



Isothermal oxidation mechanism of Nb–Ti–V–Al–Zr alloy at 700–1200 °C: Diffusion and interface reaction



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ABSTRACT

Isothermal oxidation kinetics of Nb–Ti–V–Al–Zr alloy at 700–1200 °C followed a mixed parabolic-linear law. The oxidation region consisted of an outer oxidation layer (OOL) and an internal oxidation zone (IOZ). OOL was composed of TiO₂ and Nb₂O₅ at 700–800 °C, TiO₂ and Nb₂O₅ as well as TiNb₂O₇ at 1100 °C, and TiNb₂O₇ and TiO₂ at 1200 °C. IOZ could be divided into two zones according to morphology and distribution characteristics of Ti-rich internal oxides at 1100–1200 °C. The oxidation of Nb–Ti–V–Al–Zr alloy is proposed to be controlled by chemical reaction in the oxide/alloy interface and diffusion of Ti⁴⁺ and O²⁻.

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1. Introduction

The increasing demand in thrust–weight ratio of aerospace engines has triggered the increased attention in the research of lightweight, oxidation and corrosion resistance of high-temperature materials [1–7]. Niobium alloys, such as Nb–Ti–Al, Nb–Ti–Si, and Nb–Si–Al systems, have been studied since the 1990s [8–10]. Niobium alloys used as structural materials have become strong competitors of conventional Ni-based superalloys, Co-based superalloys, and intermetallic compounds because of their excellent properties, such as high melting point, qualified high temperature strength, and low density [11]. Considering that niobium and titanium can be solid solute infinitely [12], the niobium alloys are composed by niobium the matrix, titanium the main alloying element and silicon, aluminum, chrome, vanadium and tungsten the strengthening elements [13]. The alloying elements not only ensure high temperature strength of the alloys but also lower their densities (5.5–7 g cm⁻³).

Considerable achievements in optimizing high temperature mechanical properties of niobium alloys have been made over the past decades [14–19]. However, the wide application of niobium alloys is limited by their poor oxidation resistance at high temperatures, wherein the solubility of oxygen in the niobium matrix is higher and its diffusion rate is faster compared with

conventional superalloys [12,20–24]. At present, studies on the oxidation behavior of niobium alloys mainly focus on the following aspects: the effects of alloying elements and microstructure on the oxidation resistance of niobium alloys [11,13,25,26], cyclic oxidation behavior of niobium alloys [27,28], and the fitting calculation of oxidation kinetics [28]. These studies are related to the systems, such as Nb–Cr–Si [1,29], Nb–Ti–Si [2,30,31], and Nb–W–Cr [32–34]. In-depth studies on the mechanism of high-temperature oxidation of Nb–Ti–V–Al–Zr system are rare. In the present research, both experimental and theoretical studies were conducted to identify the oxidation mechanism of Nb–Ti–V–Al–Zr alloy, which is expected to improve oxidation resistance.

2. Material and methods

Alloy was melted into ingots by using a vacuum induction furnace and then underwent a homogenization annealing. The composition of the alloy was: (wt.%) 48.49 Nb, 33.10 Ti, 9.06 V, 7.63 Al, and 1.72 Zr. Specimens with parallelepiped shape and similar dimensions ($H_0 \times M_0 \times L_0 = 2 \text{ mm} \times 5 \text{ mm} \times 10 \text{ mm}$) were ground with SiC abrasive paper and ultrasonically cleaned in acetone and ethanol.

Isothermal oxidation testing was performed at 700, 800, 1100, and 1200 °C for 5 h in air flow (40 mL/min) by using a NETZSCH STA 449C thermal analysis instrument. Specimens were placed in a corundum crucible and heated to the required temperatures as quickly as possible in high purity argon. Subsequently, the

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atmosphere was changed to air after thermal equilibrium. The mass of the specimens was recorded every minute by an electronic scale with an accuracy of 0.001 mg.

After oxidation, the macro surface morphologies of the specimens were observed to evaluate the oxidation severity. Subsequently, the micro surface morphologies were detected by scanning electron microscopy (SEM; JEOL JSM-6510A). X-ray diffraction (XRD) analysis was conducted on a Rigaku D_{MAX}-RB anode diffraction instrument equipped with a θ/θ goniometer using Cu K α radiation (40 kV, 150 mA) from 10° to 110° at a step of 0.02° to analyze the surface phase. In addition, the cross-sectional morphologies of the specimens were examined via SEM after oxidation. The contents of alloying elements in the oxide scales and substrate were measured via energy dispersive X-ray spectrometry (EDS). Electron probe microanalysis (EPMA) was carried out using a JEOL JXA-8230 instrument to obtain a precise elemental distribution.

3. Results and discussion

3.1. Oxidation behavior

Fig. 1 shows the isothermal oxidation kinetics of the alloy at 700, 800, 1100, and 1200 °C by specific weight gain vs. time.

Weight gain increased as time progressed at each temperature. After oxidation for 5 h, the specific weight gains were 1.21, 2.21, 12.0, and 16.9 mg cm⁻² at 700, 800, 1100, and 1200 °C, respectively, as listed in the table attached to Fig. 1. These results indicate that oxidation became more severe as oxidation time progressed and temperature increased.

In general, oxidation kinetics is formulated by the following relationship:

$$\frac{\Delta m}{S} = (k \cdot t)^n \tag{1}$$

where Δm is the weight gain, S is the surface area of specimen, k is the oxidation rate coefficient, t is the oxidation time, and n is the rate exponent. By fitting the data in Fig. 1 to Eq. (1), the values of n were determined to be 0.84, 0.55, 0.73, and 0.74 at oxidation temperatures of 700, 800, 1100, and 1200 °C, respectively. Given that the values of n were between 0.5 and 1, the relationship of specific weight gain vs. time was speculated to follow a mixed parabolic-linear law. Notably, the oxidation rate coefficient k was treated as constant during fitting. This treatment could not identify the oxidation mechanism because the exact physical meaning of the coefficient k was covered up. Chou et al. [35–39] proposed a model including two cases where the oxidation process is controlled by

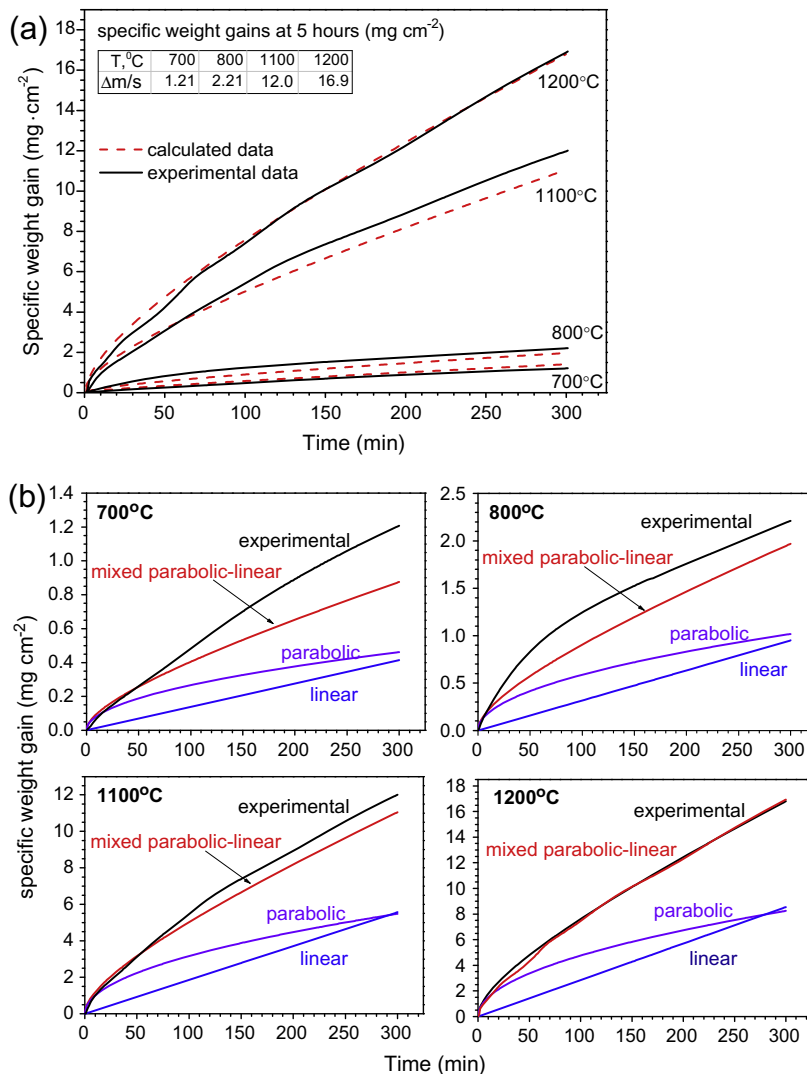


Fig. 1. (a) Isothermal oxidation kinetics of Nb-Ti-V-Al-Zr alloy at 700, 800, 1100, and 1200 °C for 5 h in flowing air; (b) plot of parabolic and linear sections at different temperatures.

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