

Corrosion Inhibition of Aluminum using Methyl-1H-Benzotriazole in Deaerated Fluoride-Sulphate Media: Corrosion Behaviour and Surface Protection

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The inhibitive effect of methyl-1H-benzotriazole (Me-BTA) on pure aluminum was investigated in fluoride-sulphate media at different sulphate concentrations. The corrosion rate, surface roughness, surface morphology and elemental composition of aluminum were evaluated after 24 h of immersion at 80 °C in deaerated fluoride-sulphate solutions both with and without Me-BTA. Weight-loss measurements showed that the addition of Me-BTA decreased the corrosion rate of aluminum under the investigated conditions. AFM analysis further revealed a reduction in the surface roughness of the aluminum specimens in the presence of Me-BTA, indicating a less damaged surface after exposure to the aggressive medium. SEM analysis revealed fewer corrosion-related surface products on the Me-BTA-treated specimens than on the uninhibited aluminum. EDX further detected nitrogen and increased carbon content after treatment, consistent with the presence of Me-BTA-derived species on the surface. These surface and elemental changes support the protective role of Me-BTA and the formation of an inhibitor-derived layer on aluminum in the investigated deaerated fluoride-sulphate media.

Keywords: Aluminum, Corrosion, Inhibition, Methyl-1H-benzotriazole, Atomic force microscopy, Scanning electron microscopy.

INTRODUCTION

Aluminum (Al) and its alloys are widely employed as structural materials in numerous industrial applications, particularly in the aerospace sector owing to their excellent mechanical and technological properties [1-4]. These include a high strength-to-weight ratio, high specific strength and stiffness, good corrosion resistance, ease of fabrication and relatively low cost compared with many other engineering materials. Despite these advantages, Al alloys are susceptible to localized corrosion, particularly in aggressive environments, due to the electrochemical heterogeneity associated with intermetallic particles and their dissolution or dealloying within the alloy matrix [5-7]. In contrast, commercially pure Al generally exhibits relatively high corrosion resistance under atmospheric conditions and requires less protection than many other struc-

tural metals, except under strongly acidic or alkaline conditions [8]. This corrosion resistance is primarily attributed to the spontaneous formation of a thin, compact, adherent and protective oxide film, predominantly composed of aluminum oxide, on the Al surface [9,10]. Although Al is thermodynamically highly reactive, this passive oxide layer effectively acts as a barrier to the transport of aggressive species and significantly suppresses further metal dissolution and corrosion.

Developing effective strategies to control aluminum and aluminum-alloy corrosion has received considerable attention in recent decades. Current methods include anodization, plasma electrolytic oxidation, surface coatings, conversion treatments and organic or inorganic corrosion inhibitors, with naturally derived compounds gaining interest as environmentally benign alternatives [11-15]. Among the conventional surface protection strategies, chromate conversion coatings have histori-

cally been regarded as highly effective for protecting Al and its alloys against corrosion. However, due to the toxicity, carcinogenicity and persistence linked to Cr(VI) species, the widespread use of hexavalent chromium-based treatments has produced significant environmental and health issues [16-20]. Consequently, the development of efficient chromate-free corrosion-protection strategies has become an important research priority. Among the available methods, the use of organic corrosion inhibitors has emerged as one of the most promising and versatile methods for reducing the corrosion rate of metals and alloys in aqueous and aggressive environments [21]. Their effectiveness arises from high inhibition efficiency at relatively low concentrations, strong metal-surface affinity, good film-forming ability and tunable molecular structures. Properly designed compounds may further offer low environmental impact and good biodegradability [22-25]. Through adsorption and formation of a protective interfacial film, organic inhibitors can reduce both anodic metal dissolution and cathodic reactions depending on their molecular structure and the metal-environment system.

The inhibition performance of organic compounds is strongly influenced by the heteroatoms and functional groups that interact with metal surfaces. Nitrogen (N), oxygen (O), sulfur (S) and phosphorus (P), along with π -electron systems, provide electron-donating sites that can facilitate adsorption onto metallic surfaces [26-29]. Depending on the molecular structure, surface condition, electrolyte composition, temperature and adsorption energetics, inhibitor molecules may adsorb through physisorption, chemisorption or a combination of both [30-33]. Adsorbed molecules compete with aggressive species for active surface sites, reducing charge transfer, metal dissolution and the penetration of corrosive species. Surface coverage, adsorption strength, film stability and persistence consequently play important roles in determining inhibition efficiency. A wide range of organic compounds containing heteroatoms and conjugated π -electron systems has been investigated as corrosion inhibitors for metals and alloys [34-36].

Nitrogen containing triazoles and their derivatives represent an important class of five-membered heterocyclic corrosion inhibitors and have been extensively studied through experimental and theoretical approaches [37-42]. Their protective action is generally associated with interactions between heteroatom-containing groups or π -electron rich moieties and the metal surface, which can promote formation of an adsorbed protective film and in some cases, metal-inhibitor complexes at the metal/electrolyte interface. These interactions can hinder anodic dissolution and/or cathodic reactions. Structural modification of triazole derivatives through substitution further allows their adsorption behaviour and inhibition performance to be tailored to specific metal-environment systems [33]. Despite extensive research on triazole-based inhibitors, information on the adsorption behaviour, interfacial film stability and durability of benzotriazole derivatives on aluminum remains limited, particularly in aggressive fluoride containing environments [43-45].

Limited information is available on the corrosion inhibition behaviour of methyl-1*H*-benzotriazole (Me-BTA) toward commercially pure aluminum in combined fluoride-sulphate

media under deaerated conditions at elevated temperature. The present study addresses this gap by examining the corrosion and surface-protection behaviour of Me-BTA on commercially pure Al in deaerated fluoride-sulphate solutions at 80 °C. The weight-loss measurements and surface-characterization techniques were used to assess corrosion behaviour, surface roughness, morphology and elemental composition in the absence and presence of Me-BTA. Particular attention was given to surface changes associated with inhibitor exposure and to developing a possible protection mechanism based on the experimental findings.

EXPERIMENTAL

Pure aluminum (Al) (99+%; Nilaco Corporation, Tokyo, Japan) was employed as the test material for the immersion studies. The Al specimens were cut into rectangular pieces with a thickness of 1 mm and an exposed surface area of 6 cm². Prior to the immersion experiments, the specimens were sequentially polished using emery papers with grit sizes of 400, 1200 and 2000 to obtain a uniform and reproducible surface finish. The polished specimens were subsequently rinsed thoroughly with acetone to remove residual contaminants and surface debris. Finally, the prepared Al specimens were immersed in 100 mL of the corresponding test solutions contained in PTFE cells under the specified experimental conditions.

Immersion conditions and test-solution preparation:

The Al specimens were immersed in aqueous solutions containing different fluoride and sulphate concentrations, namely 0.1 ppm F⁻, 0.1 ppm F⁻ + 0.1 ppm SO₄²⁻, distilled water (DW), 0.1 ppm SO₄²⁻, 0.1 ppm F⁻ + 0.5 ppm SO₄²⁻ and 0.1 ppm F⁻ + 1.0 ppm SO₄²⁻. Both with and without Me-BTA as a corrosion inhibitor, the Al specimens were submerged in the corresponding test solutions for 24 h at 80 °C. To minimize the influence of dissolved oxygen on the corrosion process, all immersion experiments were performed under deaerated conditions. The test solutions and specimens were handled in an argon purged glovebox and an Ar atmosphere was maintained throughout the experimental procedure. The concentrations of fluoride and sulphate ions were kept intentionally very low in the test solutions as these ions are very reactive and sensitive toward metallic surfaces and air pollutants and may influence the corrosion behaviour of aluminum.

The fluoride- and sulphate containing test solutions were prepared using analytical-grade sodium fluoride (NaF) and sodium sulphate (Na₂SO₄), purchased from Kanto Chemical Co., Inc. and Wako Pure Chemical Industries, Ltd., Japan, respectively. Methyl-1*H*-benzotriazole (Me-BTA; analytical grade, 99.98% purity) was employed as the organic corrosion inhibitor at a concentration of 0.1 mM. The required amount of Me-BTA was added to the respective test solutions prior to immersion to evaluate its inhibitive effect on the corrosion of pure Al. The pH of all test solutions was measured before immersion, both in the absence and presence of Me-BTA. The corresponding pH values are summarized in Table-1.

Measurement of corrosion rate: After the test specimens were submerged in the test solutions with and without 0.1 mM of Me-BTA at 80 °C in a deaerated atmosphere using Ar gas, their weight was determined. After immersion, the

TABLE-1
pH OF THE TEST SOLUTIONS WITHOUT AND WITH Me-BTA

Inhibitor	Solutions and corresponding pH					
	0.1 ppm F ⁻	0.1 ppm F ⁻ + 0.1 ppm SO ₄ ²⁻	DW	0.1 ppm SO ₄ ²⁻	0.1 ppm F ⁻ + 0.5 ppm SO ₄ ²⁻	0.1 ppm F ⁻ + 1 ppm SO ₄ ²⁻
Without Me-BTA	5.78	5.72	6.3	5.72	5.66	5.62
With Me-BTA	6.34	6.12	5.87	5.41	5.25	5.13

specimens were cleaned with acetone for 15 to 20 min using a sonication bath, then rinsed several times with distilled water and dried in air before weighing. The experiments were conducted at least three times. The following formula provided by Fontana was also used to determine the corrosion rate, or CR (mm y⁻¹) [46].

$$\text{Corrosion rate (mm y}^{-1}\text{)} = \frac{87.6W}{\rho At}$$

where ρ is the density of Al (g cm⁻³), t is the immersion period (h), W is the specimen's weight loss (mg) and A is the surface area (cm²).

Surface characterization: Surface characterization of the Al specimens after immersion in test solutions with and without Me-BTA was performed using optical microscopy (Olympus U-IF550, BX60M, Japan), atomic force microscopy (AFM; Park Systems, NX-10) for surface roughness and field-emission scanning electron microscopy coupled with energy dispersive X-ray spectroscopy (FE-SEM/EDX; JSM-7800F PRIME) for surface morphology and elemental composition. SEM-EDX measurements were conducted at a working distance of 10 mm and an accelerating voltage of 10 kV.

RESULTS AND DISCUSSION

Optical micrographic images after immersion: The optical micrographs (OM) of pure Al after 24 h of immersion in 0.1 ppm F⁻, 0.1 ppm F⁻ + 0.1 ppm SO₄²⁻, DW, 0.1 ppm SO₄²⁻, 0.1 ppm F⁻ + 0.5 ppm SO₄²⁻ and 0.1 ppm F⁻ + 1 ppm SO₄²⁻ solutions at 80 °C, in the absence and presence of Me-BTA, are shown in Figs. 1 and 2, respectively. The micrographs reveal corrosion-related surface changes on the Al specimens, with minor surface damage and no visible cracks or pits. A porous surface layer was observed, which may be associated with corrosion products. The specimens exposed to Me-BTA showed a visibly smaller corroded area, supporting the protective effect of the inhibitor.

Corrosion rate analysis: Eqn. 1 was used to calculate the corrosion rates of Al specimens immersed for 24 h at 80 °C in the test solutions, with and without Me-BTA, and the results are shown in Fig. 3. The corrosion rate decreased with increasing sulphate concentration in fluoride-containing solutions, whereas the presence of fluoride increased the corrosion rate. In the absence of Me-BTA, the corrosion rates in 0.1 ppm F⁻ and 0.1 ppm F⁻ + 1 ppm SO₄²⁻ solutions were

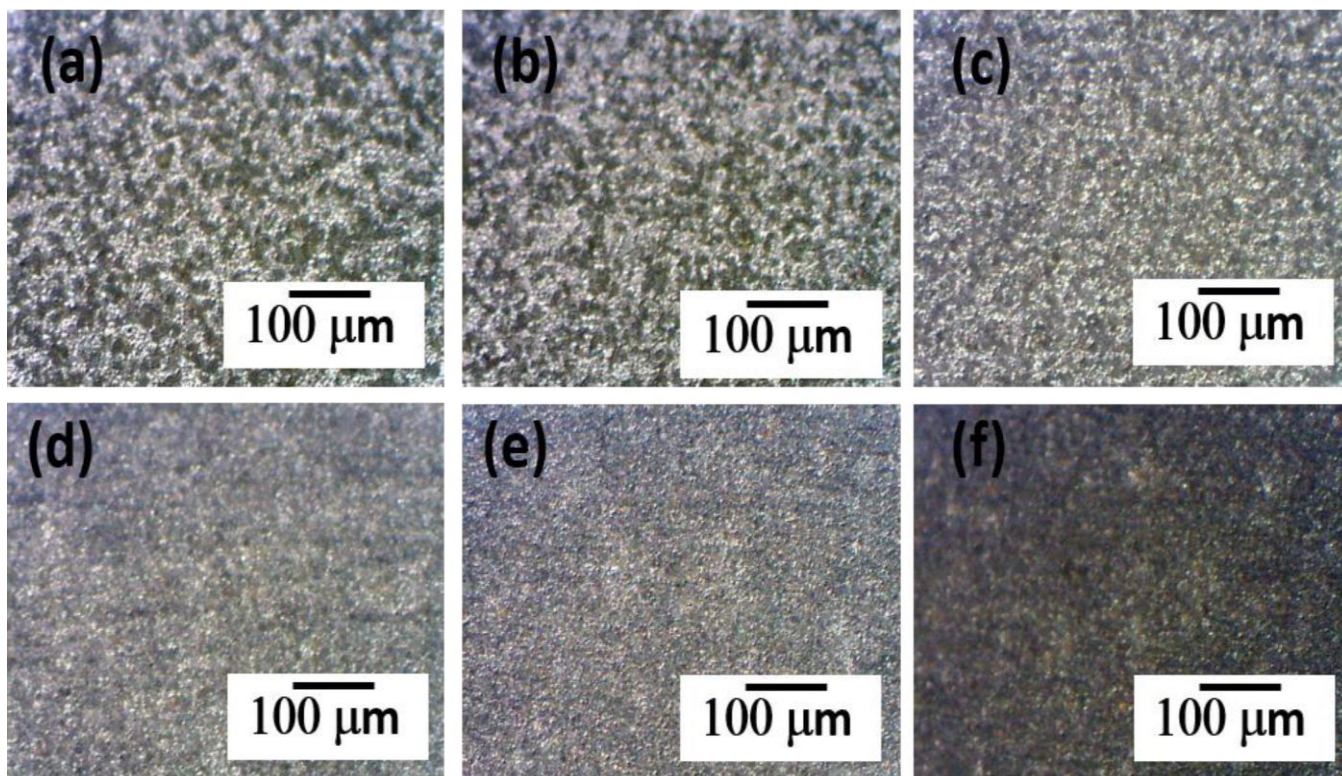


Fig. 1. Microscopic images of pure Al after immersion in (a) 0.1 ppm F⁻, (b) 0.1 ppm F⁻ + 0.1 ppm SO₄²⁻, (c) DW, (d) 0.1 ppm SO₄²⁻, (e) 0.1 ppm F⁻ + 0.5 ppm SO₄²⁻ and (f) 0.1 ppm F⁻ + 1 ppm SO₄²⁻ solutions for 24 h at 80 °C without Me-BTA

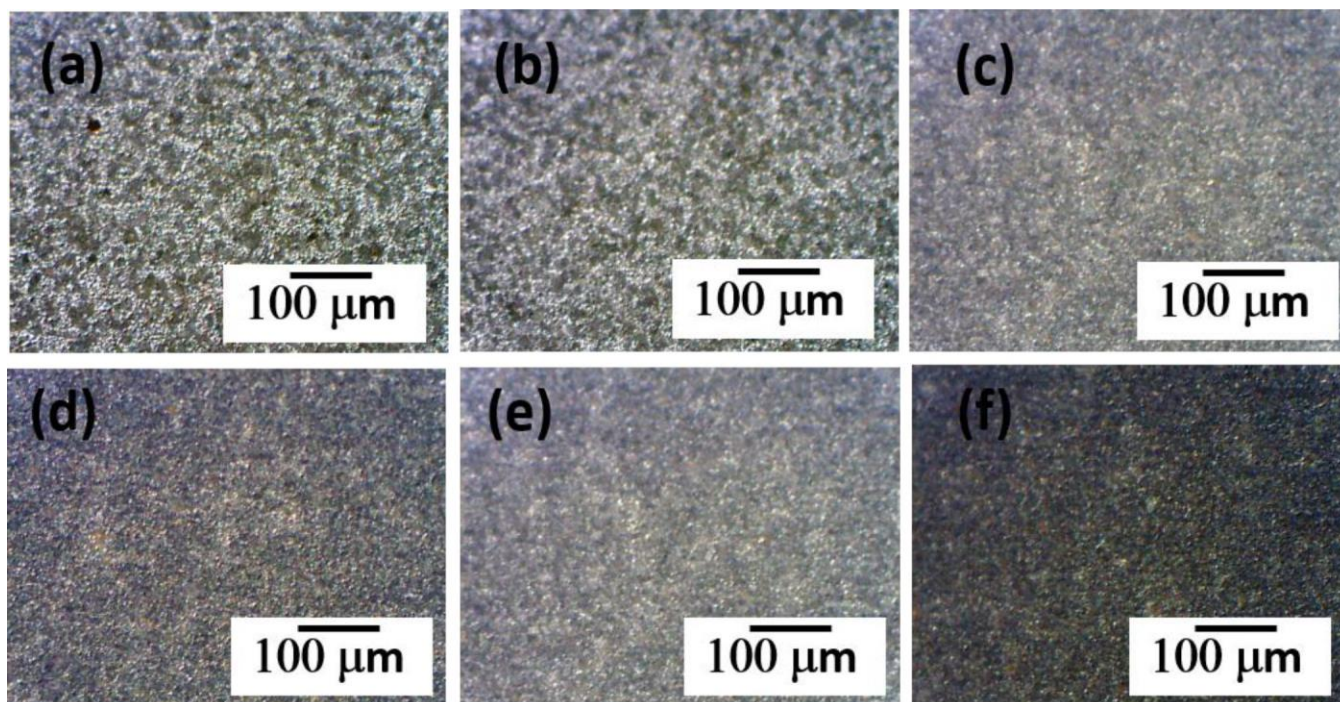


Fig. 2. Microscopic images of pure Al after immersion in (a) 0.1 ppm F^- , (b) 0.1 ppm F^- + 0.1 ppm SO_4^{2-} , (c) DW, (d) 0.1 ppm SO_4^{2-} , (e) 0.1 ppm F^- + 0.5 ppm SO_4^{2-} and (f) 0.1 ppm F^- + 1 ppm SO_4^{2-} solutions for 24 h at 80 °C with Me-BTA

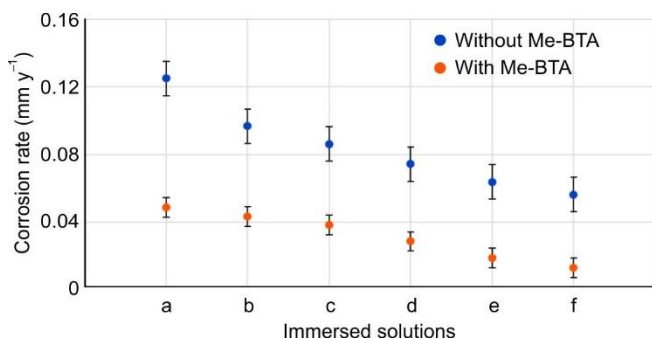


Fig. 3. Corrosion rate, C_R of pure Al after immersion in (a) 0.1 ppm F^- , (b) 0.1 ppm F^- + 0.1 ppm SO_4^{2-} , (c) DW, (d) 0.1 ppm SO_4^{2-} , (e) 0.1 ppm F^- + 0.5 ppm SO_4^{2-} and (f) 0.1 ppm F^- + 1 ppm SO_4^{2-} solutions for 24 h at 80 °C without and with Me-BTA

0.1249 and 0.0563 mm y^{-1} , respectively. Me-BTA further reduced the corrosion rate, with the value for 0.1 ppm F^- + 1 ppm SO_4^{2-} decreasing from 0.0563 to 0.0131 mm y^{-1} . These results support the protective effect of Me-BTA on Al under the investigated conditions.

Surface roughness: Atomic force microscopy (AFM) was used to assess the surface roughness of Al specimens after immersion in the test solutions with and without Me-BTA. The surface topographies are shown in Fig. 4. Uninhibited specimens exhibited relatively rough surfaces with prominent hill-like features and corrosion products, whereas Me-BTA treated specimens showed smoother and more uniform surfaces with only minor surface irregularities. The corresponding average roughness (R_a) values are given in Fig. 5. The higher surface roughness of the uninhibited specimens is consistent with greater surface deterioration, while the lower R_a values in the presence of Me-BTA support the formation of a more uniform protective surface layer.

Surface morphology: Figs. 6 and 7 show the surface morphology images of all test specimens without and with Me-BTA after 24 h of exposure to various concentrations of sulphate containing fluoride solutions at 80 °C. The SEM images (Fig. 6) show several cubic-shaped surface features on the Al specimens exposed in the absence of Me-BTA. These features were less pronounced in the corresponding inhibited specimens (Fig. 7). Based on the SEM morphology alone, these features are described as cubic-shaped corrosion-related surface products without assigning a specific chemical composition.

In contrast, the specimens exposed to Me-BTA (Fig. 7) exhibited only minor, localized corrosion products across the surface. The smoother surface morphology observed in the presence of Me-BTA suggests the formation of a protective coating. A markedly lower amount of corrosion-related surface products was observed with Me-BTA, particularly at higher sulphate concentrations. The specimen shown in Fig. 7f, exposed to 0.1 mM Me-BTA, exhibited the smoothest surface with no apparent defective regions.

EDX results: The EDX results of the Al test specimens after immersion in the different test solutions, with and without Me-BTA, are shown in Fig. 8a-b. The elemental mapping images and compositions of the Al specimens exposed to 0.1 ppm F^- + 1 ppm SO_4^{2-} , in the absence and presence of Me-BTA, are shown in the corresponding figures. EDX mapping identified the distribution of Al, C, O, F, S and N, and the corresponding elemental compositions are given in Table-2. In the absence of Me-BTA, the Al surface contained Al, C, O, F and S, which confirmed the formation of an oxide/corrosion-product layer during immersion.

As shown in Table-2, the uninhibited specimen contained 65.61% O, which is consistent with the presence of

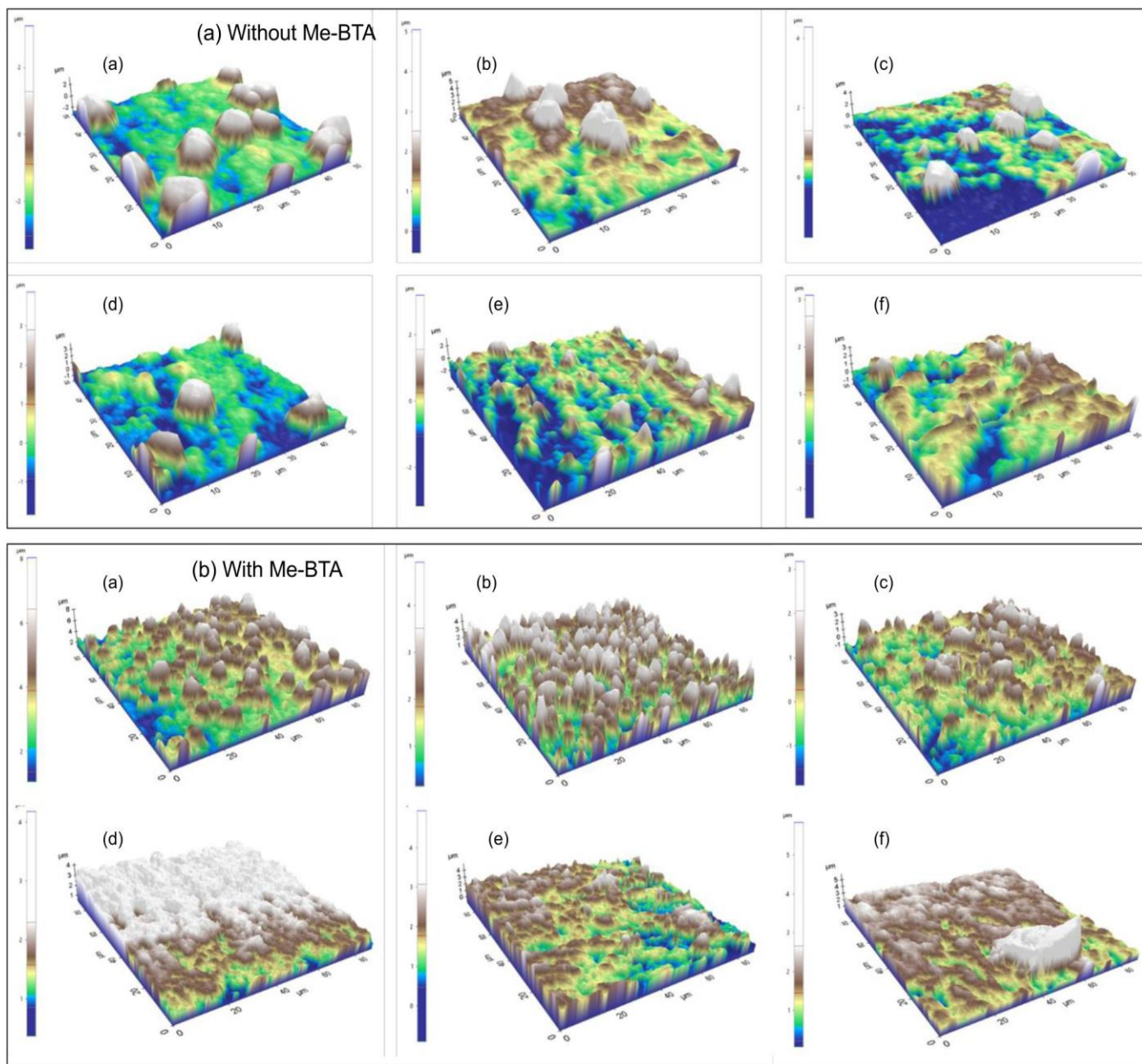


Fig. 4. AFM images of pure Al after immersion in (a) 0.1 ppm F^- , (b) 0.1 ppm F^- + 0.1 ppm SO_4^{2-} , (c) DW, (d) 0.1 ppm SO_4^{2-} , (e) 0.1 ppm F^- + 0.5 ppm SO_4^{2-} and (f) 0.1 ppm F^- + 1 ppm SO_4^{2-} solutions for 24 h at 80 °C (A) without and (B) with Me-BTA

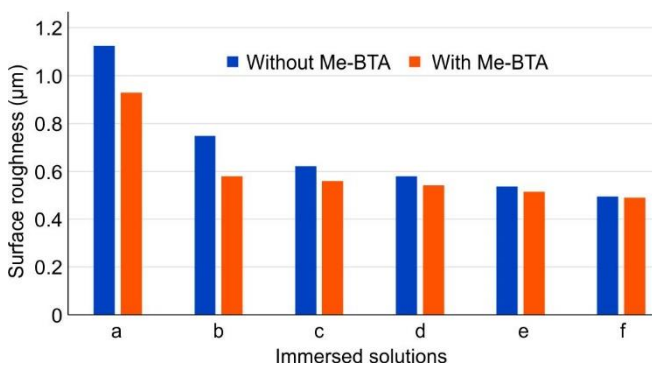


Fig. 5. Surface roughness values, Ra (μm) of pure Al after immersion in (a) 0.1 ppm F^- , (b) 0.1 ppm F^- + 0.1 ppm SO_4^{2-} , (c) DW, (d) 0.1 ppm SO_4^{2-} , (e) 0.1 ppm F^- + 0.5 ppm SO_4^{2-} and (f) 0.1 ppm F^- + 1 ppm SO_4^{2-} solutions for 24 h at 80 °C without and with Me-BTA

oxygen containing surface species [47,48]. After treatment with Me-BTA, nitrogen was detected and the carbon content increased, which support the adsorption of Me-BTA derived species on the aluminum surface. The presence of N together with Al may arise from interactions between the inhibitor molecules and the surface oxide layer. The O content decreased from 65.61% to 55.04% after Me-BTA treatment, while the C content increased to 20.85%. The increased carbon content and appearance of nitrogen provide evidence for an Me-BTA-derived surface layer on aluminum and support the formation of an inhibitor-mediated protective film.

Mechanism: A proposed mechanism for the interaction of Me-BTA with the Al surface in fluoride-sulphate solution is shown in Fig. 9. The nitrogen containing sites of Me-BTA may interact with the aluminum surface through electrostatic

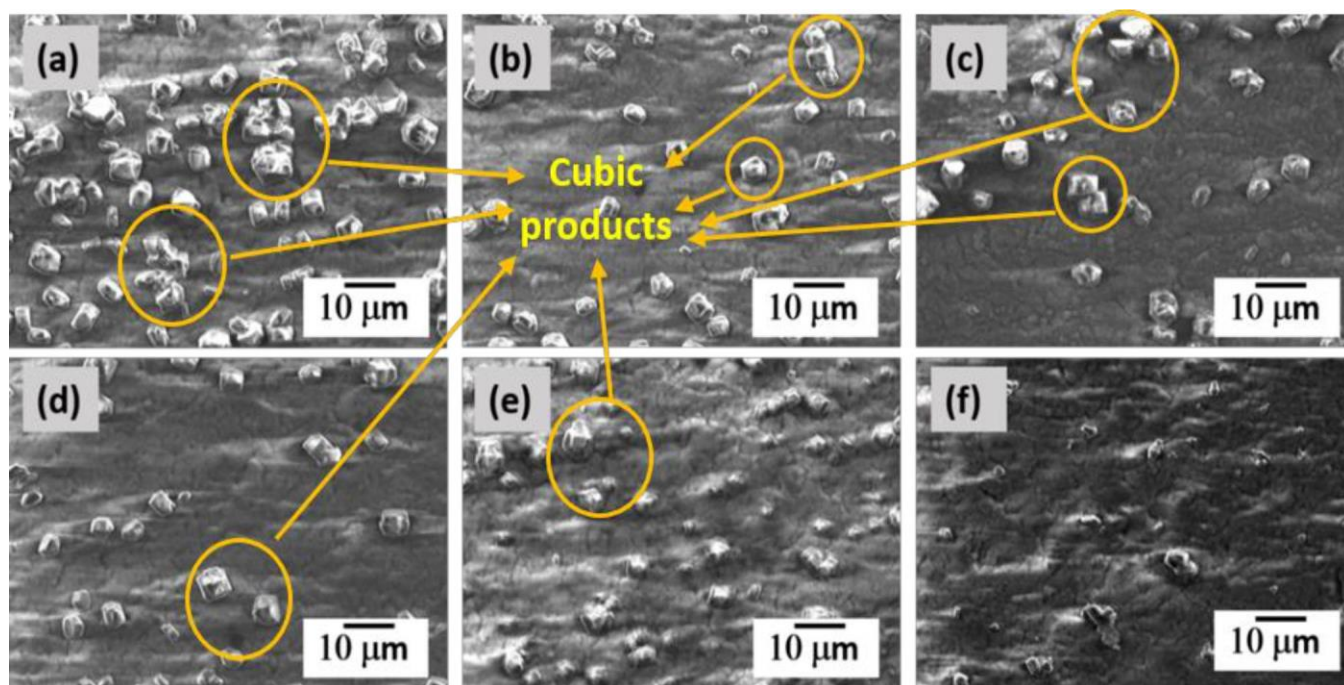


Fig. 6. SEM images of pure Al without Me-BTA in (a) 0.1 ppm F^- , (b) 0.1 ppm F^- + 0.1 ppm SO_4^{2-} , (c) DW, (d) 0.1 ppm SO_4^{2-} , (e) 0.1 ppm F^- + 0.5 ppm SO_4^{2-} and (f) 0.1 ppm F^- + 1 ppm SO_4^{2-} solutions immersed for 24 h at 80 °C without Me-BTA. The marked regions indicate representative cubic-shaped surface products

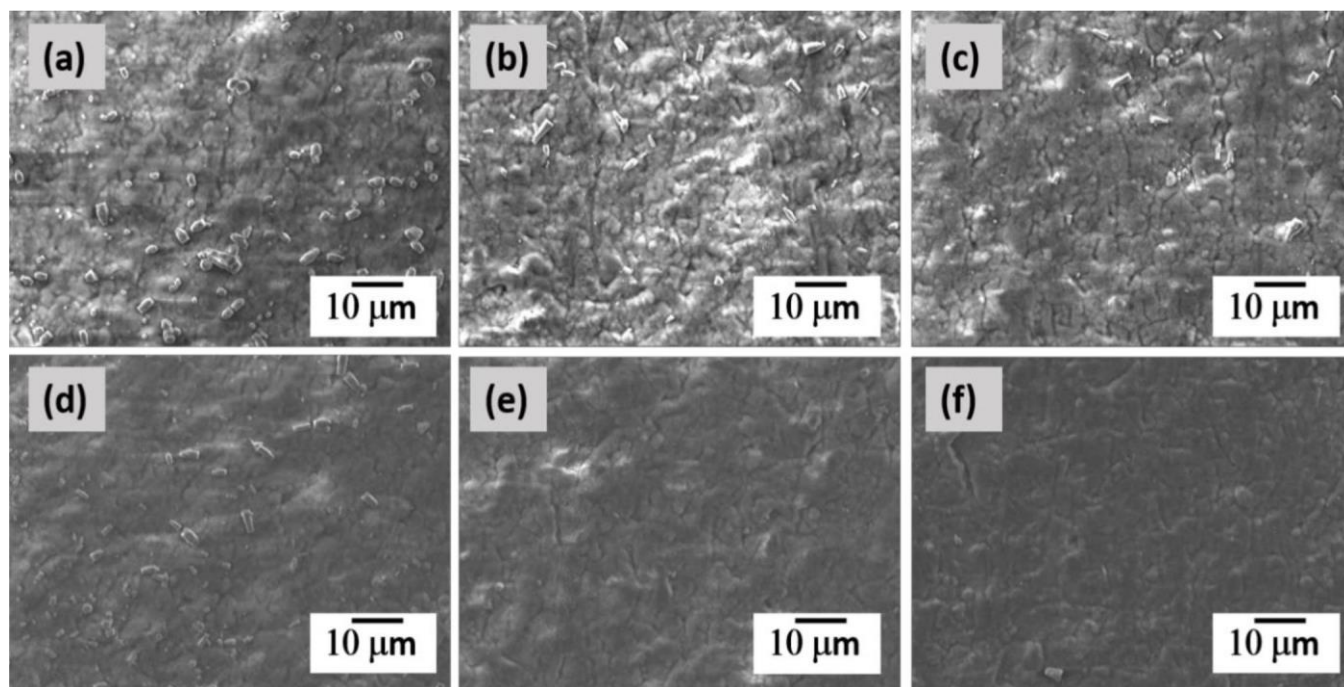


Fig. 7. SEM images of pure Al with Me-BTA in (a) 0.1 ppm F^- , (b) 0.1 ppm F^- + 0.1 ppm SO_4^{2-} , (c) DW, (d) 0.1 ppm SO_4^{2-} , (e) 0.1 ppm F^- + 0.5 ppm SO_4^{2-} and (f) 0.1 ppm F^- + 1 ppm SO_4^{2-} solutions immersed at 80 °C for 24 h

TABLE-2
FOLLOWING IMMERSION, THE ELEMENTAL COMPOSITIONS OF PURE Al IN
0.1 ppm F^- + 1 ppm SO_4^{2-} SOLUTION FOR 24 h AT 80 °C WITHOUT AND WITH Me-BTA

Solution	Elemental composition by atomic (%)						Total
	O	Al	C	S	F	N	
Without Me-BTA	65.61	26.19	7.09	0.67	0.44	—	100
With Me-BTA	55.04	22.82	20.85	0.07	0.35	0.87	100

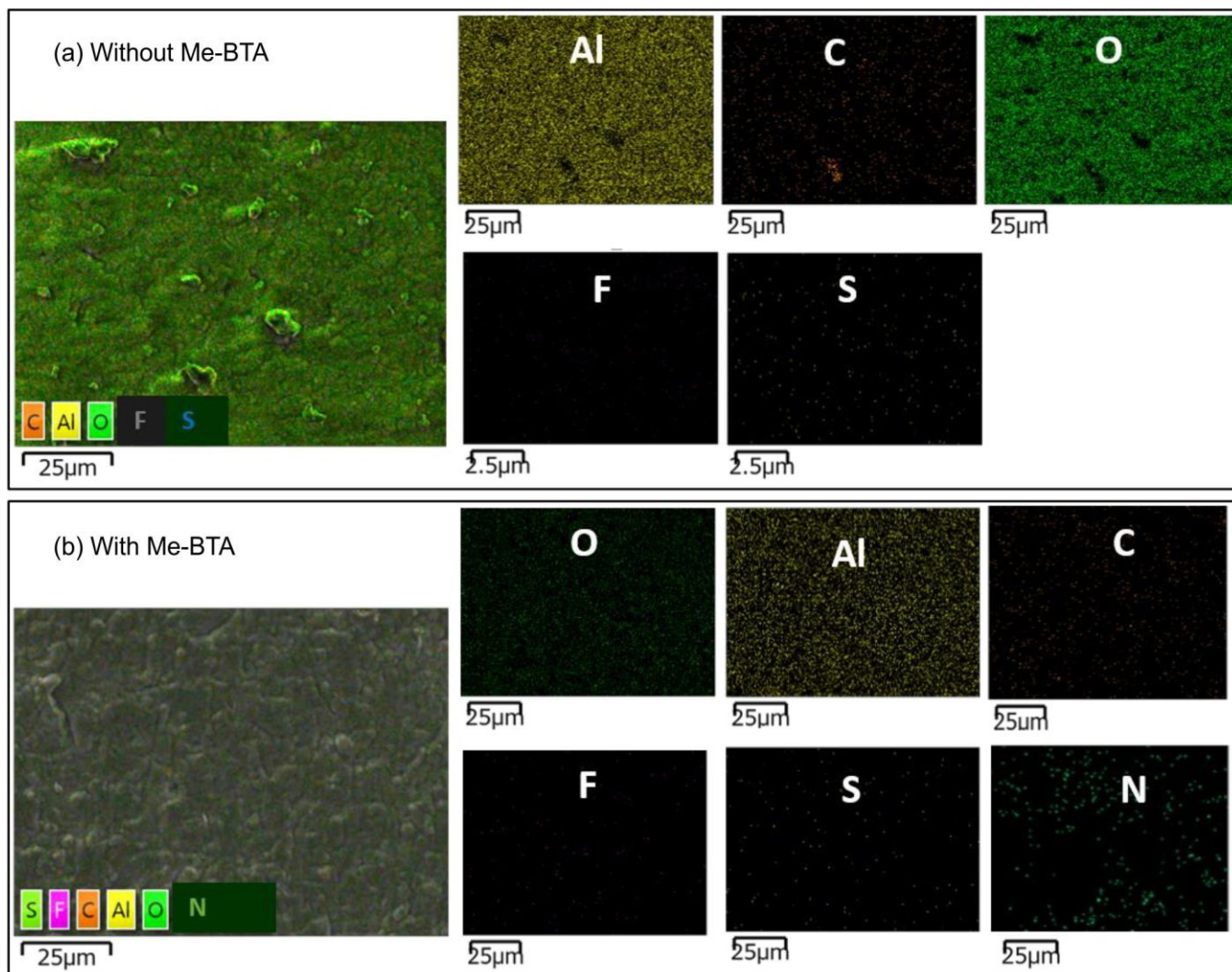


Fig. 8. EDX mapping of pure Al immersed in 0.1 ppm F^- + 1 ppm SO_4^{2-} solution for 24 h (A) without and (B) with Me-BTA

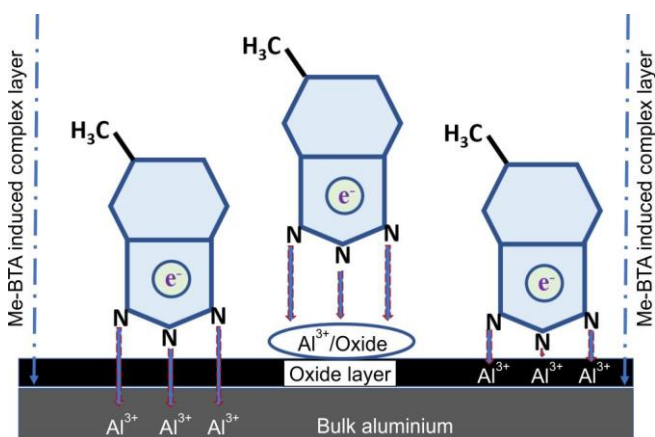


Fig. 9. Me-BTA's suggested mode of interaction with the Al surface

and/or chemical interactions, which facilitate the formation of a protective interfacial layer. This layer can hinder the access of aggressive species to the Al surface, which may account for the lower corrosion rate and surface roughness observed in the presence of Me-BTA. The extent of this interaction can be influenced by the solution composition, chemical

environment, surface condition of aluminum and molecular structure of the inhibitor. The SEM and EDX results are consistent with the presence of Me-BTA-derived species on the Al surface. The available experimental data, though, do not establish the exact nature of the interaction, distinguish physisorption from chemisorption or confirm specific Al–N coordination bonds.

Conclusions

The corrosion behaviour of pure aluminum in deaerated fluoride-sulphate solutions was investigated in the absence and presence of methyl-1*H*-benzotriazole (Me-BTA) using weight-loss measurements, AFM, SEM and EDX analyses after 24 h of immersion. Me-BTA decreased the corrosion rate of aluminum and reduced its surface roughness, which indicate improved surface protection. SEM analysis revealed a lower amount of corrosion-related surface products on the inhibited specimens, without assigning a specific chemical composition to these products. The detection of nitrogen together with the increased carbon content after Me-BTA exposure provides evidence for the adsorption of Me-BTA-derived species on the aluminum surface. The lower corrosion rate, smoother

surface and reduced accumulation of corrosion-related products after treatment point to the development of a protective interfacial layer. This layer may reduce the exposure of the aluminum surface to aggressive fluoride-sulphate species. A possible Me-BTA–Al interaction mechanism is proposed on the basis of the experimental findings, although the present results cannot establish the adsorption mode or confirm specific Al–N coordination.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

DECLARATION OF AI-ASSISTED TECHNOLOGIES

During the preparation of this manuscript, the authors used an AI-assisted tool(s) to improve the language and no data, results or scientific conclusions were generated by AI. All analyses, interpretations and conclusions are the authors' own. The authors reviewed and edited the content and take full responsibility for the published work.

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