Sensitization of Naturally Aged Aluminum 5083 Armor Plate

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A sample of aluminum armor plate, approximately 40 to 50 years old, containing a large intergranular stress corrosion crack was evaluated. Scanning electron microscopy examination of the fracture surfaces is consistent with intergranular stress corrosion cracking and some evidence of variable environmental and stress history is noted. ASTM G-67 nitric acid mass-loss values were 19 to 25 mg/cm². The transmission electron microscopy microstructure of the sample was found to be consistent with sensitization due to aging at low temperatures for a long time. The grain boundary magnesium-rich phase is continuous, although non-uniform, and only 10 to 15 nm thick.

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INTRODUCTION

Aluminum-magnesium alloys are important for both ship structures [1], and land vehicle armor [2]. These 5xxx alloys Al-Mg solid solution alloys, containing three to six percent magnesium, along with some manganese, exhibit high strength, excellent corrosion resistance, and are weldable. However, under prolonged exposure to temperatures of 50 to 200 °C, these alloys can degrade by the mechanism of sensitization, which occurs when magnesium normally in solid solution in the alloy diffuses to the grain boundaries [3,4]. The magnesium-rich phase (normally β-Al₃Mg₂) is highly anodic with respect to the surrounding aluminum phase, thus is susceptible to intergranular corrosion.

While there have been several studies of sensitization under controlled, artificial aging, usually at elevated temperatures up to 175 °C, little detailed information exists in the literature on very long-term aging at low or ambient operational temperatures. Recently, we had the opportunity to examine a piece of aluminum 5083 armor plate taken from an aging armored vehicle. The vehicle from which it was removed had been in normal service for 40 to 50 years. Unfortunately the exact environmental and thermal history of this plate are unknown, but likely there was environmental exposure to humid air, rain, saltwater, and engine exhaust; and elevated temperatures due to solar loading and proximity to the vehicle’s engine. The plate contained a large delamination crack in the mid-plane of the plate. The objective of this study was to identify if the crack in this sample was associated with sensitization.

SAMPLE DESCRIPTION

The 5083 plate sample is shown in Figure 1. The temper designation was not known, but armor plate often is H131 temper. Several specimens for transmission electron microscopy (TEM) were cored out of the region ahead of the crack tip. The specimens for the ASTM G-67 mass loss test were taken from the end of the sample as indicated. The front of the sample was cut off about one inch behind the crack tip, and the crack faces examined by SEM.

*Figure 1. Armor plate and specimen locations.*
SEM EXAMINATION

A cursory examination of randomly selected regions of crack faces was done with SEM. Most regions of the crack face, such as Figure 2a, exhibit clear, well defined flattened grain surface typical of intergranular crack growth of rolled aluminum alloys, and with varying amounts of debris scattered about the surface consistent with corrosion product, Figure 2b, that often forms over time within a crack. Figure 2c shows a region with a very thick layer of corrosion product in a “mud-crack” pattern, and may represent exposure to a particularly aggressive corrosive attack at some point in the sample’s history. There are other regions around the crack face, Figure 2d, that are consistent with a ductile fracture mechanism, perhaps as the result of some overload event.

On the basis of SEM examination, generally our assessment is that the crack appearance is consistent with intergranular stress corrosion cracking (IGSCC), although corrosion-fatigue cannot be ruled out on the basis of the SEM examination alone. Variations in the corrosion product and fracture mode would be consistent with a part that had seen a range of environment-stress conditions throughout its lifecycle.

![Figure 2. SEM micrographs of the crack faces showing (a) typical flattened grain intergranular cracking mode, (b) corrosion debris on the crack surface, (c) mud-crack pattern indicating aggressive environment exposure, and (d) localized region of ductile fracture.](image)

ASTM G-67 MASS LOSS TEST

The ASTM G-67 “Standard Test Method for Determining the Susceptibility to Intergranular Corrosion of 5XXX Series Aluminum Alloys by Mass Loss after Exposure to Nitric Acid” was used as an assessment of the degree of sensitization (DOS) of the alloy.[5] Three ASTM G-67 tests were done. The mass-loss results were 19, 20 and 25 mg/cm² with an average value of 21.3 mg/cm². The highest of the three values was a specimen from the bottom surface of the sample (the side facing the engine). The other two values were
from the middle of the sample. One other specimen from the top surface of the sample also was measured, yielding a mass loss of 27 mg/cm²; however, the test conditions on this specimen deviated from the standard test method so this result should not be regarded as valid.

TEM MICROSTRUCTURE

Sample preparation and the TEM and HRTEM techniques used are described in our several publications. [6,7] We examined two specimens cored from approximately 25 mm in front of the crack tip. One specimen was in the mid-plane of the sample; i.e., in the same plane as the crack, and the other was from the bottom of the sample; i.e., the side closest to the engine. A specimen from the top portion of the plate was not examined.

Figure 3 shows TEM images of typical grain boundaries in the mid-plane specimen. Figure 3(a) is a bright-field image showing that a continuous grain boundary phase with a thickness of about 10 nm is present. Figure 3(b) is a Z-contrast image, which displays the lighter elements darker. This shows that the grain boundary phase is composed of lighter elements than the aluminum matrix. Since the only constituent in the alloy lighter than aluminum is magnesium, it is reasonable to conclude that the grain boundary phase is magnesium rich. The fine-probe energy dispersive spectrometry (FPEDS) line scan in Figure 4 confirms the grain boundary is magnesium rich, over an extent of 10 to 20 nm, and the aluminum concentration in this region is lower. The average concentration indicated of about 19% Mg is somewhat lower than β, which would be 40%, but this is most likely an averaging artifact since the thickness of the grain boundary phase is very small and the Mg phase may not be perfectly edge-on.

We did not specifically confirm this magnesium rich phase to be β-Al₃Mg₂. The thickness is too small to use diffraction in the conventional TEM, thus would require use of high-resolution TEM and lattice plane imaging. However, we are confident that this is in fact β phase because: (1) β is the only equilibrium Al-Mg phase known to exist at these Mg concentrations; and (2) all of our other work on 5083 alloy identifies β phase as the magnesium rich grain boundary phase even when the precipitates are very small.

Figure 3. TEM images of typical grain boundary and grain boundary phase in the mid-plane specimen: (a) bright-field image and (b) Z-contrast image. In (b), darker areas indicate lighter elements and vice-versa.
Figure 4. Z-contrast image (a) and corresponding fine-probe EDS line scan (b), showing that the grain boundary phase is Mg-rich.

In addition to Mg-rich phase forming on the grain boundaries, we also see Mg-rich phase precipitating in very thin layers (less than 10 nm) on rod-shaped second-phase particles (typically Al₆Mn structure but also containing Fe or Cr) present in the interiors of grains. See Figure 5. We have seen this commonly in our other work on 5083 alloy.[3] Mg present on intragranular particles is not expected to contribute to IGSCC, but may promote pitting damage.

![Mg-rich phase](image)

Figure 5. Z-contrast image (a) and corresponding fine-probe EDS composition maps showing of an intragranular particle with a thin precipitated layer of Mg.

The specimen from the bottom half of the plate also had Mg-rich (probably β) on the grain boundaries, but was generally not continuous. Figure 6(a) and 6(b) shows the Z-contrast image and FPEDS line scan indicating the Mg-rich grain boundary phase. The higher resolution images of Figure 6(c) and 6(d) show a discontinuous grain boundary structure, very similar to what we have observed in other studies to be characteristic of the evolution of β phase prior to it forming continuous coverage.[4] Figure 6(d) shows Mg-rich phase (probably β) on an intragranular particle, just as occurs in the mid-plane region of the sample.

CONCLUSION

The microstructure of the naturally aged, cracked 5083 armor plate sample is consistent with sensitization due to aging at low temperatures for a long time. The grain boundary phase in the mid-plane of the plate is continuous, but non-uniform, and very thin, only 10 to 15 nm thick. ASTM G-67 tests showing mass loss results of around 19 - 25 mg/cm² are consistent with at least an intermediate degree of sensitization, but somewhat lower than expected for continuous β coverage of the grain boundaries.
Generally, the results suggest that even an extremely thin layer of $\beta$ below the resolution of conventional optical and SEM examination, and a mass loss that is not normally considered risky, can be associated with significant cracking.

**Figure 6.** Mg-rich phase precipitation in the specimen in the bottom half of the plate. (a) Z-contrast image and corresponding fine-probe EDS line scan (b). Images (c) and (d) showing the discontinuous morphology and (d) a bright-field image indicating Mg precipitation on an intragranular particle.

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REFERENCES


