

Chemical Cleaning for the Corrosion Studies of Open-Cell Aluminum 6101 Foams

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ABSTRACT

In recent years, there has been an increasing demand for aluminum foams for aerospace, automotive and marine applications. To provide a fundamental basis for advanced engineering developments, understanding the corrosion behavior of this novel class of porous material is vital. However, determining accurate mass loss for corrosion tests of these materials is challenging due to the difficulty of removing corrosion products on a large and inhomogeneous surface. In this project, nitric acid immersion was evaluated as a chemical cleaning technique to remove corrosion products from aluminum 6101 foams (UNS A96101) with different pore sizes and surface areas and were subjected to exposure in simulated seawater (3.5 wt% NaCl solution) environments. The cleaning protocol was performed by immersing as-received and post-corroded UNS A96101 foam alloys in 68-70 wt% nitric acid solution at room temperature for various times. The same protocol was performed on the UNS A96101 bulk alloys and the results were compared to their foam counterparts. Scanning electron microscopy (SEM) was used to characterize the corroded surfaces and qualitatively determine the optimal cleaning duration.

Key words: Open-Cell Aluminum Foams, Chemical Cleaning, Immersion Tests

INTRODUCTION

Open-cell aluminum 6101 foams (UNS A96101) have seen a growth in demand for marine, automotive, and aerospace applications due to their high surface area yet lightweight structure and tailorable physical and mechanical properties.¹ Since metal foams are a fairly new material, there is limited availability on established protocols that will aid with standardizing characterization procedures. One protocol that must be addressed is the cleaning of foam materials after being subjected to corrosion testing.² Through chemical cleaning, the corrosion products on a metal surface can be removed in order to better estimate the corrosion rate of the specimens.³ However, due to the complex geometry and high surface area of open-cell metal foams, chemical cleaning using existing standards may not be fully applicable to this novel material. Unfortunately, no published literature has studied or reviewed whether existing techniques, e.g. ASTM G1, nitric acid immersion, etc. are effective in cleaning emerging materials such as metallic foam.

The current protocol on post-corrosion chemical cleaning is the ASTM G1 standard.³ It specifies that aluminum and aluminum alloy specimens must be immersed in a phosphoric acid (H_3PO_4) and chromium trioxide (CrO_3) solution at near boiling temperature to remove corrosion products.³ If the corrosion product film remains on the surface after this step, the standard specifies that the specimen shall be immersed in nitric acid at room temperature.³ While this procedure may be the standard, concerns have arisen with the formation of CrO_3 vapor and its potential hazard to the human body.⁴ Chromium trioxide is acutely toxic and corrosive.⁵ If CrO_3 comes into contact with bare skin, it can lead to issues such as dermatitis, and ulceration; inhalation can cause shortness of breath, coughing, and wheezing.⁶ If the body is exposed to this chemical for a long time, negative consequences such as cancer, bronchitis, and cell mutations can occur.⁶ Therefore, it would be advantageous to find a less hazardous chemical cleaning method for aluminum.

An alternate approach to clean aluminum and alloys utilized nitric acid, with similar reported effectiveness to the cleaning approach outlined above.⁷ Nitric acid is a strong oxidizer and a corrosive liquid.⁸ Direct skin contact with the acid causes irritation and burns. If inhaled, it can cause breathing difficulties and lead to lung tissue damage and other respiratory problems.⁹ The Safety Data Sheet (SDS) for nitric acid (68 – 70%) describes the proper storage, handling, and disposal methods. This acid should be stored in tightly closed containers in dry and well-ventilated areas, away from direct sunlight and combustible and flammable materials.^{8,10} Once opened, it should be ensured that the container is carefully resealed and stored upright to prevent contamination and/or leakage.^{8,10} To reduce exposure to HNO_3 vapors, proper PPE and/or respirators should be worn and all operations must be conducted under a fume hood.^{10,11} The disposal method of HNO_3 includes neutralization with water and alkali and disposing in a secure and approved plant.^{9,10} Health and safety hazards would be reduced if the initial cleaning step (per ASTM G1) of immersion in hot H_3PO_4 and CrO_3 is eliminated, and only room temperature nitric acid is utilized. Therefore, cleaning with nitric acid simplifies the process (use of one chemical vs three), minimizes the hazardous chemical waste, and improves procedural safety.⁷ Additionally, it eliminates the long-term health risks that come from exposure to CrO_3 .^{5,6}

In this project, the effect of nitric acid immersion on the mass loss and topography of pre- and post-corroded UNS A96101-T6 foams was studied. This procedure eliminates the need of subjecting the test specimens in a H_3PO_4 and CrO_3 solution at near boiling temperature and relies solely on immersing the test specimens in nitric acid at room temperature. This cleaning method ensured that only the corrosion products (e.g., aluminum hydroxide) are removed while the base material is intact.¹²

EXPERIMENTAL PROCEDURE

Open-cell aluminum 6101 foams (UNS A96101) with pore densities of 10, 20, and 40 pores per inch (PPI) and relative densities between 6 and 8% were utilized for this project. The manufacturer heat treated the test specimens with T6 temper, where they were solution treated at high temperatures followed by artificial ageing at a certain temperature. These foam materials were fabricated through an investment casting process in which molten metal is poured into a mold in the shape of a porous foam.¹³ The specimens were cut with a diamond saw into the dimensions of approximately 0.30" x 0.25" x 0.50". The surface area of the coupons was determined through the Brunauer-Emmett-Teller (BET) technique performed by the manufacturer with slight scaling as a result of cutting the main foam specimens. The approximate surface areas of the metallic foams in relation to their pore density are shown in Table 1. The composition of the UNS A96101-T6 foams is listed in Table 2.

Table 1. Surface Area of UNS A96101-T6 Foams

Pore Density (PPI)	Relative Density (%)	Surface Area (cm^2)
10	6-8	4-6
20	6-8	7-9
40	6-8	10-12

Table 2. Chemical Composition of UNS A96101-T6 Alloy (wt%)¹⁴

Al	Si	Fe	Cu	Mn	Cr	Zn	B
Bal.	0.3-0.7	0.50	0.10	0.03	0.03	0.1	0.06

The foam specimens were subjected to immersion testing in accordance with ASTM G31-12a to study their corrosion behavior in immersion and salt fog testing in simulated marine environments.¹⁵ The results of these experiments were published in our previous work.¹⁷ In this project, the nitric acid cleaning procedure adapted a trial-and-error method to determine the optimal duration of chemical cleaning. Pre-corroded and corroded UNS A96101 specimens (10 PPI, 20 PPI, 40 PPI foams) were immersed in 68 to 70 wt% nitric acid at room temperature between 1 to 60 minutes. The mass loss of each coupon was recorded at increasing time intervals. The mass loss versus immersion duration was examined to determine the optimal duration.

Scanning electron microscopy (SEM) was utilized to examine and characterize the changes in corrosion morphology and microstructure as a function of exposure periods. SEM micrographs were taken after 1, 3, 5, 10, 15, and 20 minutes of immersion in 68 to 70 wt% nitric acid (HNO₃) to observe the changes in surface topography through time. This procedure enabled the observation of the gradual removal of corrosion products on the surface and ensured that the base material remains intact.

RESULTS

Figure 1 depicts the mass loss normalized by area of as-received (pre-corroded) 10, 20, and 40 PPI foams upon immersion in nitric acid. Mass loss was observed on the as-received 10 PPI foam after 15 minutes of immersion. Up to the 60-minute time range, it continued to exhibit a trend in which the mass loss curve showed a plateau, followed by an increase of mass loss. This trend wherein constant mass is followed by an increase of mass loss was also observed for the as-received 20 and 40 PPI foams upon 55 and 35 minutes of immersion, respectively. Results in Figure 1 indicate that as-received UNS A96101 foams should not be exposed 68-70 wt% HNO₃ for more than 15 minutes as mass loss can be observed past this time. This indicates that the base material is being corroded by the HNO₃.

The utilization of nitric acid for chemically cleaning aluminum and aluminum alloys is possible because this material forms a protective nitric oxide layer on the surface that delays the onset of acid corrosion.¹⁶ This ensures that there is low reactivity between the base material and the acid solution.¹⁶ It is noted that the pore density (PPI) appears to influence the extent of mass loss of each coupon. This may be due to the effect of pore size and exposed surface area on the mass transport of oxidizing species and corrosion products during the immersion process. This behavior will be investigated further in future work.

The same cleaning procedure was performed on post-corroded foams to observe the changes on the surface with the presence of corrosion products on the surface. Figure 2 shows that all post-corroded foams at all pore densities (10, 20, and 40 PPI) exhibit mass loss at about 15 minutes of exposure, which is then followed by a plateau in mass. This trend in mass loss is indicative of the complete removal of corrosion products. Further increases in mass loss following the flattening of the curve would indicate that the base material is being corroded by the acid solution. These results suggest that 15 ± 5 minutes is the optimal cleaning duration for aluminum foam with the dimension 0.30" x 0.25" x 0.50". The discrepancies in time can be attributed to the increasing of surface area with increasing pore density.

To accurately observe the changes in surface topology of metal foam specimens before and after HNO₃ immersion, SEM analysis was performed. Figure 3a reveals the surface of a post-corroded 40 PPI foam strut, which shows the presence of the corrosion products, aluminum hydroxide Al(OH)₃ and a plate-like Al-Fe-Si intermetallic.¹³ Al-Fe-Si intermetallic was formed due to the slow solidification associated with investment casting process.¹⁸ The intermetallic was found to be stable relative to the alloy matrix in a simulated marine environment.¹⁴ Figures 3a through 3f show a chronological series of backscattered