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Oxidation of in situ synthesized TiC particle-reinforced titanium matrix composites

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Abstract

The oxidation behavior of TiC particle reinforced titanium matrix composites (TMCs) was studied in a temperature range 550–650 °C in atmosphere. The in situ oxidation observation at the very initial stage was investigated by high-temperature optical microscopy in air. The oxide layer of long-term oxidation behavior was examined by scanning electron microscopy (SEM) combined with an energy dispersive X-ray spectroscopy unit (EDX), X-ray diffraction (XRD) and transmission electron microscopy (TEM). The oxidation kinetics follows a parabolic rate law. The oxidation rate decreases gradually as the oxidation proceeds. The oxidation of the composite took place firstly on Ti because of the higher reactivity of Ti and O_2 than that of TiC and O_2. However, the TiC reinforcement can decrease the overall oxidation rate at 550, 600, and 650 °C. It is attributed to the formation of thin and dense oxidation, the enough strong interface cohesion between reinforcements and the titanium matrix alloy.

Keywords: High-temperature; Oxidation; In situ; Titanium matrix composites; Oxidation kinetics

1. Introduction

Titanium-based metal-matrix composites (TMCs) attract extensive attention and are considered technically superior and cost-effective primarily owing to their high specific modulus, high specific strength, high strength at elevated temperature and wide potential application in the field of aviation, aerospace and automobile [1,2]. In recent years, we highlight a novel in situ process in which traditional ingot metallurgy plus self-propagation high-temperature synthesis (SHS) techniques were used to produce TMCs [3]. The formation mechanism of in situ synthesized TMCs has been discussed in ref. [4]. The microstructure and mechanical properties at room temperature have also been investigated in refs. [5,6]. The main aim to develop the TMCs is to widen the utilization field at high-temperature which is limited by a lack of oxidation resistance [7]. Almost no work, however, has been carried out on in situ TMCs oxidation. It is necessary to investigate the oxidation of TMCs.

In the present work, the oxidation behavior of TMCs at temperatures of 550, 600, and 650 °C in air were investigated. The isothermal high-temperature oxidation of TiC/Ti composite was determined. A characterization of the oxidation products, and morphology of the oxide scale were observed in order to throw further light on the oxidation mechanisms of TMCs.

2. Experimental procedures

On the basis of the former paper [8], the materials used in this program included commercially pure titanium and TMCs reinforced with TiC were in situ fabricated by consumable vacuum arc remelting. A common casting technique was used to fabricate the in situ TiC/Ti composites. The reagents used were grade II sponge titanium (99%), graphite powder (99.8%, average particle size: 5–7 μm). The theoretical total volume percent of the reinforcements TiC was 8 and 5%.

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Table 1
Compositions and reinforcements volume percentage in TiC/Ti composites

Sample no.	Ti (wt.%)	C (wt.%)	TiC (vol.%)
TMCs1	98.9	1.1	5
TMCs2	98.24	1.76	8
Pure Ti	100	0	0

The composites of the raw material and the percentage of the reinforcements are listed in Table 1.

The specimens were polished on series of SiC polishing paper of up to #1000, then ultrasonically washed in an acetone bath, dried in air, and weighed before oxidation. In order to obtain information about the oxidation kinetics, coupons of about 10 mm × 10 mm × 3 mm were cut out of the ingots, and the isothermal oxidation studies were carried out in air at a temperature range 550–650 °C for a period of 300 h. The oxidation behavior was evaluated by generating mass-gain data as a function of time.

Samples of about 2 mm × 2 mm × 1 mm for optical microscopy (OM) were taken from the ingots. Then they were prepared using conventional techniques of grinding and mechanical polishing. Their oxidation microstructures were characterized by in situ high-temperature optical microscopy (HTOM) in static air, at 550, 600, and 650 °C for up to 4 h.

The specimens after the oxidation tests were examined. Scanning electron microscopy (SEM) combined with an energy dispersive X-ray spectroscopy (EDX) unit, X-ray diffraction (XRD) analysis, and transmission electron microscopy (TEM) were employed to understand the nature, composition properties of the oxide scales formed during exposure to elevated temperatures. A Phillips TEM 420 with a maximum acceleration voltage of 200 kV and an EDAX energy dispersive X-ray analysis system with windowless detector were available. The TEM samples were prepared by focus ion beam (FIB).

3. Results and discussion

3.1. Oxidation kinetics

The respective kinetics of isothermal oxidation (in terms of mass gain per unit area as a function of time) for pure titanium, TMCs1, and TMCs2 at 550, 600 and 650 °C are presented in Fig. 1. It can be concluded that oxidation rate of pure Ti is higher than that of TMCs1 and TMCs2 at all temperatures. TMCs displayed better oxidation resistance than did pure titanium. Moreover, the oxidation rate decreases with the increase of the volume percentage of TiC reinforcements. At 550, 600, and 650 °C, TMCs2 shows the highest oxidation resistance. The mass gain data in Fig. 1 indicates that the oxidation behavior obeys parabolic rate law.

In general, the isothermal-oxidation kinetic in the parabolic oxidation process is expressed by a power law as

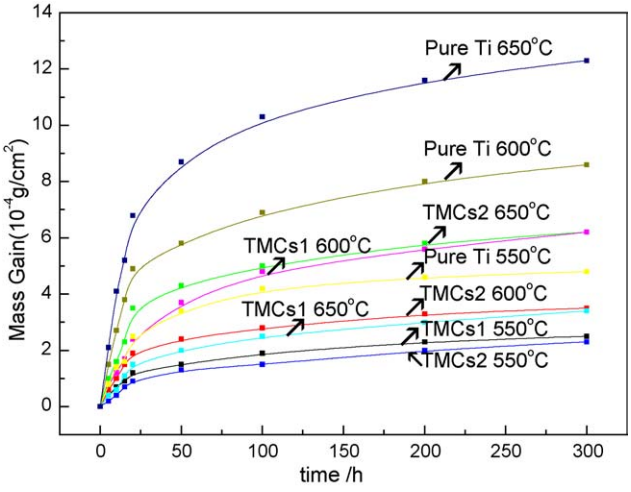

Fig. 1. Mass gain-time relationship of TMCs1, TMCs2 and pure Ti in air at 550, 600, and 650 °C for 300 h.

[9]:

$$\Delta m = (k_p t)^{1/2} \quad (1)$$

where Δm, t, and k_p are the mass gain per unit area, time, and rate constant, respectively. k_p follows an Arrhenius relation of the equation

$$k_p = k_0 \exp\left(\frac{-Q_{eff}}{RT}\right) \quad (2)$$

where Q_{eff} is the effective activation energy for oxidation, k_0 the constant for a given material, T is the absolute temperature and R is the gas constant.

Fig. 2 shows a plot of k_p in logarithm scale versus $1/T$. The slopes of the plots yield the activation energy of oxidation of TMCs1, TMCs2 as 249.8 and 256.3 KJ/mol, respectively. These values show that oxidation activation energy of TMCs2 is higher than that of TMCs1. Moreover, the oxidation acti-

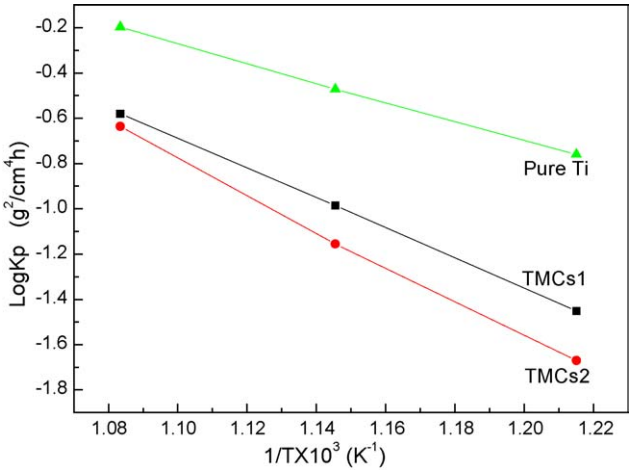

Fig. 2. Arrhenius plot of parabolic rate constant (k_p) for oxidation of TMCs1, TMCs2, and pure Ti in the temperature range 550–650 °C.