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Effects of AC Pretreatment on the Formation of Anodic Titanium Oxide Films

Masaki YAMAZAKI,* Mitumoto NAKAGAWA,** and Hiroshi NOZAKI*

*Institute of Industrial Science, The University of Tokyo, Roppongi, Minato-ku, Tokyo

**Department of Applied Chemistry, Faculty of Engineering, Tokai University, Shibuya-ku, Tokyo

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Recently, we have reported the formation of electrolytic titanium oxide films in an aqueous solution of barium hydroxide.[1] In this formation, the pretreatment of titanium was done by applying an AC current to titanium in an aqueous solution of sulphuric acid. It has been found that the AC pretreatment is effective in increasing the bath voltage in the anodization of titanium; this pretreatment is also useful in producing good insulating films on the surface of titanium. It would be of interest to know whether a good dielectric film could be formed on titanium by using the AC pretreatment in connection with electrolytic capacitors. Thus, in this work we will report the effects of the AC pretreatment of titanium on the formation of anodic oxide films in an aqueous solution of phosphoric acid.

Two titanium electrodes, 50 mm × 20 mm × 1 mm, were prepared from ST-40 titanium (H, <0.015%; O, <0.20%; N, <0.05%; Fe, <0.20%). One of the electrodes was mechanically polished, while the other was pretreated in a 16 wt% H_2SO_4 solution at 25°C, using AC (50 Hz) at 50 mA/cm², for 2 hr.[1] After these procedures, the surfaces of both electrodes were examined with an optical microscope. The microphotographs obtained are shown in Fig. 1A for the mechanically-polished electrode and in Fig. 1B for the AC-pretreated electrode. Figure 1 shows that the electrode is partially dissolved in the H_2SO_4 solution by the AC pretreatment. This fact was also confirmed by measuring the weight decrease in the titanium electrode during the AC pretreatment.

The anodizations of both the electrodes mentioned above were carried out under the same conditions; the electrodes were anodized in a 13 wt% aqueous solution of phosphoric acid at 25°C with a constnat current density of 3 mA/cm². In these anodizations, the growth of the voltage with the time was examined. The results obtained are shown in Fig. 2. It is obvious from Fig. 2 that the voltage for the AC-pretreated electrode increases rapidly with the time up to 180 V, while the voltage for the mechanically-polished electrode hardly increases at an over 50 V. These results indicate that the AC pretreatment is useful in producing anodic titanium oxide films which have good insulating properties.

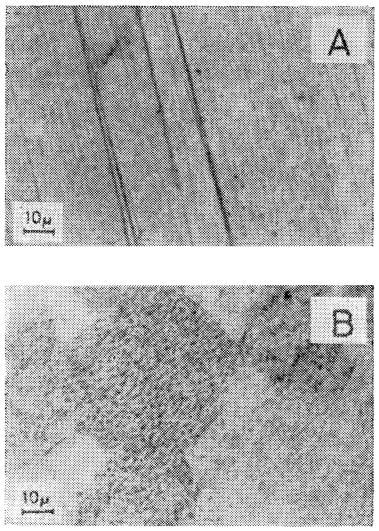

Fig. 1. Microphotographs of the surfaces of titanium electrodes.
A: Mechanically-polished electrode.
B: AC-pretreated electrode.

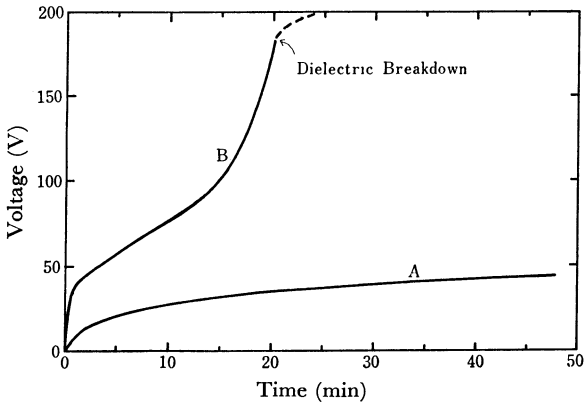

Fig. 2. Bath voltage-time curves for the anodization of titanium at a constant current density of 3 mA/cm².
A: Mechanically-polished electrode.
B: AC-pretreated electrode.

1) a) M. Yamazaki and H. Nozaki, *J. Electrochem. Soc.*, **118**, 400 (1971). b) M. Yamazaki and H. Nozaki, *Kogyo Kagaku Zasshi*, **74**, 1265 (1971). c) M. Yamazaki and H. Nozaki, *J. Phys. Chem.*, **75**, 1279 (1971).