Materials Characterization 131 (2017) 157–167

Contents lists available at ScienceDirect

Materials Characterization

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

The comparative study on nanostructured tribolayers of Alloy 690TT subjected to fretting wear under different oxygen contents

Long Xin[a], Yonghao Lu[a,*], Tetsuo Shoji[a,b]

[a] National Center for Materials Service Safety, University of Science and Technology Beijing, Beijing 100083, China
[b] Frontier Research Initiative, New Industry Creation Hatchery Center, Tohoku University, 6-6-10, Aramaki Aoba, Aoba-ku, Sendai 980-8579, Japan

ARTICLE INFO

Keywords:
Fretting wear
Nickel-based superalloys
Nanostructure
Oxidation
TEM

ABSTRACT

The nanostructured tribolayers of Alloy 690TT subjected to dry fretting wear at 320 °C under different oxygen contents were studied. Results indicated that the low oxygen content (5 vol%) produced the nanostructured tribolayer containing large amount of Cr_2O_3 with an average grain size of 15 nm, which was mainly due to the limited oxidation and preferential oxidation of Cr, resulting in lower friction coefficient, wear volume, dissipated energy, smoother worn surface and thicker tribolayer. The farther from the worn surface, the weaker was the Cr_2O_3 concentration. Only a small quantity of microcracks perpendicular to the worn surface were observed in the Cr_2O_3-rich tribolayer under a low oxygen content, which indicated the Cr_2O_3-rich tribolayer had a better wear resistance. By comparison, under a high oxygen content (21 vol%), the tribolayer mainly consisted of $NiCr_2O_4$ and Fe_2O_3 with an average grain size of 7 nm due to the sufficient oxidation during fretting wear, resulting in higher friction coefficient, wear volume and dissipated energy. The plenty of microcracks without specific orientation and voids appeared in the oxide layer and along the interface of oxide/metallic grains.

1. Introduction

Fretting wear is a type of wear phenomena resulting from minute oscillations that is characterized by removal of materials from two contacting surfaces [1]. Thermally treated Alloy 690 (Alloy 690TT), a nickel-based superalloy, has been widely used as a steam generator (SG) tubing material in nuclear power plants (NPPs) [2]. During the process of heat exchange, the thin tubes are subjected to flow induced vibration (FIV), resulting in fretting wear between SG tubes and their anti-vibration structures [3,4].

Friction and wear, a kind of severe plastic deformation (SPD), is regarded as an effective way to create ultrafine-grained (UFG) or nanocrystalline (NC) structure and concomitant strengthening in near-surface regions of metals and alloys [5–7]. Furthermore, introducing the UFG structure via grain refinement by SPD strengthens the metals and alloys [8,9]. The wear resistance of materials can be enhanced by controlling the grain size of UFG structure [10,11] or reducing the grain size of bulk material [12]. Under fretting condition, the UFG or NC structure without oxidation is always called as tribologically transformed structure (TTS) which results from the dynamic recrystallization of deformed structure in the subsurface [13–15]. Both SPD and oxidation simultaneously dominate the wear behavior. Especially, the oxidation behavior can be enhanced by elevated temperature during fretting wear process [16]. Consequently, a superficial oxide layer, which is always regarded as “glaze layer” during high temperature wear, generates and results in the reduction of friction coefficient and wear rate [17,18]. In a steel contact the oxides that are expected to be iron oxides (Fe_2O_3 and Fe_3O_4) [19]. In a nickel-based alloy contact the oxides should be NiO or spinel oxides [18,20]. The formation of these compounds is highly associated to the wear behavior of bulk material. Also, there is a distinction between oxides that are either beneficial (protective films) or harmful (damaging debris) [21–24]. On the other hand, fretting wear behavior is affected by the environment conditions, such as the composition of atmosphere. It is believed that the composition of the atmosphere can dictate the chemical reactions in the contact zone and therefore change the fretting response [19,25], which directly impacts the friction coefficient and formation of wear debris. Especially, the Alloy 690TT tube in NPP experiences high temperature of 320 °C [2,26] and low oxygen density [27]. Therefore, fretting wear resistance is closely related to the temperature, specific atmosphere and the formation and stability of the tribolayer on the contact surfaces.

In this study, fretting wear behavior and mechanism of Alloy 690TT were investigated under different oxygen contents. Detailed microstructural analyses of the subsurface were performed. Such a study is expected to be important for understanding the relationship between the nanostructured tribolayer and oxygen content under fretting

* Corresponding author.
E-mail addresses: mcsohumoon@126.com (L. Xin), lu_yonghao@mater.ustb.edu.cn (Y. Lu), tshoji@fri.niche.tohoku.ac.jp (T. Shoji).

http://dx.doi.org/10.1016/j.matchar.2017.04.034
Received 3 March 2017; Received in revised form 24 April 2017; Accepted 27 April 2017
Available online 28 April 2017

Table 1
Chemical composition of Alloy 690TT and 304SS in wt%.

Specimen	Element								
	Ni	Fe	Cr	C	Ti	Mn	Si	P	S
Alloy 690TT	Bal	11.6	29.9	0.025	0.30	0.25	0.33	0.086	0.0025
304SS	9.35	Bal	18.3	0.018	–	1.31	0.31	0.034	0.0025

condition, which can provide necessary information for the optimization of fretting wear resistance of Alloy 690TT.

2. Experimental details

The dry fretting wear tests were performed using a ball-on-plate type test rig with a version of SRV IV. The samples of Alloy 690TT with a hardness of HV ~ 235 were machined in the form of rectangular blocks with dimensions of 10 mm × 10 mm × 1 mm. The counterface material was Type 304 stainless steel (304SS) ball with a hardness of HV ~200 and a diameter of 10 mm. The chemical composition of the friction pair is shown in Table 1. The normal load, frequency, stroke, test duration and temperature were constant at 100 N, 20 Hz, 150 μm, 30 min and 320 °C, respectively. During the fretting tests in air, the relative humidity in the environmental chamber built around the tribometer was RH = 45 ± 2%. The fretting tests in nitrogen atmosphere were performed in order to study the wear resistance of Alloy 690TT under a low oxygen content. During these tests, the chamber was first flushed with compressed nitrogen gas with purity higher than 99.99, and then the tests run under a continuous flow of nitrogen gas that was directly blown onto the samples. After the fretting wear test, the mixed gas in the chamber was quickly and discreetly collected by the dedicated gas sampling bag. The constituent of mixed gas was measured by the Gas Chromatography showing the composition of atmosphere was 95 vol% nitrogen and 5 vol% oxygen. It is well known that the normal atmosphere in air consists of typically 79 vol% nitrogen and 21 vol% oxygen. Three repeated tests were performed under different oxygen contents. Therefore, the effect of oxygen content on fretting wear of Alloy 690TT can be found and discussed.

The morphologies of worn surface and cross-section were investigated using scanning electron microscope (SEM). In addition, the compositional and chemical states of worn surfaces were analyzed by Raman and energy dispersive X-ray (EDX). The Raman spectroscopy contains a laser with an excitation wavelength of 532 nm and a maximum power of 1.5 W, and the Raman shift range is 100–2000 cm^{-1} with the integration time of 15 s depending on the signal intensity of the samples. The location of the Raman testing was in the middle of the wear scar. The microstructure of subsurface was further analyzed by using transmission electron microscopy (TEM). TEM analyses were carried out using a FEI Tecnai F20 equipped with an EDX detector at 200 kV. TEM works were performed to obtain the bright field (BF), dark field (DF) and high angle annular dark field (HAADF) images taken in the scanning transmission electron microscope (STEM) mode. The thin foil TEM specimens were prepared using a TESCAN LYRA 3 FEG-focused ion beam (FIB) equipment combining with SEM. Prior to FIB milling, a thin platinum layer was deposited on the surface to protect it from the beam damage. The method for measuring grain size from the TEM images was that about 200 grain boundaries from several DFTEM images were retraced to obtain binary images, which can be evaluated using an image analysis software. The grain areas were interpreted as circles and the distribution of the corresponding circle diameters can be calculated.

3. Results

Fig. 1 shows the comparison of friction coefficient (FC) trends observed for fretting tests carried out under different oxygen contents. As shown in Fig. 1(a), for both investigated oxygen contents, FC is found to increase initially, and then decrease to a rather steady value for the rest of the test. The running-in period under 5 vol% oxygen is found to be shorter than that of 21 vol% oxygen. FC under 5 vol% oxygen reaches the maximum of ~1.49, which is higher than that of 21 vol% oxygen. As shown in Fig. 1(b), the lower value of average FC under 5 vol% oxygen is observed (5 vol%: 0.69, 21 vol%: 0.86).

Fig. 2(a) shows the profile micrographs of wear scars of Alloy 690TT along the fretting direction. The profile under 5 vol% oxygen resembles a "U" shape. However, resembling a "W" shape is observed under 21 vol % oxygen. Besides, the maximum wear depth of wear scar under 5 vol% oxygen is about 19 μm, which is far lower than that of 21 vol% oxygen (41 μm). Fig. 2(b) shows the corresponding maximum values of wear volume under different oxygen contents. It is found that the value under 5 vol% oxygen is lower than that of 21 vol% oxygen.

The curve of Ft-D (Ft is the tangential force; D is the displacement) is the most essential kinetic information obtained from the tangential fretting wear test. Fig. 3 shows the curve of Ft-D as a function of cycle number from 6 × 10^3th to 36 × 10^3th under different oxygen contents. It is found that all Ft-D curves show the shape close to parallelogram, which imply that fretting was running in the gross slip condition and the relative motions were essentially accommodated by the plastic deformation of contacting surfaces [28]. In addition, the area enclosed within the fretting loop is the dissipated energy in contact per cycle (E_d) due to gross sliding of the contact. Noticed that $E_{d(21vol\%)}$ has almost no change with the increasing of cycle number. However, the $E_{d(5vol\%)}$

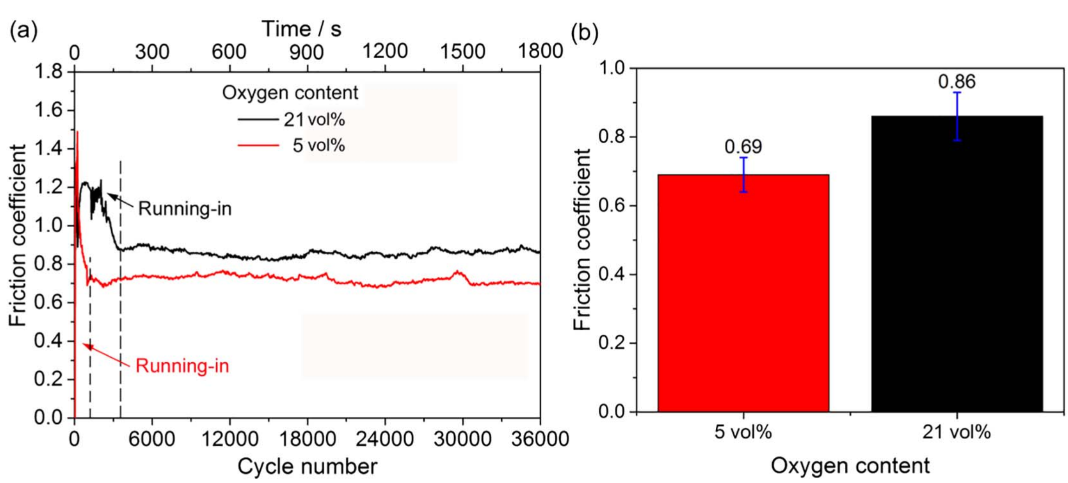

Fig. 1. (a) Friction coefficients versus cycle number/time and (b) the histogram of average friction coefficients under different oxygen contents.