Wear 414–415 (2018) 289–295

Contents lists available at ScienceDirect

Wear

journal homepage: www.elsevier.com/locate/wear

The effect of subsurface degradation on tribocorrosion behaviors of Inconel 690 in 3.5% sodium chloride

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ARTICLE INFO

Keywords:
Nickel
Polarization
Fretting corrosion
Oxidation

ABSTRACT

The fretting corrosion behaviors of Inconel 690 under different electrochemical polarization condition in 3.5% sodium chloride were investigated. The results indicated that the degradation mechanism of subsurface microstructure, which strongly depended on polarization condition, played significant influence on fretting corrosion behaviors. In cathodic polarization condition (CPC), oxidation was suppressed. Tribologically transformed structure (TTS) was stripped by plowing, thereafter mixed with the transferred material to form the wear debris layer (WDL). In anodic polarization condition (APC), oxidation was promoted. TTS and WDL were directly dissolved into solution by oxidation. Therefore, the wear mechanism in CPC is mainly abrasive wear, but is abrasive wear and oxidation in APC. Correspondingly, the wear volume in CPC is smaller than that in APC.

1. Introduction

Fretting wear, which could cause the reduction of service life of steam generator (SG) tube and even the leakage of radioactive materials fission products, has been one of the most important safety and economics related issues in pressurized water reactor nuclear power plants. Fretting wear of SG tube usually occurs at the tube support sites due to the flow vibration or pressure pulsation in tube [1–3].

In the early, the effort has been mainly devoted to research the effect of mechanical factors (such as normal force, displacement amplitude and number of cycles) as well as environment factors (such as temperature, solution property) on fretting wear behavior of Inconel 690 and 600, which have been widely used for the SG tube [4–7]. Recently, with the development of technology, a number of electrochemical techniques have been employed to study fretting corrosion behavior as the working environment of SG tube is corrosive. These applied electrochemical techniques mainly include open circuit potential measurements, potentiodynamic polarization measurements and electrochemical impedance spectroscopy measurements [8,9]. For example, Lgried et al. used open circuit potential to investigate the effect of sliding regime on the fretting-corrosion damage of Inconel 600. Employing this method, authors conclude that the regional transformation from gross slip to stick-slip regime could prevent passive film breakdown and excessive mass lost [10]. Meng et al. have studied the localized corrosion behavior of scratches on nickel-base alloy 690. They found that scratches displayed more extensive electrochemical activity than the unworn matrix. The intense localized corrosion could contribute to crack initiation at the bottom of scratch groove [11,12]. In short, the researches about tribocorrosion behavior of SG tube have mainly focused on worn scar surface degradation behavior, such as the repair and destruction process of passive film. However, more and more research works indicate that the subsurface deformation plays great important role in fretting wear behavior [13–16]. Lee et al.'s research work indicated that Inconel 690 presents more excellent fretting wear-resistance than Inconel 600 because a smaller cells structure could be formed in worn scar subsurface of Inconel 690 during fretting wear [16].

For further investigate the effect of subsurface degradation behavior on tribocorrosion of Inconel 690, the fretting wear tests were firstly conducted in 3.5% NaCl solution under different electrochemical polarization condition by controlling the applied potentials. Then, the worn scars were characterized and analyzed. Finally, the evolution mechanisms of microstructure in worn subsurface were proposed.

2. Experimental procedure

The test specimens of Inconel 690 were split from a commercialized SG tube preceded by flattened, heat treatment, ground and polished. The microstructure of Inconel 690 was revealed by electrolytic etchings in 10% oxalate solution at 5 V for 25 S. From the scanning electron

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https://doi.org/10.1016/j.wear.2018.08.017
Received 12 February 2018; Received in revised form 24 August 2018; Accepted 24 August 2018
Available online 25 August 2018

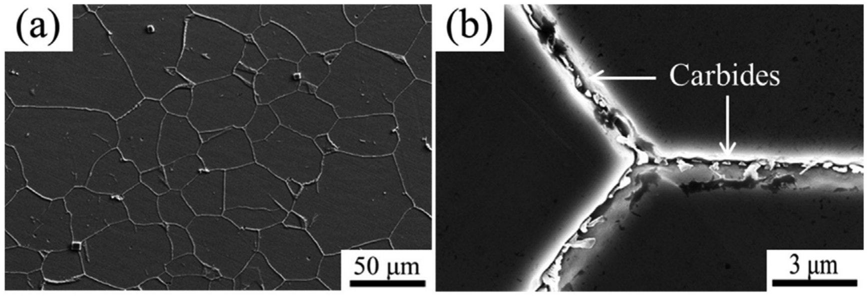

Fig. 1. The SEM morphologies of Inconel 690: (a) grains and (b) carbides located at the grain boundary.

Table 1
Chemical compositions of Inconel 690 and 304 SS.

Specimen	Element (wt%)											
	Ni	Fe	Cr	C	Ti	Mn	Si	N	S	P	Co	Mo
Inconel 690	Bal	11.6	29.9	0.025	0.30	0.25	0.33	0.020	0.025	0.01	0.015	–
304 SS	9.35	Bal	18.3	0.018	–	0.25	0.31	–	0.025	0.034	–	0.18

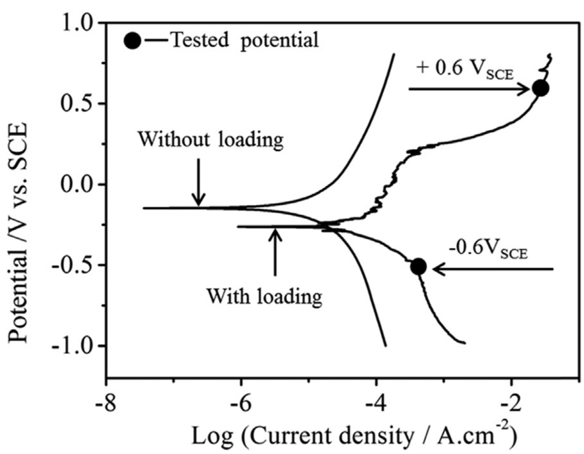

Fig. 2. Potentiodynamic polarization curve of Inconel 690 and 304 SS with and without rubbing in 3.5% NaCl solution.

microscope (SEM) morphology shown in Fig. 1a, the sample of Inconel 690 exhibited equiaxed grains structure. And the average size of 300 grains was about 28 μm. In addition, the enlarged SEM morphology shown in Fig. 1b indicated that nano-sized carbides precipitated along grain boundaries. The other material in friction pair was 304 stainless steel (304 SS), which was usually used as the SG tube support material in nuclear sector. The chemical compositions of 304 SS and Inconel 690 were listed in Table 1.

An oscillating friction and wear tester with a solution tank was used in this work. The tribocorrosion tests were performed in 3.5% NaCl solution at room temperature using ball-on-flat contact configuration. The upper specimen was 304 SS ball with a diameter of 10 mm and stationary, which was mounted inside the bakelite fixture to insulate the rest of the equipment. The lower specimen was Inconel 690. Then a three-electrode setup was built up, in which the friction pairs acted as working electrode, the saturated calomel electrode (SCE) acted as reference electrode and the high purity graphite acted as counter electrode. Meanwhile, the three-electrode would connect with CS-350 electrochemical workstation. Before potentiostatic tribocorrosion test, the potentiodynamic polarization curve was measured by sweeping the potential from $-1.0\ V_{SCE}$ to $0.8\ V_{SCE}$ at a rate of 2 mV/s with and without rubbing. The state of without rubbing was that the friction pairs didn't contact with each other as unloading, but the lower specimen oscillated. The surface area, which was used for calculating surface density in this research, was the total exposed surface of nickel-based alloy and stainless steel. Fretting parameters was respectively set as 60 N in normal load, 100 μm in displacement amplitude and 20 Hz in frequency, which located in the actual range occurred between SG tubes and their support. Thereafter, potentiostatic tribocorrosion tests were conducted under the potentials of $-0.6V_{SCE}$ (cathodic condition) and $+\ 0.6\ V_{SCE}$ (anode condition) with 30 min in test duration. In order to avoid the influence of the worn scar on the subsequent corrosion wear test, only one tribocorrosion test was performed for each set of friction pairs. Each test was repeated at least twice to check for repeatability.

After fretting corrosion tests, the wear volume, profiles and surface morphology of worn scar were successively characterized using a 3D surface profilermeter (America ADE MicroXAM-3D) and ZEISS Auriga field emission SEM (FESEM) equipped with an energy dispersive X-ray (EDX) detector. Thereafter, in order to research the worn subsurface degradation behavior under different electrochemical conditions, two transmission electron microscopy (TEM) samples were prepared using ZEISS Auriga focused ion beam (FIB) equipment then characterized by FEI Tecnai F20 equipped with an EDX detector at 200 kV.

3. Results

Fig. 2 shows the potentiodynamic polarization curve of Inconel 690 and 304SS with and without rubbing in 3.5% NaCl solution. It is found that the open circuit potential (without rubbing) is $-0.125\ V_{SCE}$, which is shifted to the more cathodic potential of $-0.263\ V_{SCE}$ as rubbing. As a result, the electrochemical property of investigated surface during fretting wear could be divided into two conditions: anodic polarization condition (APC, above $-0.263\ V_{SCE}$) and cathodic polarization condition (CPC, below $-0.263\ V_{SCE}$).

Figs. 3a and 3b show the change of coefficient of friction (COF) and current with test time at different applied potentials in 3.5% NaCl solution, respectively. As shown in Figs. 3a and 3b, three regions could be defined for the evolution of COF and current during fretting corrosion in all conditions. In region I, without rubbing, the value of COF is zero and the current value is small. In Region II, after rubbing, both COF and current values firstly dramatically increase then fluctuate with test time. However, it should be mentioned that compared with CPC, the COF of APC present a larger value and greater fluctuations. In region III, without rubbing, the COF immediately decreases to zero and current also decreases to a small value.

Fig. 4 shows the typical cross-sectional profiles of worn scars at