasted less than 3 h. The strengthening mechanism, therefore, appears to be different for quenched versus air

Table I. Creep-Rupture Data for Alloy FA-180 as a Function of the Method Used for Cooling from the 1150°C Heat Treatment

Method of Cooling	Creep-rupture Test Conditions	Life (h)	Elongation (%)	MCR (/s)
air	593°C, 207 MPa	1959	10	8.3×10^{-7}
oil quench	593°C, 207 MPa	>6480	5	2.8×10^{-8}
air	650°C, 241 MPa	2.7	42	2.2×10^{-3}
water quench	650°C, 241 MPa	1637	9	2.2×10^{-7}

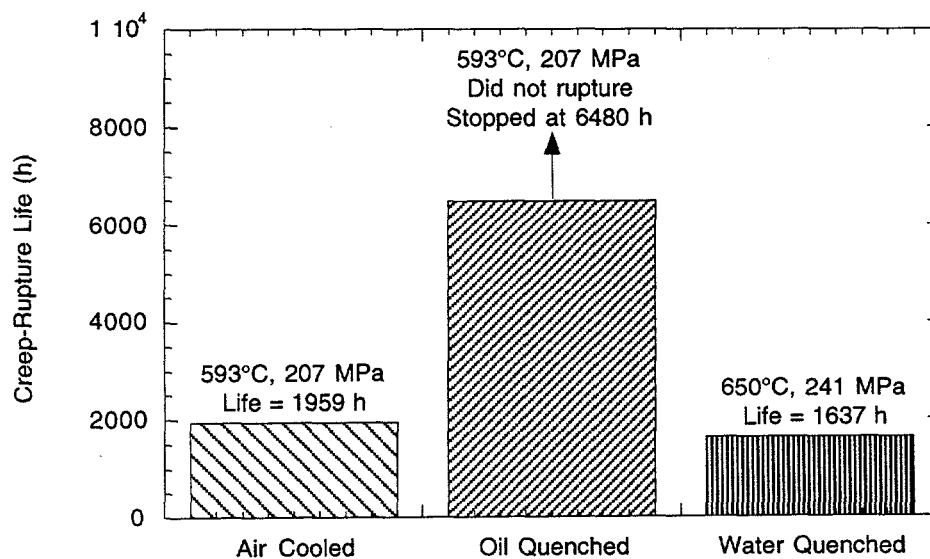


Fig. 2. Creep-rupture life of air-cooled versus quenched specimens of alloy FA-180.

cooled specimens, although the quenching medium itself does not appear to be the cause of that effect.

Creep tests were conducted as a function of temperature and stress in order to determine if the activation energies for creep (Q) and the creep exponents (n) were the same in both the air-cooled and oil-quenched conditions. The results are shown in Figs. 3 and 4. At a stress of 207 MPa (30 ksi), the activation energy for creep in the oil-quenched specimens was determined to be 76.6 kcal/mole, compared to a Q of 141.1 kcal/mole for air-cooled specimens. (The value of Q reported here for the air cooled specimens is slightly different from that reported earlier¹⁵ because it was derived from more data points.) This significant difference in activation energy suggests that a

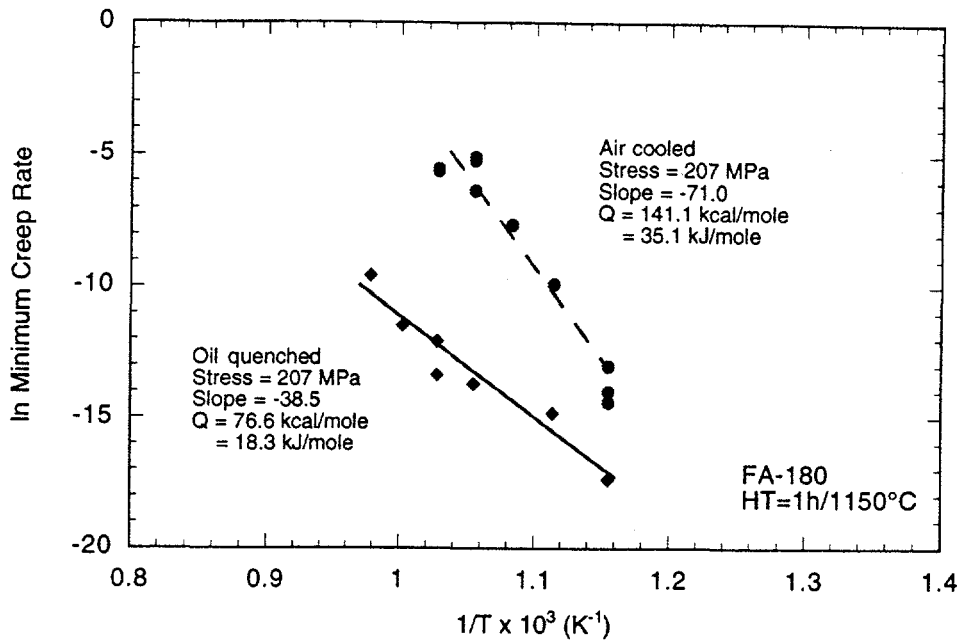


Fig. 3. Minimum creep rate versus temperature data for air cooled versus oil quenched specimens of alloy FA-180 heat treated at 1150°C. All tests were conducted at a stress of 207 MPa (30 ksi).

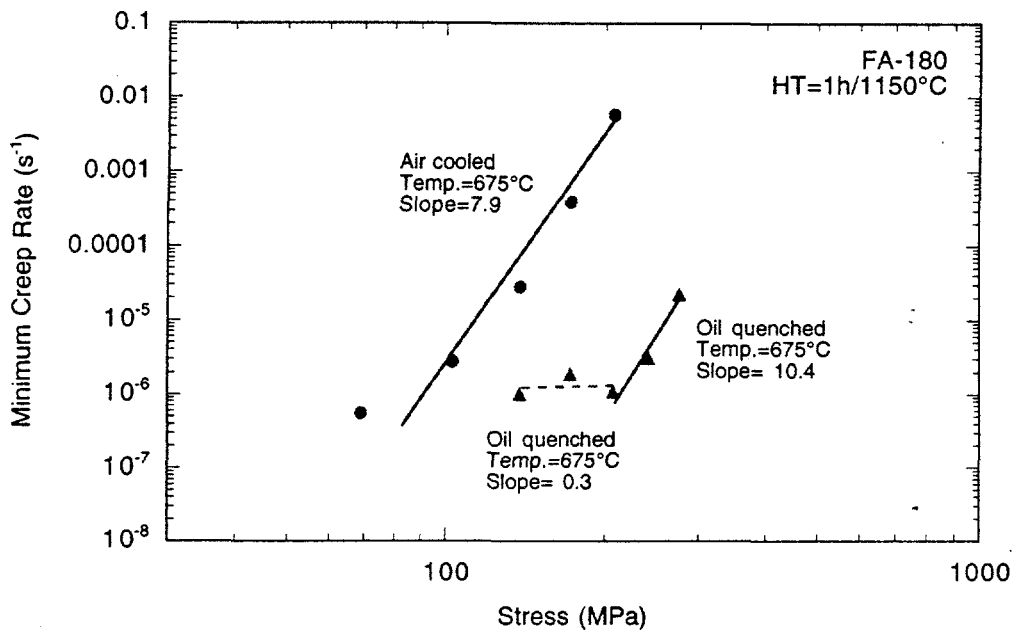


Fig. 4. Minimum creep rate versus stress data for air cooled versus oil quenched specimens of alloy FA-180 heat treated at 1150°C. All tests were conducted at 675°C.

different rate-controlling mechanism is active in the oil-quenched specimens. A plot of minimum creep rate versus stress (Fig. 4) shows a distinct break at approximately 200 MPa, with the creep exponent increasing at higher stresses. This behavior is indicative of a change in the rate-controlling creep mechanism with stress and is common in the data for many precipitate-hardened alloys.^{16,17} The creep exponent of 10.4 shown in Fig. 4 for the oil-quenched specimens and the creep exponent for the air cooled specimens, which was earlier calculated to be 7.9 (ref. 15), are both indicative of alloys which have been hardened by precipitates or some other dislocation-pinning mechanism.^{18,19}

Transmission electron microscopy was used to study the microstructure of the oil-quenched specimen that had been tested for 6480 h at 593°C. The micrograph in Fig. 5 shows that the creep-tested specimen contained networks of two-fold dislocations and many dislocation loops. At a higher magnification (Fig. 6), a few fine precipitates were also visible. Closer examination of the loops (Fig. 7) indicated that they were square or rectangular in shape and were restricted to the orthogonal habit planes (either $\langle 110 \rangle$ or $\langle 100 \rangle$). Additionally, the dislocation segments in the loops appeared to be single rather than 2-fold and the four segments of each loop were of the same Burgers vector. For comparison, Fig. 8 shows the microstructure of a specimen in the as-heat-treated-and-oil-quenched (untested) condition. The dominant features are the B2 ordered domains and many black dots visible by black-white strain contrast imaging. In this case, by rotating the specimen through several diffracting conditions, most of the black dots visible in the microstructure in Fig. 8 were identified as vacancy dislocation loops, not precipitates. It therefore appears that these fine loops were created in the microstructure as a result of the rapid quench from the 1150°C annealing temperature. This is in contrast to the results from air-cooled specimens reported earlier in which fine Zr-based precipitates that formed during cooling were found to provide some measure of strengthening during creep.¹⁴ Since it is known that vacancies are created in the Fe-Al system by quenching from high temperatures, especially for the B2 structure,^{20,21} the identification of these dislocation loops as being vacancy loops is not unreasonable. Vacancy loop nature would also be consistent with their growth during further heat treatment or creep testing. During creep-rupture testing at the temperatures used in this study, the loops provided strengthening by pinning dislocations and grew in size in the process (as indicated by the larger loop size in the as-tested specimen, Fig. 5).

CONCLUSIONS

In earlier studies the creep-rupture strength of Fe₃Al-based alloy FA-180 (Fe-28Al-5Cr-0.5Nb-0.8Mo-0.025Zr-0.05C-0.005B, at.%) was shown to be improved

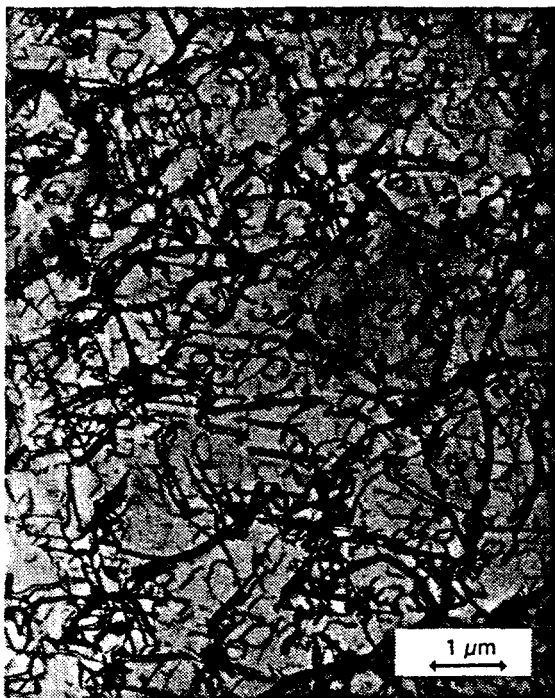


Fig. 5. TEM micrograph of FA-180 oil-quenched from a heat treatment at 1150°C and creep tested at 593°C for 6480 h.

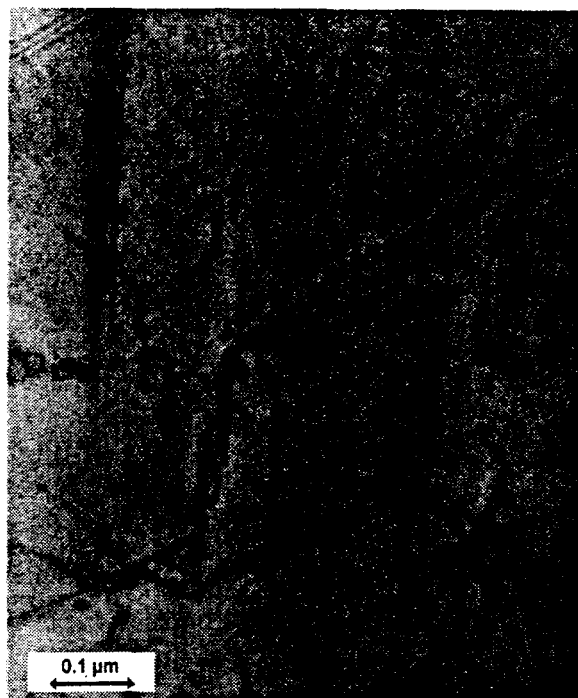


Fig. 6. Higher magnification of Fig. 5 showing presence of fine precipitates.



Fig. 7. Higher magnification TEM micrograph of dislocation loops visible in Fig. 5.

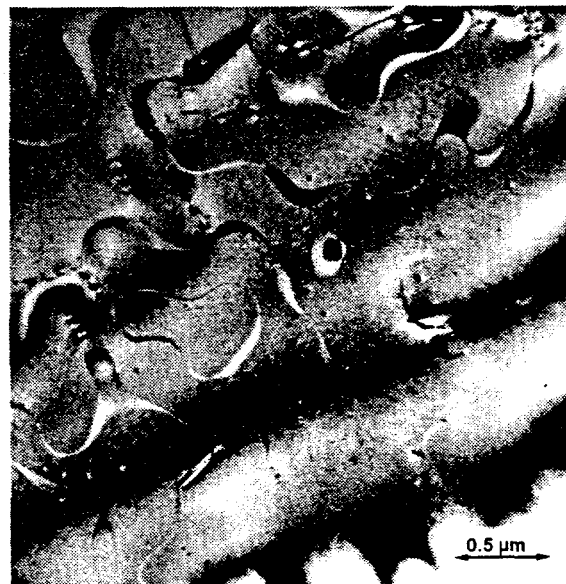


Fig. 8. TEM micrograph of alloy FA-180 showing B2 domains and fine dislocation loops (black dots) produced by oil-quenching from a heat treatment at 1150°C.

significantly by heat treating for 1 h at 1150°C. This strengthening was attributed to the dissolution at 1150°C of coarse particles left over from the melting and casting process and reprecipitation during cooling or during testing at 593°C of fine Zr-based matrix and grain boundary precipitates. In this study, creep-rupture tests were conducted on specimens of FA-180 that had been cooled quickly (oil or water quenched) from the 1150°C heat treatment. The results were compared to the previous data on specimens that were cooled more slowly in air. The more rapidly cooled specimens exhibited the best resistance to creep. This additional strengthening was attributed to the formation of dislocation loops as a result of the rapid quench. These loops pinned dislocations and grew slowly during testing at temperatures of 593-700°C, resulting in a stabilized deformation microstructure.

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