The Effects of Anodization Treatment on the Microstructure and Fatigue Behavior of 7075-T73 Aluminum Alloy

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The fatigue behaviors of 7075-T73 alloy with/without anodization/sealing treatment were investigated in this study. The microstructure of different samples was analyzed by electron microscopy. As the experiments, the microstructure were observed from the anodized/sealed samples to indicate that precipitates trapped at subsurface near film/metal (f/m) interface would partly dissolved and the deformed matrix existed high fractions of high angle grain boundaries. The bare 7075-T73 alloy samples achieved fatigue strength of 225 MPa and the anodized/sealed samples enhanced their fatigue strength at 10^7 life cycles with 10–20 µm film thickness. The fractured anodized/sealed sample showed fine striation spacing than bare sample after subjected to low stress amplitude. [doi:10.2320/matertrans.M2014121]

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1. Introduction

AA 7075 aluminum alloys contain high amounts of Zn, Mg and Cu. After T6 and/or T73 heat treatments, the alloy will develop excellent tensile strength along with proper elongation. There are complex intermetallic compounds within the matrix of the 7075 alloy. The precipitates and/or intermetallic compounds acquire a specific morphology due to the application of the heat treatment and the manufacturing process. These intermetallic compounds and the fine precipitates existing in the aluminum matrix greatly influence the anodizing performance and mechanical properties of AA7075 aluminum alloys.1–4)

The coating of aluminum alloys with anodic aluminum oxide (AAO) films can protect the alloy from corrosion in severely corrosive environments. However, localized dissolution tends to occur around intermetallic particles and precipitates during anodization. The oxidation rate is faster with large intermetallic particles such as the Mg2Si, Al2Cu, Al2Cr, ß-AlMg and AlZnMg than in the aluminum matrix, meaning that the particles could dissolve into the electrolyte during the anodizing treatment.5,6) On the other hand, immobile elements such as silicon will form silicon containing particles that will be trapped in the AAO film. The effects of intermetallic particles on the fatigue life cycles of high strength 7075 alloys are of concern because this material is commonly used in industrial applications.

Precipitates and intermetallic compound particles tend to be trapped in AAO films during the anodization of the aluminum alloy. Pitting that preferentially occurs at the interface between the silicon/iron particles and reduces the corrosion resistance of the films. Effect of anodization on the fatigue life of 7050-T7451 alloys has been studied and discussed by Sharzad et al.7) They found that the inclusion particles trapped in the AAO film had a significant effect on the formation of corrosion pits, which accelerated fatigue crack initiation. As a result, the fatigue life of the aluminum alloy was reduced. Fratila-Apachitei et al.8) utilized TEM to observe the particles trapped in anodized alumina oxides on an Al–Si alloy. The alumina film was encroached upon by silicon particles in association with a non-uniform porosity. In addition, gas-filled voids above the silicon particles were also observed to influence the morphology of the enveloping porous AAO film, pore termination above the particle and pore branching and deflection around and beneath the particles. Mukhopadhyay et al. used electron probe microanalysis (EPMA) to examine the constitution of intermetallic particles in the matrix. They found particles containing an iron rich phase that could be trapped in hard anodic coating films to locally inhibit the formation of anodic oxide films.9)

The behavior and mechanisms of sealing treatment, which affect the quality of AAO film have been examined in many studies.10–14) The basic reaction in the sealing process is a hydrothermal reaction which turns amorphous alumina into crystalline boehmite, AlO(OH). The four steps of the sealing process have been described by Lopez et al.,15) starting with the filling of the pores by the sealing solution, plugging the pore mouths, the formation of acicular pseudoboehmite crystals and a compact intermediate layer on the surface and finally the growth of hydrated alumina crystals during the sealing process. The entrapped intermetallic compound particles and/or precipitates in the AAO films are affected by the sealing process, which alters the quality of the resultant AAO films.

In this study, we examine the relationship of the number of cycles to failure versus stress amplitude for 7075-T73 samples with/without anodization treatment. After sealing of the coated AAO film, there was improvement in the corrosion resistance of the 7075-T73 alloy.16) However, there has been no published study discussing the effects of the matrix structure on the relation between the number of cycles to failure versus the stress amplitude of an anodized 7075-T73 alloy. We provide experimental results along with microstructure observations to explain the effects of microstructure on changing fatigue behavior on a given stress amplitude.

2. Experimental Procedure

2.1 Materials

The chemical composition of the material used in this

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study, 7075-T73 extruded bars (15 mm in diameter and 150 mm in length) is listed in Table 1. After annealing at 415°C for 120 min, all samples were removed for the purpose of carrying out the T73 treatment which involved solution treatment at 475°C for 90 min, followed by rapid quenching in water and finally two-step age hardening at 110 and 175°C for 480 min.

2.2 Specimen preparation

The bar-shaped samples were machined to achieve the tensile test with a gage diameter of 6 mm, according to the ASTM B557 specifications,\(^1^7\) and the rotating-bending fatigue test was carried out following the JIS Z2274 specifications,\(^1^8\) with a gage diameter of 8 mm.

After the tensile test, the 7075-T73 alloy samples received 591 (9.8) MPa (ultimate tensile strength, UTS), 564 (12) MPa (yield strength, YS) and the elongation which was 11.5 (0.4)\(^%\) was calculated from the average of at least ten tests. Deviations are listed in the parentheses. For the anodized/sealed sample, the tested UTS is 590 (2.6) MPa, YS is 560 (6.2) MPa and the elongation which was 11.5 (0.4)\(^%\) was calculated from the average of five tests. A strain rate of \(1.87 \times 10^{-3} \text{s}^{-1}\) was used in the tensile test.

2.3 Surface treatment

Before anodization, all samples were polished to have surface roughness about \(R_{a} \leq 0.1 \mu m\), followed by being dipped into methanol and ultrasonic vibration. The specimens were initially degreased by immersed in an alkaline solution (5 mass% \(\text{NaOH}\) at 60°C for 30 s and then water rinsing for 1–2 min. For the pickling processing, specimens were submerged in an aqueous solution of \(\text{HNO}_3\) (30 vol%) for 90 s at room temperature followed by 1–2 min of rinsing in water. The anodization was conducted at 15 mA cm\(^{-2}\) at 15°C for 900 s in a 15 mass% sulfuric acid solution. Furthermore, the anodized samples were sealed in hot water at 95°C for 1200 s. After sealing, the thickness of the AAO film was measured by SEM inspection.

2.4 Fatigue test

The fatigue tests were carried out in air at a frequency of 40 Hz. All tests were conducted under load-controlled conditions using an Ono’s rotary bending fatigue testing machine with a maximum momentum of 100 N·m. The tests were stopped if the specimen did not fail after \(1.0 \times 10^7\) cycles.

2.5 Specimen inspection

After the rotating-bending test, the fractured samples were removed and cut in a longitudinal direction for optical micrograph (OM) observation. The cut sample was first polished with #2000 abrasive sandpaper followed by further polishing using an alumina powder slurry 1 \(\mu\)m and then colloidal silica (0.03 \(\mu m\) prior to OM observation and intermetallic particle count measurement.

In preparation for TEM observation, the anodized/sealed sample was milled with a Versa 3D FEI Dual-Beam Focus Ion Beam (FIB) device equipped with a Ga\(^+\) beam source. A \(5 \times 5 \mu m\) and 100 nm thick piece was cross-sectioned from the tested sample using the FIB technique. The TEM (JEOL-2000 FX II) operated at 160 kV was used to observe the microstructure of the sample.

Different fractured samples were also observed by Field Emission Scanning Electron Microscopy (FE-SEM) to get their specific fractured surface. Prior to analysis, pieces of the specimens were cut longitudinally from the fractured samples then polished with \#2000 abrasive sandpapers followed by electron polishing using an electrolyte of 730 mL ethanol, 78 mL perchloric acid, 100 mL 2-butoxy ethanol and 90 mL deionized water at 27 V/1.5 A for 10 s. The samples were examined with a SEM (JEOL 6330) to reveal crystallographic contrast. Electron back scatter diffraction (EBSD) pattern examination was carried out at 20 keV with a probe current of 0.1 \(\mu A\) and a working distance of 15 mm. The diffraction data were acquired as an orientation map from raster scans of 600 \(\times\) 300 \(\mu m\) with step sizes of 1 \(\mu m\).

Samples were prepared for hydrogen testing by cutting pieces about 0.2–0.5 grams from the bare and anodized/sealed samples at locations close to the subsurface and the core. Before testing, the samples were dry-polished with abrasive sandpaper then cleaned with acetone. The hydrogen content was detected by using a hydrogen analyzer (Horiba, EMGA-521). This analyzer used inert gas (Ar) fusion and impulse furnace-column separation-thermal conductivity detection (for more details about this procedure please refer to reference).\(^1^9\)

3. Results and Discussion

3.1 Microstructure observation

According to the tensile test results, the anodized/sealed samples gained a relatively low elongation compared to the bare samples. The mechanical properties of different samples were affected by the alteration of the microstructure by the anodization process, which will be discussed in more detail in later sections.

The coarse intermetallic compound particles apparently located at the grain boundaries were aligned along the longitudinal (extrusion) direction of the bar sample, as shown in Fig. 1(a). Some subgrains are visible and likely formed due to thermal-activated recrystallization in the solution treatment, which was affected by soluble Mg and Zn atoms in the Al matrix.\(^2^0,2^1\)

The intermetallic compound particles could amount to \(363 \text{ counts mm}^{-2}\) according to reference.\(^1^6\) The particles that ranged in size from 6–10 \(\mu m\) mostly contained Mg–Si or Mg–Zn, while those that ranged in size from 2–5 \(\mu m\) were mainly composed of Al–Si, Al–Cu or Al–Cu–Fe. Figure 1(b) shows the shape and size of precipitates that are located in the matrix of the bare sample. Some precipitates were apparently located at the grain boundaries. The needle-like precipitates

| Table 1 Chemical compositions for the 7075 aluminum alloy. |
|----------------|----------------|
| **Material**   | **Si** | **Fe** | **Cu** | **Mn** | **Mg** | **Cr** | **Zn** | **Ti** | **Pb** | **Al** |
| 7075           | 0.08   | 0.1    | 1.62   | 0.03   | 2.5    | 0.23   | 5.78   | <0.02 | <0.01 | Bal.  |
in the matrix are likely from the Al₂CuMg (S phase) while other precipitates have been reported to be comprised of Al₂Cu (θ'-phase) and Al₂Mg₃Zn₃ (T-phase).²²

3.2 S-N curves of different samples

After the rotating-bending fatigue test, the relationships of the number of cycles to failure versus stress amplitudes (S-N curves) for three batches of test samples are constructed. The S-N curves shown in the Fig. 2 clearly display the differences in the 7075-T73 samples with/without anodization treatment in terms of the changes in high-cycle fatigue life (>10⁵ cycles). Yhar²⁵) applied the following equation to get a best fit S-N curve of aluminum alloy.

\[ S = \left( \frac{E}{4\sqrt{N}} \right) \ln\left( \frac{100}{100 - A} \right) + B \ldots \] (1)

Where \( E \) is elastic modulus, \( S \) is stress amplitude, \( N \) is number of cycles to failure, and the parameters \( A \) and \( B \) are constants that are selected to fit the equation to match the data. The value for \( B \), as this is the endurance limit, which applied for this study is 225 MPa for bare sample and 240 MPa for anodized/sealed samples. The \( A \) value was the percentage of reduction in area from tensile test; 34 and 27% were obtained from testing bare and anodized/sealed samples, respectively. The fitted experimental results are shown in Fig. 2. They are applicable to predict the fatigue life cycles at different stress amplitudes lower than about 90% of yield stress.

Experimental results show that the anodized/sealed samples achieved lower fatigue strength at 10⁴–10⁵ life cycles than the bare sample. This is mainly due to a fact that crack initiation occurred relatively easily in the anodized/sealed samples. Therefore, it is important to identify the origin of fatigue cracking for the different specimens.

Figures 3 and 4 revealed the fractured surface morphologies for the bare and anodized/sealed samples, respectively. Cracks tended to be initiated in both samples at multiple sites when they were subjected to high stress amplitudes of 560 MPa, as shown in Figs. 3(a) and 4(a). Lowering the stress amplitude led to a remarkable decrease in the number of crack initiation sites on the fractured bare samples, as shown in Fig. 3(b) for 390 MPa and Fig. 3(c) for 260 MPa. The anodized/sealed samples showed notable crack initiation at multiple sites when the stress amplitude decreased to 390–400 MPa; Figs. 4(b) and 4(d). This was due to that after anodization, the emergence of Si-containing particles and/or Al–Fe particles at the film/metal (f/m) interface. The Si-containing particles trapped at the f/m interface were accompanied by a concave interface nearby. Cirik et al. also explained the particle-induced irregularity was caused by the preferential dissolution in anodization process.⁴) The irregularity (or notch effect) at the f/m interface offered more potent sites for crack initiation, which acted to reduce number of cycles to failure when the stress amplitude decreased to about 300–400 MPa. Such notch effects also led to deterioration in the tensile properties to reduce elongation from 13.4% (bare sample) to 11.5% (anodized/sealed sample with 10 µm film thickness).
Different bar samples were prepared for cross-sectional observation in the longitudinal direction with EBSD analysis, as shown in Figs. 5(a)–5(c). The fractions of different misorientation angle grain boundaries were counted and recorded from observations on different fractured samples that tested at 240 and 390–400 MPa stress amplitudes, as shown in Fig. 6. There was an increase in the fractions of high angle grain boundaries (HAGBs) in the fatigue-fractured anodized/sealed samples and an increase in the fractions of low angle grain boundaries (LAGBs) in the fatigue-fractured bare sample. During a rotating-bending test, the bare sample was subjected to a repeated shear stress to increase the storage of energy in the matrix and to form high-density dislocations, which acted to preferentially increase LAGBs within the matrix. The dislocations blocked the movement of Cu and Mg atoms at the sites of the dislocation cells. The addition of copper and magnesium to the aluminum alloy could significantly reduce the alloy’s stacking fault energy (SFE). For the alloys with low SFE such as Al–Cu, Al–Mg and Al–Zn alloys, planar slips readily occurred during deformation. Dislocations were finally blocked or pinned at the intersection (sessile jogs) and/or grain boundaries. Dynamic recrystallization would occur at the intersection and/or grain boundaries.

The fatigue-fractured bare samples tested at a stress level of 240 MPa were prepared for TEM observation. Figures 7(a) and 7(b) shows the complex structure with tangling dislocations and fine grains 100–200 nm in size, which were apparently driven by dynamic recrystallization caused from tests. The selected area diffraction pattern shown in Fig. 7(c) confirms the polycrystalline structure of the matrix in Fig. 7(b).

The microstructure of the fatigue-fractured anodized/sealed sample was apparently affected by anodization, especially at the subsurface region. We observed at location about 10 µm beneath the film/metal interface. This location was also selected to be close to fracture surface and is shown in Fig. 8. During anodization the Al₉CuMg and Al₉Mg₉Zn₃
precipitates displayed anodic electrochemical behavior with respect to the matrix.30) Coarse precipitates along the grain boundaries are partly dissolved (Figs. 9(a) and 9(b)) together with low amounts of fine precipitates remained within grains; the needle-like precipitates notably disappeared. As a consequence, early-crack propagation would be influenced by the altered microstructure on the anodized/sealed samples especially at low stress amplitudes.

Aluminum alloys are inherently taking some amounts of hydrogen in the matrix. The presence of hydrogen also plays an important role in influencing the structure of 7075-T73 alloys. Soluble hydrogen in the aluminum matrix could diffuse into AAO films during anodization to further introduce the crystallization within AAO film. Hydrogen content was slightly decreased at matrix close to f/m interface.31,32) Hydrogen content test that prepared from subsurface area and from core portion of anodized specimens respectively as listed in Table 2. The data indeed show lower hydrogen content on sample taking from subsurface than that from the core portion. Decreasing the hydrogen content in the aluminum matrix allowed the cross-slip process to continue.33) Therefore, dislocations are readily to move and accumulate at the grain boundaries leading to preferentially form HAGBs in the deformed matrix of anodized/sealed samples.

In summary, when sample was subjected to high stress amplitude, high stress induced dense dislocations moving readily along shear plans. They would interact to initiate early-crack at film/metal interface due to increasing irregularity. Therefore, fatigue life was decreased comparing with that of bare sample. There are two factors significantly affect the fatigue behavior of tested sample at low stress amplitudes. First, the anodized/sealed samples got an increase in fraction of HAGBs due to decreasing hydrogen content in matrix. Second, the precipitates (partly) dissolved in matrix and coarse precipitates notable reduced their size at grain boundary near f/m interface. These factors would affect the fatigue behavior of tested sample when they were subjected to low stress amplitudes. Lower peak stress could develop at the grain boundaries with higher tilt angles than with low tilt angles.34) As a result, the matrix at the subsurface area was toughened to resist the early-crack propagation in the anodized/sealed sample leading to increase fatigue life cycles at low stress amplitudes; the fatigue limit was raised from 225 to 240 MPa at 10^7 life cycles. In contrast, increasing AAO film thickness from 10 to 20 µm degraded the integrity...
of f/m interface to raise the effect of surface notch due to dissolution of precipitates. The fatigue strength was therefore decreased from 240 to 230 MPa as the thickness of AAO film increased.

4. Conclusion

After anodization and sealing treatment, the coated AAO film brought irregularity at the f/m interface leading to a reduction in elongation of 13.4% (bare sample) to 11.5%. When sample was subjected to high stress amplitude, high stress induced dense dislocations moving readily along shear plans. They would interact to initiate early-crack at film/metal interface due to increasing irregularity. The stress amplitude at $10^7$ life cycles for the bare samples was 225 MPa and those for anodized/sealed samples were 240 and 230 MPa for the samples with AAO film thickness of 10 and 20 µm respectively. The fatigue strength of anodized/sealed samples was improved by an increase in the fractions of HAGBs and partly dissolution of precipitates at subsurface area near f/m interface.

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