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August 1999

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CREEP OF NICKEL BASE ALLOYS IN HIGH TEMPERATURE WATER

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Abstract

Creep tests were performed to compare the creep behavior of commercial nickel-base alloys as a function of stress, temperature, and the environment. Alloy 600 (nominal and low carbon) and the precipitation strengthened Alloys 625 and X-750 in the AH and HTH conditions were tested at constant load in deaerated primary water containing 40-60cc/kg hydrogen. The stress dependence of Alloy 600 in the mill-annealed (MA) condition was obtained at 337°C and 360°C. The stress exponent was determined to be between 3 and 9. The activation energy of creep was 64 kcal/mole. Both results support a dislocation climb controlled mechanism of creep in commercial Alloy 600. The creep results were compared to the known stress corrosion cracking (SCC) performance of these alloys. The creep rates of the low carbon (LC) alloy are higher on average than those with nominal carbon level. Intergranular (IG) cracking occurred only in the X-750 AH alloy and the precipitation treated LC A600 alloy. These results support earlier work that showed that low carbon alloys are more susceptible to creep and IG cracking than are high carbon alloys. However, these results also show a smaller influence of a water environment on the creep rate of commercial, creep-resistant alloys compared to high purity alloys.

I. Introduction

Alloy 600 has been used as the tubing material in steam generators of pressurized water reactors for over 30 years. However, it is susceptible to intergranular stress corrosion cracking (IGSCC) on both the primary and secondary sides of the steam generator. Precipitation hardened nickel-base alloys and austenitic stainless steels are commonly used in core components, and these also exhibit IGSCC. Although numerous studies have dealt with this phenomenon the mechanism of IGSCC is still undetermined. Several potential mechanisms that may be responsible for the observed IGSCC have been suggested, but none of them seem to fully explain all of the observations. One factor that has not been studied extensively is the role of creep in IGSCC. Although operating temperatures are relatively low on a homologous temperature scale, dislocation creep of the alloy at temperatures as low as 0.3 Tm can be expected based on the deformation-mechanism map for a Ni-20Cr alloy. Significant creep rates were obtained in a study using high purity alloys. Increased chromium and carbon were found to improve the creep and IGSCC cracking resistance. A study using high purity Ni-16Cr-9Fe also showed that the primary water environment caused an increase by a factor of 5 to 10 in the steady state creep rate of the alloy in 360°C compared to an inert argon environment. Creep is important not only for its intrinsic effect on the material behavior, but also for its effect on the integrity of the oxide film. For this reason, there is significant interest in determining the creep behavior of nickel-base alloys in nuclear reactor environments.

This study focuses on the effect of stress, temperature, environment, and alloy composition on creep behavior of commercial Alloy 600. The stress dependence of the steady-state creep rate and the apparent activation energy for Alloy 600 in the mill annealed (MA) condition were obtained from constant load tests at temperatures between 337°C and 360°C in hydrogen deaerated primary water. Based on the results, the creep mechanism and the correlation between creep and IGSCC are discussed.

II. Experimental Procedure

The materials used in this study were commercial Alloy 600, low carbon Alloy A600 LC, and the precipitation strengthened Alloys 625 and X-750. The chemical compositions and heat treatment conditions of each alloy are given in Tables I and II, respectively. Alloy 600 was mill-annealed (MA) at 1100°C and cooled in air, resulting in discrete carbides precipitated on grain boundaries and in the matrix. Two different heat treatment conditions were used for A600 LC. Solution annealing (A600 LC-SA) was done to remove carbides from grain boundaries, and a precipitation treatment (A600 LC-PT) at 704°C for 10 hrs was used to precipitate intergranular (IG) carbides. No carbides were found on A600 LC-SA, while continuous IG carbides were found in A600 LC-PT. Alloy X-750 was used in AH and HTH conditions as described in Table II.

The creep apparatus consists of a four liter autoclave, a loading system, and a water circulation system as shown in Figure 1. Constant load tensile tests were conducted on round bar samples with a gage length and diameter of 25.4 mm and 2.25 mm, respectively. Experiments were conducted in a multi-sample autoclave system in which three samples can be loaded independently and in parallel. Load was applied to specimens by means of a lever arm via a pull rod. The amount of creep strain was measured by an external Linear Variable Differential Transformer (LVDT) attached to the pull rod. In some tests an internal LVDT attached to the internal load frame leg in the autoclave provided simultaneous, in-situ measurements of sample displacement. Fluctuation in room temperature
The stress exponent determined from the external LVDTs matches the values from the literature quite well. It should also be noted from this figure that data from early experiments (1966-1984) show much higher creep rates than from more recent experiments. In particular, the recent data of Leclercq and Vaillant, reported in Reference 14, agrees much better in magnitude with our data than do any of the early data. Furthermore, note that the creep rates of the high purity alloys tested by Angeliu and Was lie above this data but below the early works on a commercial Alloy 600. All of these results point to the early data as being significant over-estimates of the creep rate of commercial Alloy 600. All of these results point to the creep rates deduced from the early data as being significantly over-estimates of the creep rate of commercial Alloy 600.

**Effect of alloy on creep rate**

The effects of heat treatment and alloy composition are shown in Figures 6 and 7. The creep rates were plotted as a function of true stress to allow comparison among the different alloys that have different yield strengths and work hardening behaviors, and hence different reductions in area at a given load. Figure 6 shows the creep rates of Alloy 600 LC compared to those for Alloy 600 MA in 337°C deaerated primary water. The data show only a very small increase if any. The same is true at 360°C shown in Figure 7. The effect of carbide precipitation in the LC alloys is shown in Figures 6 and 7 by comparing the results from the PT and SA conditions. There is no discernable difference. While the data on high purity alloys show an increase in creep rate at low carbon levels, the carbon level of the A600 LC alloy is well above that at which increased creep rates were observed. However, it is evident from the creep rates of the precipitation-hardened alloys at much higher stresses in Figure 7, that they are indeed more creep-resistant.

![Figure 1. Schematic of multiple sample constant loading apparatus](image1)

![Figure 2. Examples of creep strain vs. time for A600 MA in 360°C primary water (a) at 85 ksi and (b) a step-load test](image2)