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Effect of thermal exposure on the room temperature tensile properties of Grade 2 titanium

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ABSTRACT

During exposure to the atmosphere at elevated temperature an α stabilised region develops on the surface of titanium alloys, the thickness of which is dependent on the temperature and cumulative time of exposure. In this research, samples of ASTM Grade 2 titanium were treated at a range of temperatures and times to investigate the effects of surface oxygen contamination on tensile properties. The stabilised α region that developed during the heat treatment was brittle and cracked easily after small amounts of strain, however these cracks were mostly arrested at the boundary between the transformed region and the bulk metal. The tensile strength of the heat-treated samples was compared to predicted values that assumed the transformed α -region did not contribute to the load bearing area. The comparison is reasonable when predicting the strength of samples treated at temperatures below the bulk α - β transition temperature but has limitations when applied to higher temperatures.

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1. Introduction

Oxygen is a common and useful alloying addition in titanium alloys, stabilising the hexagonal close packed (α) phase and providing solid solution strengthening without seriously compromising the corrosion resistance [1]. At typical bulk analysis levels (<0.5 wt.%), the tensile strength of titanium increases linearly with oxygen concentration at the rate of approximately 120 MPa per 0.1 wt.% O at the expense of a small amount of ductility [2,3]. However, exposure to atmospheric conditions during high temperature processing or during in-service applications, results in the formation of an oxygen concentration gradient with locally high oxygen levels at the exposed surface of the component. During treatment at elevated temperatures, deeper to the oxide layer, an oxygen diffusion zone (ODZ) exists where the oxygen concentration is higher than that of the bulk alloy, but lower than that required for oxide formation. Together, the oxide layer and the ODZ are often referred to as an ' α -case'.

The dominant oxide which results from thermal processing in air has consistently been shown to be rutile (TiO_2), although a number of minor species may contribute to the oxide depending on the exact alloy/atmosphere combination [4–7]. The oxide is non-

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protective against further oxidation due to its porous structure and tendency to fracture and flake from the substrate [8]. Consequently, the thickness of the α -case that forms during heat-treatment or processing depends on the temperature and cumulative time of exposure. Pure titanium undergoes an allotropic phase transformation at 883 °C [2] between the low temperature HCP (α) structure and the high temperature BCC (β) structure. It is somewhat unclear if there is a difference in the temperature dependent diffusivity of oxygen when comparing the data for α and β phases due to large variations in available data [9]. However, at the oxide/ODZ boundary the locally high oxygen concentration results in complete α phase stabilisation even at temperatures much higher than the bulk β transus temperature (T_{β}) . For this reason there should be little difference in the mechanisms of α -case development at temperatures above or below the bulk $T_{\rm B}$ although this does not seem to have been verified experimentally. Although nitrogen is present during heat-treatment in air and nitrogen affects the physical and mechanical properties of titanium, it has been shown that the amount of nitrogen that diffuses into the bulk alloy is small in comparison to oxygen [5,7].

The microstructure of the titanium alloy can be influential in both the mechanisms of α -case formation and the effects it has on mechanical properties. It has been shown that for a given near- α alloy the α -case penetrated deeper into the sample when a bimodal rather than a lamellar microstructure was present [10]. Also, oxygen penetration was found to have a more significant effect on hardening the bimodal structure.

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Table 1Composition of as-supplied Grade 2 titanium.

	0	С	N	Fe	Ti
wt.%	0.154	0.016	0.0097	0.11	Balance

Table 2Mechanical properties of the as-supplied, Grade 2 (average and standard deviations from 5 samples).

	Average	SD
0.2% YS (MPa)	428.3	10.0
UTS (MPa)	548.0	1.72
ε_{f} (%)	19.3	1.2

Traditionally the α -case is considered to have an undesirable effect on mechanical properties and unless innovative techniques are used to prevent excessive oxidation [11,12] the α -case is generally removed by a machining operation before the component is put into service [13]. For some applications however, including increased wear resistance [4] and the reduction of hydrogen absorption [1], a surface oxide or significant ODZ may be desirable. In these later cases it is important to understand the relationship between the surface structure of the alloy and the bulk mechanical properties.

This paper investigates the effect of α -casing on the room temperature mechanical properties of a commercially pure titanium alloy (ASTM Grade 2). In particular, it looks at these effects in small samples where the α -case can contribute a significant proportion of the cross-sectional area and aims to establish what, if any, thickness of α -casing can be present without compromising tensile properties. Additionally, it compares the effect of α -casing that is developed at elevated temperatures above the β -transus, to the effects of α -casing that develops at lower temperatures.

2. Experimental details

Commercial wrought ASTM Grade 2 titanium alloy tensile specimens, supplied in the mill-annealed condition, were subjected to oxidation, in air, at elevated temperatures. The as-supplied bulk composition and tensile properties are shown in Tables 1 and 2 respectively. Fig. 1 shows the results of DSC for the experimental alloy in the as supplied condition. The β -transition onset temperature is 870 °C and the reaction is complete by approximately 925 °C, the transition has an enthalpy of 33 J/g. Treatment conditions are shown in Table 3 and range from 800 to 1050 °C at time intervals ranging from 10 min to 3 h. These conditions were selected based

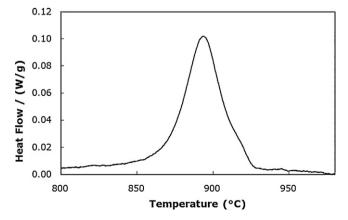


Fig. 1. DSC results for the Grade 2 titanium of composition shown in Table 2. The commencement of the β transus is 870 °C and the reaction is complete by approximately 925 °C.

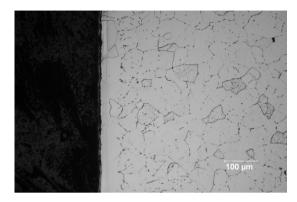


Fig. 2. Microstructure present after treatment at 850 °C for 120 min.

on preliminary trials, to produce a similar range of α -case depth for temperatures both below and above T_{β} . While the table reports both actual and nominal α -case thickness, actual thickness measurements are used in the remainder of this paper. All treatments were performed in a laboratory type, air-circulated furnace before removal and subsequent air cooling. Tensile testing was done at room temperature in accordance with ASTM Standard E8/E8 M [14]. Tensile bars had a gauge diameter, length and fillet radius of 3 mm, 20 mm and 3 mm respectively. Tensile testing was performed using an Instron 5584 electromechanical testing machine at a crosshead speed of 1.2 mm/min with elongation recorded using a knife edge extensometer (15 mm in length).

Following heat-treatment and tensile testing, samples were prepared using standard metallographic techniques and etched in a solution of 5HF, 30HNO $_3$ and 65H $_2$ O $_2$ (vol%). Microstructural observations were carried out using optical and scanning electron microscopy and the α -casing depth was determined though a combination of microstructural observations and the analysis of microhardness measurements. To investigate the mechanical integrity of the α -casing during early stages of deformation, a series of interrupted tensile tests were performed on selected samples, involving straining the samples to approximately 0.1% before unloading the specimen and preparing it for standard metallographic analysis.

3. Results and discussion

3.1. Characterisation of microstructure

Micrographs of typical samples treated below and above T_{β} are shown in Figs. 2 and 3 respectively. In the samples treated below T_{β} , the microstructure is comprised of small equiaxed grains of α titanium as expected for the mill-annealed condition. At the surface of these samples the α -case is visible as an area that is relatively free of equiaxed grain boundaries. In the samples treated above T_{β} the sample has undergone a full transformation into the β -phase field during processing, and upon cooling, colonies of aligned α -laths have developed inside of each of these grains. The α -case is easier to detect in these samples because it has a higher degree of contrast when compared to the bulk microstructure. It consists of a layer of oxide at the surface and deep to this layer there is a well demarcated oxygen-rich region that contains no transformed lath-like α .

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