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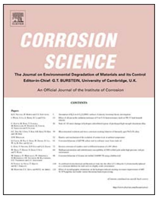

Short Communication

Microstructure evolution of FeNiCr alloy induced by stress-oxidation coupling using high temperature nanoindentation

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ABSTRACT

The microstructure evolution of a FeNiCr alloy oxidized at 600 °C by simultaneously applying stress via high temperature nanoindentation is reported. Analysis using transmission electron microscopy shows that a sharp crack was induced beneath the indentation area under the stress-oxidation coupling condition. Nanotwins beneath the indentation area were also observed, which acted as a barrier that ceased the crack propagation beneath the indenter by altering the path of the crack. Results reveal a transformation from inter-granular crack propagation along the oxide grain boundaries to intra-granular crack propagation through the nanotwin structure with a zig-zag pattern.

1. Introduction

Oxidation of metals/alloys with applied mechanical loading at elevated temperature is a long standing issue that is of great importance for both fundamental understanding of oxidation process and engineering application [1–3]. The advancing of nanotechnology and experimental instruments has made it possible to investigate the oxidation of various materials at small scale. For instance, Viskari et al. [4] used high-resolution analytical techniques to investigate the oxidation at inter-granular crack tip in a Ni-base superalloy and showed that oxidation took place at and immediately ahead of the tip of an open crack. Kitaguchi et al. [5] investigated the oxide growth ahead of the inter-granular crack tip in an advanced Ni-base superalloy using (scanning) transmission electron microscopy and observed different oxide intrusion lengths and oxide formation ahead of the crack tip under various loading conditions. However, in their experiments the cracks in the samples were pre-fabricated using fatigue pre-cracking method at macroscale, and the analysis was mainly focused on the nature of the formation of layered structure oxide ahead of the crack tips, while further understanding of the stress effect on stress-oxidation interaction and microstructure evolution including crack nucleation and propagation is lacking.

In addition, microstructures such as twin boundaries, grain boundaries, second phase particles as well as other interfaces all have strong interaction with cracks when locally the cracks encounter such microstructures [6–8]. For instance, when a crack meets a grain boundary in a material that exhibits preferred crack paths within a grain, the orientation change of such a preferred path in the adjacent grain could retard the crack propagation and thus enhance cleavage-cracking resistance [6]. The crack may also deflect in a manner of intra-granular propagation to enhance the fracture toughness [7,8]. Both crack nucleation and crack growth depend closely on the microstructure at sub-micro scale (length scales of ~100 nm–~1000 nm) [6]. When microstructure evolution and crack formation encounter oxidation at elevated temperature (which is common since oxidation degrades the material properties and results in microstructure evolution as well as crack formation/propagation), the challenges remain to probe into structures at such small scales to investigate the interaction between the cracks and local structures under oxidation conditions with applied mechanical loading. Conventionally, it is difficult to apply the mechanical loading with precise control in a region of interest (e.g. grain boundary, etc.) at such a small scale to study the crack nucleation and propagation during oxidation at high temperature.

In order to understand the stress-oxidation coupling effect [9–11] on the microstructure evolution, as well as to investigate this coupling effect on the failure of materials at high temperature in oxidation environment, here in this work the high temperature nanoindnentation is adopted for the design of small scale experiments. High temperature nanoindentaion has been attracting more attention in recent years to measure material properties at high temperatures [12–14], for instance,

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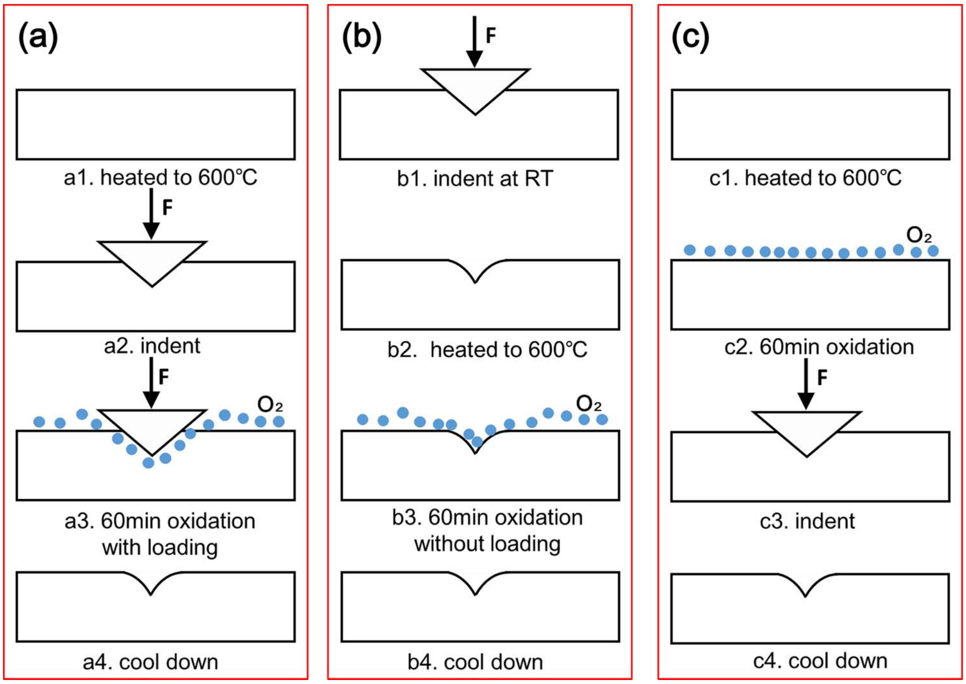

Fig. 1. Schematic illustration of the experimental flow of the three comparative tests.

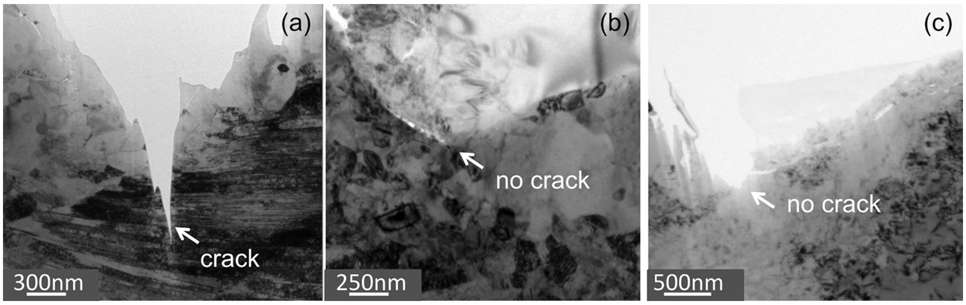

Fig. 2. TEM images showing the morphologies in the vicinity of the indentation areas and the white arrows indicate the tip area of each indent: (a) Sample A with crack; (b) Sample B with no crack; (c) Sample C with no crack.

on Ni-base superalloys [15,16]. Nanoindentation instrument equipped with high temperature stage can be used as high temperature scanning probe microscope (SPM) to first scan and pinpoint specific locations at small scale and then apply load as normal high temperature indentation. The combination of high temperature SPM and nanoindentation makes it a useful tool to study the oxidation effect on the evolution of microstructures [17–19] at small scale with the ability of precise targeting and loading. Precise targeting at positions of interest also facilitates the post-mortem characterization of the targeted position to better understand the coupled process of stress-oxidation-structure evolution.

2. Experiments

2.1. Sample preparation

A Fe-20Ni-33Cr (wt.%) alloy (Central Iron & Steel Research Institute, China) was used in the present experiment. The surface of the specimens cut from the bulk was first grinded using an automatic grinding machine with 200–1200-grit silicon carbide papers, followed by polishing with diamond paste with particle size of 2.0 μm and 0.5 μm. Finally vibration polishing using solutions with nanosized silica was carried to remove the mechanically deformed surface layer. The surface of the specimens was then cleaned with ethanol in ultrasonic bath and dried afterwards.

2.2. Mechanical loading using nanoindentation

The TI 950 Tribo-indenter (Hysitron Inc., USA) was adopted to conduct the experiments. The equipment has a displacement resolution of 0.02 nm and load resolution 1 nN. A Berkovich diamond indenter, which is brazed to a Macor shaft, is adopted to apply the mechanical loading. The thermal conductivity of Macor has a very small value of about 1.5 $W\,m^{-1}\,K^{-1}$ [20]. The indenter is designed for tests at high temperature by reducing thermal conduction from the surface of the specimen to the transducer as well as preventing the standard probe holder from being oxidized or melted due to the high temperature effect [17]. In the present experiment, the operation stage for carrying out nanoindentation test was heated first to maintain the sample surface temperature at 600 °C within a fluctuation of ± 0.5 °C. Then the Berkovich indenter was brought in contact with the specimen under a small contact load (2 μN). It is noted that such a contact process would introduce a temperature fluctuation, thus the indentation test was not conducted until the monitored temperature of the specimen became thermally stable again (e.g. the temperature fluctuation is within ± 0.5 °C). The indentation was then performed with a linearly increasing period of 5 s to the maximum load 11,000 μN and the indenter was maintained in contact with the specimen for 60 min at this maximum