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Tensile cracking of a chromia layer on a stainless steel during thermal cycling with hold periods

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Abstract

This study has used thermogravimetric, acoustic emission and finite-element modelling techniques to examine the tensile cracking of a chromia layer on a 20Cr25Ni steel during thermal cycling from an oxidation temperature of 900 °C. It is found that the process is highly sensitive to the temperature amplitude of the thermal cycle and that hold periods at the bottom temperature result in enhanced oxide cracking whereas hold periods at the peak temperature can inhibit the onset of cracking. For standard saw-tooth cycles, the finite-element analysis shows that these effects can be explained by a creep hysteresis effect. This arises from stress relaxation during the cooling half-cycle, which results in the development of in-plane tensile stresses within the oxide layer on the return to temperature. Low-temperature hold periods in the creep regime increase the in-plane tensile stress in the oxide on the return to temperature. High-temperature hold periods tend to reduce the peak tensile stress produced in subsequent cycles but, for the example examined here, the effect is small.

Keywords: Thermal cycling; Tensile cracking; Creep hysteresis; Chromia; Stainless steel

1. Introduction

The spallation of protective oxide layers, usually chromia or alumina, during cooling occurs when the oxide layer experiences in-plane compression. This mode of oxide failure is well-recognised and has been the subject of extensive experimental and modelling studies [1–5]. It is becomingly increasingly recognised, however, that in-plane tensile stresses can develop in protective oxides as a direct result of thermal cycling and that these can produce tensile cracking of the oxide layer. This additional form of damage has been reported in experimental studies on alumina-forming alloys [6,7] and has been predicted in finite-element computations [8–11]. In these latter studies, it was shown that tensile stress develops in the oxide layer on the return to temperature during the thermal cycle and is the consequence of creep relaxation within the alloy during the cooling half-cycle. This "creep hysteresis" will be considered further in this present work.

The main purpose of this paper is to present the results of a combined experimental and finite-element study of tensile cracking of a chromia layer formed on a 20Cr25Ni austenitic steel during thermal cycling. Particular attention will be paid to the sensitivity of the cracking process to the amplitude of the temperature cycle through the use of in situ monitoring by acoustic emission. In addition, the influence of substrate creep will be examined by the insertion of hold periods at the minimum and maximum temperatures of the thermal cycles.

2. Experimental

2.1. Procedures

The alloy used in this study was a 20Cr25Ni, Nb-stabilised austenitic steel with composition (wt.%): 19.2Cr, 25.3Ni, 0.56Si, 0.6Mn, 0.7Nb, 0.054C, balance Fe. It was supplied by the former Nuclear Electric in the form of cold-rolled strip of 0.38 mm thickness. Prior to testing, specimens measuring 20 mm × 10 mm, were annealed in commercial purity argon for 1 h at 930 °C to produce a uniform recrystallised grain size of approximately 15 μm. This structure remains stable during subsequent exposures at 900 °C. The oxidation tests were performed at 900 °C in an environment of CO_2/1%CO by volume at 1 atm total pressure, giving an oxygen partial pressure of

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7.98×10^{-13} atm. This oxygen potential is sufficient to oxidise the main constituents, chromium and iron but not nickel. These test conditions have been used previously in extensive studies of the basic oxidation [10,11] and spallation [1,12] properties of this alloy. From these it is known that a protective chromia layer, together with a thin silica interlayer, will form quickly at 900 °C and continue to thicken with continued exposure to reach. For the test conditions examined here (984 h at 900 °C) a chromia layer of approximately 5 μm and a silica layer of <0.1 μm will have formed. Thermal cycling commenced after this isothermal exposure.

The basic saw-tooth cycling history used in this work after this isothermal period is shown schematically in Fig. 1(a) and incorporated controlled cooling and heating rates at 100 K h^{-1}. It is also known [12] that, at this thickness of surface layer, oxide spallation will begin to occur after a single temperature drop of approximately 270 °C and, in order to avoid this during cycling, temperature amplitudes ≤225 °C were used. A hold period of 24 h at the minimum temperature, i.e. cycles of the type shown in Fig. 1(b), were also used with temperature amplitudes of 190 and 200 °C to examine further the influence of substrate creep. In addition, the influence of a 4 h hold period at the top temperature of 900 °C was also examined for a single temperature amplitude of 190 °C as shown in Fig. 1(c). Again, for these modified cycles, controlled cooling and heating rates of 100 K h^{-1} were used and also maintained during final cooling at the end of the test to a temperature of 250 °C after which this cooling rate could not be maintained and the specimens were allowed to cool at the natural rate in the furnace.

In one series of tests, specimens were exposed in a Cahn TG171 thermobalance so that mass change during isothermal and cyclic conditions could be monitored. The data were corrected to allow for buoyancy effects during the temperature changes. It was confirmed that there was negligible change in mass of the platinum hangdown wire during the exposure period. In another set of experiments, specimens had an acoustic emission (AE) waveguide and a thermocouple welded to them and were exposed, also in a CO_2/1%CO environment, in conventional tube furnaces. AE signals and temperature were monitored during thermal cycling using commercial Physical Acoustics MISTRAS™ equipment.

After cooling, selected specimens were nickel plated to retain surface integrity, sectioned, mounted in resin and mechanically polished to a final finish of oxide polish silica (OPS)™. Elemental distribution maps (digimaps) were generated in the SEM using EDX controlled by a Link AN10000 analysis system.

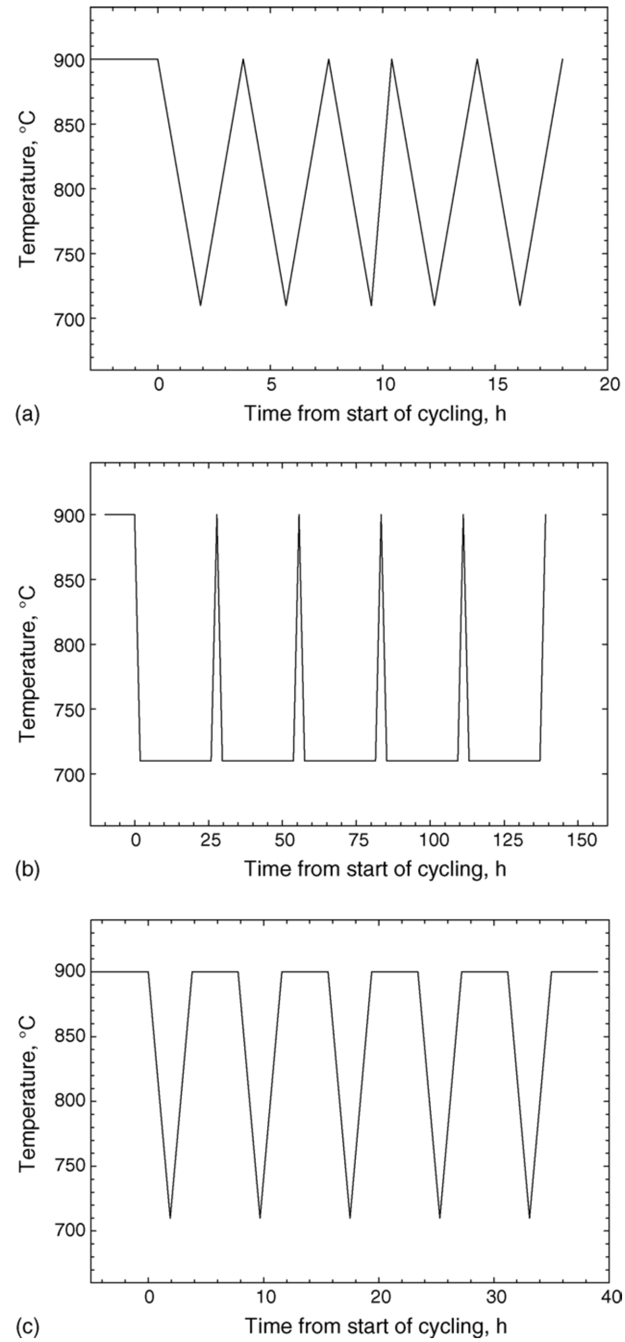

Fig. 1. The three types of thermal cycles used: (a) simple saw-tooth; (b) 24 h hold period at the bottom temperature and (c) 4 h hold period at the top temperature. Changes of temperature occur at 100 °C h^{-1}.

2.2. Experimental results and discussion

Some of the mass gain results from the thermobalance tests for the simple saw-tooth thermal cycles (Fig. 1(a)) are given in Fig. 2. These show the nett mass gain (on the return to 900 °C) above that recorded at the end of the initial 1000 h period of isothermal oxidation as a function of the number of complete thermal cycles. Temperature amplitudes of 180, 190 and 200 °C were examined. It can be appreciated that, even over this narrow temperature range, significant differences in response were found. Thus, for the smallest amplitude considered, saw-tooth cycling had no appreciable effect whereas, for an amplitude of 200 °C, there was a dramatic increase in mass during the first cycle. The specimen cycled from 900 to 710 °C showed intermediate behaviour with the mass gain starting at cycle 13. Also shown in Fig. 2 are the gravimetric results obtained from tests in which a 24 h hold period was introduced at the minimum temperature, i.e. cycles of the type shown in Fig. 1(b). Cycles amplitude of 200 and 190 °C were used and it can be seen that