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The color changes and tensile properties of oxidized Ti–6A1–2Mo–1.5Cr–2Zr–2Sn–2Nb alloy

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ABSTRACT

The oxidation of titanium alloy at elevated temperature severely limits its property and service life. It is a common and urgent issue to assess the mechanical property of oxidized titanium component without destroying its structural integrity. In this work, the color changes and tensile properties of oxidized Ti–6A1–2Mo–1.5Cr–2Zr–2Sn–2Nb alloy with initial basket-weave microstructure were investigated in the temperature range of 100–1000 °C. It was found that the oxidized specimens exhibited brilliant and regular color changes. X-ray diffraction (XRD) analysis indicated that the color changes were mainly attributed to the mixing of colored titanium oxides in the oxidation layer (such as yellow TiO, white TiO_2 and blue Ti_2O_3). It was also found that mechanical properties were significant influenced by the oxidation treatment. The specimens oxidized below 600 °C exhibited excellent strength and plasticity due to its fine basket-weave microstructure and dense oxidation layer. In the temperature range from 600 to 800 °C, the strength slightly decreased while the plasticity rose a little. The change of mechanical properties during the temperature range was mainly determined by the microstructure change. Eventually, both strength and plasticity decreased sharply when the temperature was over 800 °C, which was mainly caused by the thick oxidation layer with porous and loose microstructure. The fracture mode of tensile specimens also changed from ductile fracture (below 800 °C) to transgranular cleavage fracture (over 800 °C). On the basis of experiments, a corresponding relation between the color changes and tensile properties was established. The royal blue should be regard as critical color between the acceptable and unacceptable tensile properties of the oxidized titanium alloy.

1. Introduction

In recent years, titanium alloys have a wide range of applications in the aerospace field due to attractive properties such as low density, high specific strength, good heat resistance and excellent corrosion resistance. However, titanium alloys readily react with oxygen when exposed in air at elevated temperature leading to oxidation, which severely limits the high temperature property and service life of titanium alloys [1–3].

The oxidation not only results in the formation of oxidation layer (mainly consists of oxides), but also leads to the generation of a brittle oxygen-enriched zone (generally known as "alpha-case") below the oxidation layer [4]. The formation of oxidation layer results in the loss of material and the reduction of load-bearing cross section, which may eventually limit the service time of titanium components [5]. Furthermore, the brittle alpha-case directly affects the mechanical properties of titanium alloys by embrittlement [6]. All kinds of degradation and embrittlement induced by hot corrosion require specific attention. Thus in recent years, the oxidation behavior of titanium alloys has drawn a lot of attention in recent years. Du et al. [7] investigated the air oxidation behavior of Ti–6Al–4V alloy between 650 and 850 °C, and claimed that the multilayered oxide scales formed on the oxidized alloy consisted of alternate layers of Al_2O_3 and TiO_2. The number of Al_2O_3 and TiO_2 layers increased with increasing exposure time and temperature. Jia et al. [4] studied the oxidation behavior and the effect of oxidation on tensile properties of Ti60 alloy, and proposed that the oxidation product was TiO_2 after thermal exposure at 700 °C for 100 h, but a mixture of TiO_2 and a small amount of Al_2O_3 was found in the oxidation layer of specimens oxidized at 750 °C for 50 h. By comparing the tensile property at room temperature before and after oxidation, both of the strength and plasticity decreased for the specimens with oxide scale. Gurrappa et al.

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[8,9] systematically analyzed the oxidation behavior of IMI 834 alloy at different temperatures and a degradation mechanism was proposed under oxidizing environments. They claimed that the alpha-case formation was the dominant degradation mechanism than the formation of oxide scale for titanium alloy at higher temperatures.

Furthermore, the oxidation behavior of titanium alloys during hot working process should be concerned as well. The performance of semi-finished titanium products is also degraded by oxidation reaction. Therefore, in order to eliminate the effect of oxidation and ensure the surface quality, the oxidation layer is generally machined off or chemically milled away before the titanium components are put into service [10]. However, after the last process (such as the welding, grinding and vacuum stress relief annealing process), removing the oxidation layer with a large degree is difficult due to the small machelloning allowance. As a consequence, the performance of such oxidized titanium components are usually assessed by destructive test, which not only destroys the integrity of components but also consumes large amount of time and money. Hence, it is a common and urgent issue to assess the mechanical property of oxidized component without destroying its structural integrity. It is worth noting that brilliant and regular color changes are observed when titanium alloys suffered a series of hot working. The interesting phenomenon has attracted extensive attentions. Alcisto et al. considered that there was not a simple correlation between the brilliant color and thermal history for titanium 6242 alloy, the color changes of oxidation layer relied on the pretreatment of specimens [11]. Peng et al. investigated the oxidation behavior of Ti–5Al–5Mo–5V–1Cr–1Fe alloy, and they found that the generation and change of the oxidized color was correlated with phase composition and microstructure of the oxidation layer [12,13]. Pino et al. analyzed the color of purity titanium by surface oxidation in air with a pulsed Nd:YAG laser operating at high repetition rate. They also considered that there had certain relations between the composition and color [14]. Even more important, the oxidized color has been considered as an evaluation index to assess the mechanical properties of the oxidized titanium alloy after welding process in the American national standard (AWD D17.1:2001), as listed in Table 1 [15]. It provides certain guiding significance and reference value to the practical production, particularly for the last process of hot working. But more concrete details about the corresponding relationship could not be found. Therefore, it is significant to analyze and characterize the oxidized color changes and its relationship to mechanical properties in detail.

Ti–6A1–2Mo–1.5Cr–2Zr–2Sn–2Nb alloy, a newly developed alpha + beta damage tolerance titanium alloy, has been employed as structural material in aircraft applications [16,17]. The alloy, which exhibits specific fracture toughness and strength superior to that of Ti–6Al–4V alloy and Ti–6–22–22s alloy [18,19], has attracted great attentions recently. A number of beneficial researches were extensively conducted and well documented in terms of the effect of processing parameters on the microstructures and mechanical properties [20,21], the hot deformation, microstructures and deformation mechanism [18,22], the kinetics of hydrogen absorption/desorption under various experimental condition [23,24], effect of hydrogen content on superplastic forming/diffusion bonding [25]. These researches were mainly focused on the microstructure evolution and corresponding mechanism. Unfortunately, seldom work has been done for the oxidation behavior. Moreover, there is no report about the detailed investigation of oxidized color changes and its effect on the mechanical properties of the alloy. Therefore, it is of great significance to analyze and study the oxidation behavior for characterizing the effect of oxidation on mechanical properties.

In this work, the oxidation behavior of Ti–6A1–2Mo–1.5Cr–2Zr–2Sn–2Nb alloy including the color changes, phase composition and morphology of oxidation layer are studied. Meanwhile, the tensile properties of the oxidized alloy are investigated, and a corresponding relation between oxidized color changes and tensile properties has been established to assess the effect of oxidation on the performance of the alloy. Additionally, an oxidation model is proposed for predicting the diffusion distance of oxygen, which could be used to estimate the service life of the titanium components.

Table 1
The corresponding relationship between oxidized color and performance of titanium alloy after welding process (AWS D17.1) [15].

Color scale	Weld class		
	Class A (most critical)	Class B (moderately critical)	Class C (least critical)
Bright silver	Acceptable	Acceptable	Acceptable
Silver	Acceptable	Acceptable	Acceptable
Light straw	Acceptable	Acceptable	Acceptable
Dark straw	Acceptable	Acceptable	Acceptable
Bronze	Acceptable	Acceptable	Acceptable
Brown	Acceptable	Acceptable	Acceptable
Violet	Reject	Acceptable	Acceptable
Green	Reject	Acceptable	Acceptable
Blue	Reject	Reject	Reject
Gray	Reject	Reject	Reject
White	Reject	Reject	Reject

2. Experiment

The alloy used in the present study with the nominal composition of Ti–6A1–2Mo–1.5Cr–2Zr–2Sn–2Nb was provided by Western Superconducting Technologies Co., Ltd. (WST) in billet form (390 × 255 × 125 mm). The chemical analysis showed that the billet composition was in good agreement with nominal composition (Table 2). The alpha/beta transformation temperature of the billet was identified as 970 °C by metallographic techniques. The as-received alloy was forged in β phase region for obtaining the fine basket-weave microstructure. The initial microstructure consists of lamellar alpha phase with random orientation in length of 10–40 μm and width of 2–3 μm, as displayed in Fig. 1. The microstructure has excellent combination of mechanical properties [26].

To investigate the oxidation behavior and characterize the oxidized color changes, the oxidation treatment was performed in air at 100–1000 °C with an interval of 50 °C, followed by air cooling. For such alpha + beta titanium alloy, the solid solution treatment is one of the most common heat treatment. Generally, the solution time for the titanium alloy is about 2 h [17,22]. As a consequence, the oxidation time in this study was set as 2 h.

Before the oxidation treatment, the rectangular specimens with dimension of 70 × 25 × 5 mm were mechanically polished and ultrasonically cleaned. Previous research has shown that the oxidized color would be influenced by the surface condition [11]. For eliminating the influence, in present study, the specimens have been polished before oxidation treatment, and its surface roughness can reach about 0.8 μm. The initial specimen presents a lustrous silvery white color, as presented in Fig. 2. Two kinds of electric furnaces with different resistance wires were employed in the oxidation treatment to accurately and precisely control the oxidation temperature in a wide range (100–1000 °C). In the temperature range of 100–700 °C, one electric furnace (N60/85HA) with Fe–Cr–Al alloy resistance wire was adopted; meanwhile, the other electric furnace (SX2-10-12) with Ni–Cr alloy resistance wire was used above 700 °C. In addition, both electric furnaces were installed intelligent temperature controllers for monitoring the temperature changes.

After the oxidation treatment, the optical microstructure (OM) for cross section of the oxidized specimens was got by Olympus/PMG3 optical microscopy for observing the morphology and measuring the thickness of oxidation layer. Meanwhile, X-ray diffraction (XRD) analysis was carried out for characterizing the phase composition of the oxidation layer by X'Pert MPD Pro X-ray diffractometer (PANalytical, the Netherlands, Cu K radiation, λ = 0.154056 nm).

Table 2
The tested chemical composition of Ti–6A1–2Mo–1.5Cr–2Zr–2Sn–2Nb alloy (in wt.%).

Ti	Al	Mo	Sn	Nb	Zr	Cr	Si
Bal.	6.44	3.05	2.27	2.03	2.26	1.69	0.061