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TEM Observation of Oxide Scale Formed on a Ti–Al–Zr Alloy Oxidized at 360 ^{o}C in an Alkaline Steam

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Titanium and its alloys are considered to be among the most promising engineering materials, due to a unique combination of high strength-to-weight ratio, melting temperature and corrosion resistance. Working safely for prolonged periods in high-temperature and high-pressure environments, titanium alloys and its products are required to have excellent oxidation resistance and corrosion resistance. Recently, the study of oxidation behavior was mainly concentrated at high temperature (>700 ^{o}C), and most detailed studies have focused on the surface of bulk oxide TiO_2, due to its importance in heterogeneous catalysis and photocatalysis. In order to simulate the oxidation behaviour of heat-conducting tubes in nuclear reactors, this study focused on the oxide films formed on Ti-Al-Zr alloy oxidized in an alkaline steam containing LiOH at 360 ^{o}C. The oxide distribution and structure were investigated by cross-sectional TEM observations and energy dispersive spectroscopy (EDS) measurements.

Figure 1 is the cross-sectional TEM image showing the morphology of the oxidized sample and the interfacial microstructure of the Ti-substrate and oxides. Four different layers were observed, including Ti-substrate, Ti_2O, TiO and TiO_2 anatase surface layer. The thickness of Ti_2O, TiO and anatase-TiO_2 is approximately 50, 100 and 400 nm, respectively. The corresponding selected area electron diffraction patterns (SAED) obtained from these four layers were showed in Figs. 1(B) (C) and Fig. 2. The tetragonal anatase-TiO_2 is the major phase near the surface of the sample, and as the depth increases, the TiO (NaCl crystal structure type) and Ti_2O (hexagonal) appear consequently, suggesting that oxide scale on titanium alloys grows through oxygen diffusion from the environment side to the oxide/metal interface. The microstructure of the interface region between the oxide layers and the titanium substrate is shown in the high-resolution TEM images (Figure 3). There are few of traces of imperfections, such as voids, precipitates, or disordered regions observed at the interfaces of Ti_2O/TiO and TiO/TiO_2-anatase. High-resolution TEM images (not shown here) of TiO and anatase-TiO_2 layer show that both oxide films are highly crystalline, and no other second phases were identified. EDS measurements indicate that the O content increases as across from the substrate to outer layer. The quantitative analysis of these EDS spectra indicated that the Ti/O ratios in these four layers are close to these of Ti, Ti_2O, TiO, and TiO_2, consistent with the TEM observations. Anatase-TiO_2 were found near the surface; while TiO and Ti_2O formed at greater depths from the surface, consistent with the fact that oxide scale on titanium alloys grows through oxygen diffusion from the environment side to the oxide/metal interface. These results have significant implications in understanding the oxidation and corrosion behaviors of titanium alloys used in alkaline environment.

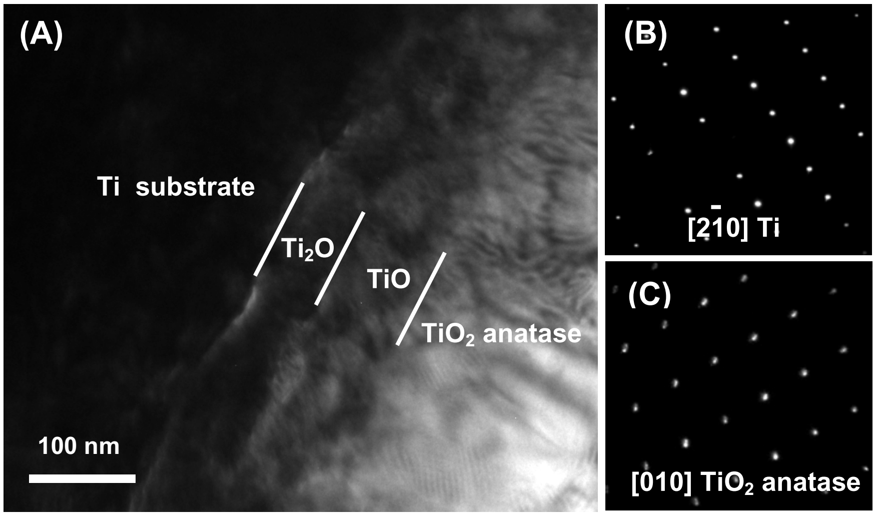

Figure 1 (A) Bright-field cross-sectional TEM image of Ti-Al-Zr sample oxidized at 360 ^{o}C in an alkaline steam, and the selected-area electron diffraction patterns of Ti substrate (B) and TiO_2 anatase surface layer (C).

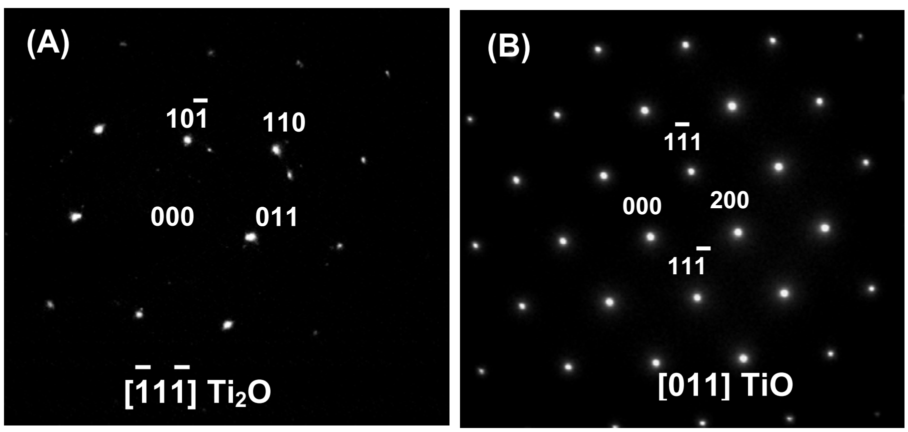

Figure 2 Selected-area electron diffraction patterns of Ti_2O layer (A) and TiO layer (B).

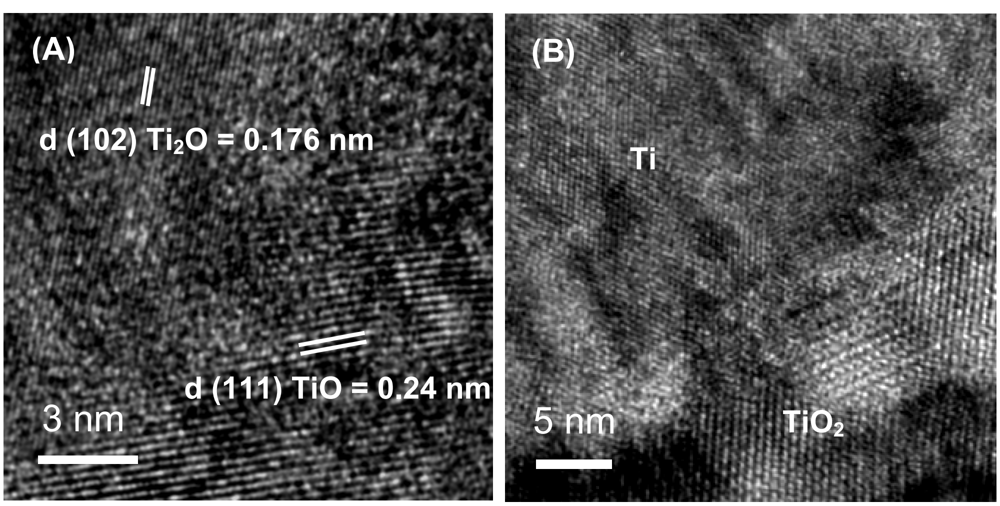

Figure 3 High-resolution TEM images showing the interfacial structure of Ti/Ti_2O (A) and TiO/TiO_2 (B).

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