Structural Characterization of Aluminum Oxidation due to Exposure to Hyperthermal Atomic Oxygen

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Hyper-thermal atomic oxygen (AO) is the primary species in the Low Earth Orbit (from 200 to 700 km above the earth), hastening premature failure to materials. We studied AO oxidation of aluminum since aluminum and its alloys are used as structural components on space vehicles, as well as coating material to enhance reflectivity [1, 2]. Al(100) single crystal was maintained at 220°C and exposed to a 5 eV AO beam with a total fluence of 8×10^{19} atom·cm⁻². The AO beam for the exposure of samples was created by detonation of O₂ with a pulsed CO₂ laser. Cross-sectional transmission electron microscope (TEM) samples were prepared that could be examined in both TEM and scanning electron microscope (SEM). For observations of the growth of the oxide scale, Al(100) single crystal was thinned by Ar⁺ milling at low angles, and then exposed to the 5 eV AO source at room temperature for incremental time periods and taken out of the AO chamber for characterization by *ex situ* TEM and SEM in order to observe the evolution of the oxide scale.

Fig. 1 is images of structural investigations of the cross-sectional samples of Al(100) oxidized at 220° C with a fluence of 8×10^{19} atom·cm⁻². The SEM image on **Fig. 1** (a) reveals that two types of interfaces: smooth (noted by arrows) and rough areas, which exist along the interface of the oxide scale/Al(100) substrate. The smooth area shows a thin, uniform oxide layer on the Al crystal. The rough interface may be formed from the imperfect area of Al substrate, where AO diffuses through oxide layer to react with Al. The high-resolution TEM (HRTEM) image shown in **Fig. 1**(b) reveals that the AO creates a rough interface, where oxide/metal interfaces along Al(111) preferentially formed. **Fig. 1**(c) and (d) are HRTEM and high-angle annular dark field images from the same area of the sample, revealing a uniform amorphous oxide layer with a thickness of 5.7 nm.

Fig. 2. is a set of SEM images of the Al(100) oxidized in AO at room tempera-ture as a function of exposure time. The AO exposure time was 5.5, 60 and 230 minutes with corresponding fluences of 6×10^{17} , 6.6×10^{18} and 2.5×10^{19} atom·cm⁻², respectively. **Fig. 2.** (a) is an image of the Al(100) surface before exposure to AO. The large, flat terraces were produced by low angle Ar⁺ milling. After 5 minutes of AO exposure, a severe surface roughness was produced (see **Fig. 2.** (b) and its magnified image on **Fig. 2.** (e)). The unusual surface roughness relates to an intriguing initial mass loss, which requires further studies. Upon further AO exposure, the surface became smoother (**Fig. 2.** c and d). TEM studies reveals a thin, amorphous alumina layer developing on the surface of Al(100).

References

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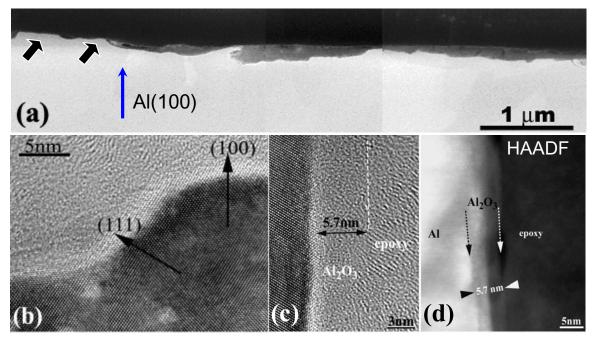


Fig. 1. Cross-sectional structural investigations of the oxide scale that formed on Al(100) at 220°C. (a) SEM image of the interface, (b) and (c) HR-TEM images, and (d) high-angle annular dark-field (HAADF) image.

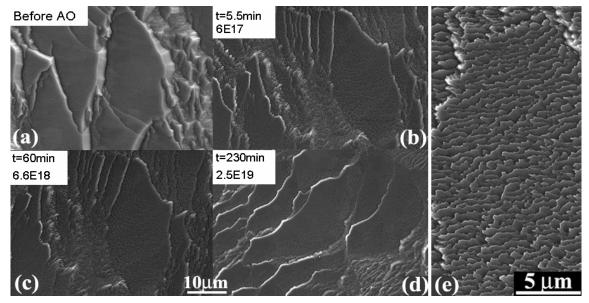


Fig. 2. A time sequence of SEM images revealing the surface evolution of Al(100) when it is exposed to 5eV atomic oxygen at room temperature. Fig.2 (e) magnified image of (b) where a significant surface roughening is noted.