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Laser damage mechanism of porous Al_2O_3 films prepared by a two-step anodization method

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ABSTRACT

Pure aluminum films were deposited on a B270 glass by electron beam evaporation technique. These aluminum films, which were used as anode, were put in sulfuric acid and oxalic acid to prepare porous alumina films using a two-step anodization method. The microstructure and laser damage characteristics of the alumina films were then tested. Results show that the microstructure of the alumina films formed in sulfuric acid and oxalic acid were vertical (cylindrical) pores with different diameters. The laser-damaged spot of the porous films was formed by innumerable small damaged pits with no mutual influence. Films prepared in different acids reveal different damage characteristics and reflect different mechanisms.

1. Introduction

In compact films, laser damage is almost caused by impurity absorption when the laser irradiation has a pulse width in the nanosecond- or picosecond-level. Many damaged pits are discretely distributed in the damaged spot. The damage is distinctly probabilistic, that is, laser damage happens because of impurity absorption once the impurity locates in the laser irradiation area. From experiments, it can verify that porous films commonly have a high laser-induced damage energy threshold [1,2]. At present, the general viewpoint regarding the mechanism is that the porous microstructure partially relieves the stress caused by absorbing the laser energy. However, for porous films, the data from damage experiments are not enough and the damage mechanism is seldom reported. Previously, we have reported the laser-induced damage characteristic of porous alumina optical films which were prepared in phosphoric acid [3]. Different from usual compact films, the laser-damaged spot of porous films is formed by discrete damaged pits with high density and no mutual influence.

In this current article, the microstructure and laser-damaged spot of porous alumina films prepared in different acids were tested. Laser damage traits of the porous films were emphasized and the damage mechanism was analyzed. Films prepared in different acids reveal different damage characteristics and reflect different mechanisms.

2. Experimental

2.1. Sample preparation

Porous alumina films were prepared based on aluminum thin films, which were deposited on a glass substrate. The aluminum thin films were deposited by electron beam heating in a physical evaporation deposition system [4]. Pure aluminum (purity > 99.999%) was used as coating material. Base pressure in the deposition chamber was about 3.0×10^{-3} Pa, and working pressure was about 6×10^{-3} Pa. Deposition beam current was 380 mA. Source–substrate distance was 18 cm. Film thickness was monitored by the deposition time (about 60 s).

Porous alumina films were prepared by the anodization method. The aluminum films mentioned above were used as anode and a cleaned aluminum sheet was used as cathode. Anodization was carried out in a 1.0 mol/L sulfuric acid solution and a 0.2 mol/L oxalic acid solution at about 5 °C with a DC constant oxidation voltage (18 and 30 V, respectively). The first anodization lasted for 0.5 h and the second for 1 h. After the first anodization, the strip-off process was carried out in a mixture solution (6% H_3PO_4 + 1.8% H_2CrO_4) at 60 °C for 10 min. After the second anodization, the bottom of the pores was subsequently opened by 5% H_3PO_4 at room temperature for 30 min. The current profile was monitored to verify whether the alumina layer became porous or not and whether it was exhausted or not [4]. The surface and cross-section of the films were then examined with a field emission scanning electron microscope.

2.2. Laser-induced damage test

Laser-induced damage experiment was performed in the "1-on-1" regime according to ISO 11254-1.2. The experimental set-up for laser

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damage is shown schematically in Ref. [5]. The laser pulse width was 12 ns and the laser wavelength was 1064 nm. Ten sites of the sample were exposed at the same fluency and the fraction of damaged sites was recorded. A Nomarski microscope with 100× magnification was used to determine whether radiation sites were damaged.

3. Results and discussion

3.1. Scanning electron microscope results of porous alumina films

Fig. 1(a) shows that the film prepared in sulfuric acid is hexagonal close packed. The pore, which is regular round, is in the center of the close-packed unit. The aperture is 16–25 nm and the distance between pores is 50 nm. Some of the units are hexagonal close packed. The homogeneity of the distribution is inferior to that of Al substrate films. From the section image [Fig. 1(b)], the pores are parallel and the columnar pores are perpendicular to the glass substrate. Average thickness is about 742 nm. The barrier layer is from 36 to 50 nm. Macroscopically, the alumina films are transparent.

Fig. 2(a) presents the structure of the film prepared in oxalic acid. The pores are not regular round because the time for chambering was short. The distance between pores is 100 nm. From the section image [Fig. 2(b)], the pores are parallel to one another while the columnar pores are perpendicular to the glass substrate. Average thickness is about 1250 nm. Macroscopically, the films prepared in oxalic acid are straw yellow. Alumina films with great quality can be prepared

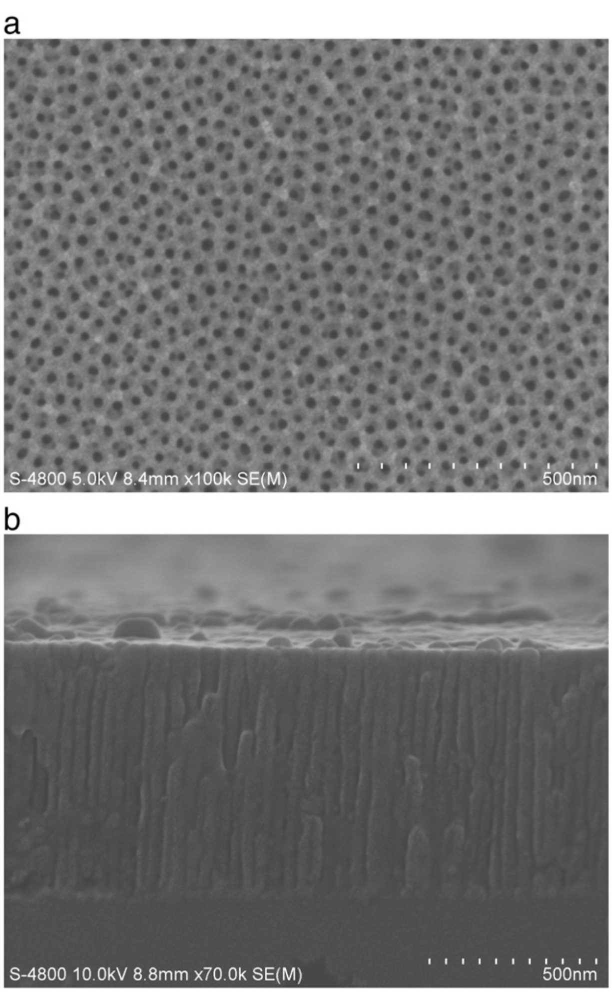

Fig. 1. SEM image of the surface and cross-section of the film in 1 mol/L sulfuric acid with 18 V. (a) Surface morphology. (b) cross-section morphology.

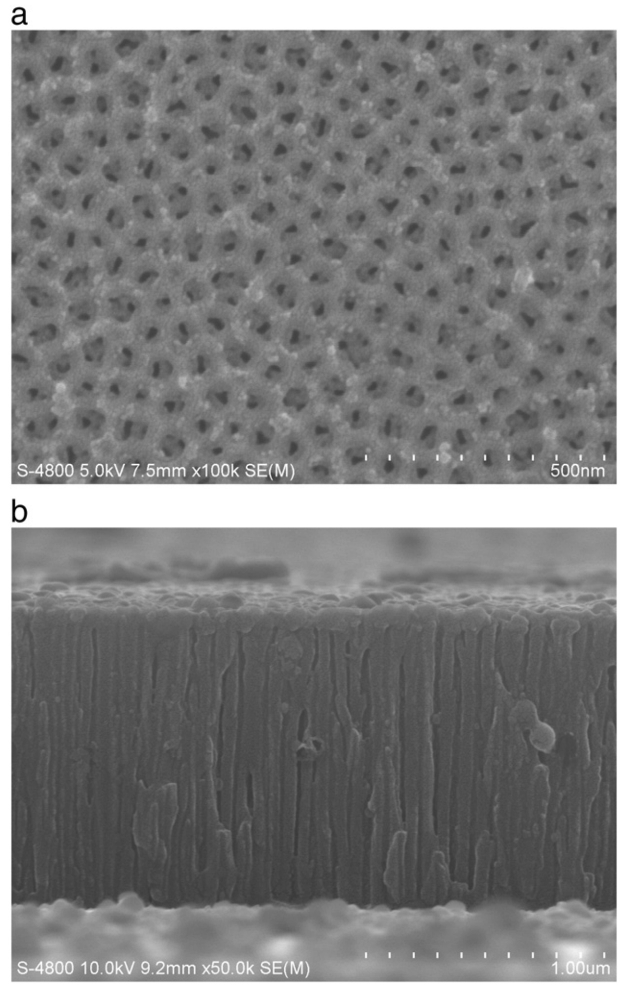

Fig. 2. SEM image of the surface and cross-section of the film in 0.2 mol/L oxalic acid with 30 V. (a) Surface morphology. (b) cross-section morphology.

in 0.2–0.3 mol/L oxalic acid with 20–30 V. The repetition of the experiment is good.

3.2. Damage morphology detection

Due to the high-purity material, laser damage was not caused by the absorption of impurity but by the material itself, as a result, the damage is not distinctly probabilistic.

3.2.1. The damage traits in sulfuric acid

Fig. 3 presents the image of the laser damage in sulfuric acid. The laser-damaged spot of the films was formed by discrete damaged pits with high density and no mutual influence. Most of the damage pits are small and damaged independently. As Fig. 3(b) shows, there is an obvious boundary of damage spots in high laser density. Inside the boundary, the pit density is high, while outside, the pit density is low. Pit density increased with the increase in laser energy density. This is apparently different from the pit density of usual compact films [6,7]. Pit density in the damage spot of the compact films is low, but the pit size is big because of the single damage source. The mutual effect of each pit is obvious. Every damage source works together to bring greater laser damage.

Fig. 1 shows that aperture size and distance between them are not equal to one. The electric field distribution of local nonuniform pore in the homogeneous distribution area is calculated by the finite-difference method (FDM). Fig. 4 shows that the peak value of the electric field intensity around pores with a small aperture is enormous.