The effect of plasma electrolytic oxidation on the mean stress sensitivity of the fatigue life of the 6082 aluminum alloy

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Abstract. In this work the mean stress influence on the high cycle fatigue behavior of the plasma electrolytic oxidized (PEO) 6082 aluminum alloy (AlSi1MgMn) is investigated. The present study is focused on the fatigue life time and the susceptibility of fatigue-induced cracking of the oxide coating and their dependence on the applied mean stress. Systematic work is done comparing conditions with and without PEO treatment, which have been tested using three different load ratios. For the uncoated substrate the cycles to failure show a significant dependence on the mean stress, which is typical for aluminum alloys. With increased load ratio and therefore increased mean stress, the fatigue strength decreases. The investigation confirms the well-known effect of PEO treatment on the fatigue life: The fatigue strength is significantly reduced by the PEO process, compared to the uncoated substrate. However, also the mean stress sensitivity of the fatigue performance is reduced. The fatigue limit is not influenced by an increasing mean stress for the PEO treated conditions. This effect is firstly shown in these findings and no explanation for this effect can be found in literature. Supposedly the internal compressive stresses and the micro-cracks in the oxide film have a direct influence on the crack initiation and growth from the oxide film through the interface and in the substrate. Contrary to these findings, the susceptibility of fatigue-induced cracking of the oxide coating is influenced by the load ratio. At tension-tension loading a large number of cracks, which grow partially just in the aluminum substrate, are present. With decreasing load ratio to alternating tension-compression stresses, the crack number and length increases and shattering of the oxide film is more pronounced due to the additional effective compressive part of the load cycle.

1. Introduction
Aluminum alloys are widely used for lightweight structures in the automotive and aircraft industries due to their high specific strength and fatigue resistance. To improve their poor tribological behavior, anodic oxidation is a well known method to meet the requirements. By plasma electrolytic oxidation (PEO) treatment excellent wear and high corrosion resistance can be achieved. Improved mechanical properties, i.e. hardness and E-modulus, a good adhesion on the substrate and also good biocompatibility are positive characteristics of PEO coatings [1–3].

Literature data to fatigue behavior of PEO treated aluminum alloys are limited, but the existing investigations show an inferior fatigue performance in comparison to the uncoated substrate [4–7]. With increased coating thickness this effect is more pronounced [6,7]. The loss in fatigue strength is attributed to the compression residual stresses in the coating which initiate tensile residual stresses in the substrate and lead to early crack initiation and fatigue fracture [4,6].
The high cycle fatigue performance usually depends on the applied load ratio and therefore the attached mean stress [8]. With increasing mean stress the fatigue strength is reduced. This effect can be presented graphically in Haigh or Smith diagrams. As numeric method, the mean stress influence factor \( M \) can be calculated according to Schütz [9]:

\[
M = \frac{\sigma_{ad}(R=-1)}{\sigma_{ad}(R=0)} - 1
\]

(1)

With the fatigue strength amplitudes at the load ratios \( R = -1 \) and \( R = 0 \) a factor \( M \) between 0 (fatigue strength is not sensitive to mean stress) and 1 (major influence of mean stress on fatigue strength) is calculated.

The fatigue strength of uncoated aluminum alloys shows a significant dependence on the load ratio \( R \) [10,11]. To the best of our knowledge, the effect of plasma electrolytic oxidation on the mean stress sensitivity of fatigue life has not been investigated, yet. However, Shazad et al. [12] examined the influence of load ratio on the fatigue performance for an electrolytic anodic oxidized and sealed 2214 aluminum alloy. In their study, the presence of the anodic oxide film did not significantly modify the effect of the mean stress in comparison to the uncoated substrate.

The purpose of the present study is to investigate, which effect PEO treatment has on the mean stress sensitivity of the fatigue strength. Further, the mean stress influence on the susceptibility for cracking of the anodic film is examined. As substrate material the 6082 aluminum alloy was chosen, as it is widely used in structural applications and anodic oxidation processes can be conducted without complications.

### 2. Experimental Methods

For this study the age-hardening 6082 aluminum alloy (AlSi1MgMn) of commercial purity was used. The chemical composition is shown in table 1. The material was solid-solution treated at 530 °C for 1 h, quenched in water and subsequently aged at 170 °C for 65 h to achieve peak strength. Mechanical properties determined by tensile testing are given in table 2.

**Table 1.** Chemical composition of the 6082 aluminum alloy (AlSi1MgMn).

| Element | Si  | Fe  | Cu  | Mn  | Mg  | Zn  | Al   |
|---------|-----|-----|-----|-----|-----|-----|------|
| wt.-%   |     |     |     |     |     |     | balance |
|         | 1.00| 0.21| 0.05| 0.76| 0.90| 0.02|       |

**Table 2.** Tensile properties of the uncoated peak-aged 6082 aluminum alloy.

| Yield strength in MPa | Ultimate tensile strength in MPa | Uniform elongation in % | Elongation to failure in % |
|-----------------------|---------------------------------|--------------------------|---------------------------|
| 297 ± 1               | 307 ± 4                         | 5.2 ± 0.8                | 23.7 ± 0.9                |

Prior to PEO, fatigue testing specimens with a geometry shown in figure 1 were machined from the peak-aged substrate.

**Figure 1.** Specimen geometry for fatigue testing.
In preparation for the PEO treatment, the peak-aged aluminum alloy was pickled using 3 vol.-% aqueous sodium hydroxide solution (NaOH) for 1 min at 50 °C and subsequent 10 vol.-% aqueous nitric acid (HNO₃) for 30 s at room temperature. For the anodic oxidation process, a silicate-hydroxide-electrolyte, which contained 5 g/l sodium metasilicate (Na₂SiO₃•5H₂O) and 5 g/l potassium hydroxide (KOH) in aqueous solution, was used. The PEO treatment was carried out under a symmetrical rectangular bipolar current regime with a current density of 30 A/dm² and a frequency of 50 Hz. The temperature of the electrolyte was kept constant at 20 °C and the duration of the process was 45 min. Afterwards the coated specimens were rinsed in water and dried in air.

The fatigue testing was performed in a RUMUL Testronic resonant testing machine (Russenberger Prüfmaschinen AG, Switzerland) under alternating compression-tension and tension-tension loading with load ratios of R = -1, R = 0 and R = 0.1. The fatigue tests were carried out until the endurance limit of N₀ = 10⁷ cycles was reached or until a crack occurred, which was detected by a drop in the resonant frequency of 2 Hz or more.

After fatigue testing from each specimen, longitudinal- and cross-sections were cut from the gauge length and the coating thickness and hardness was determined at the metallographically polished samples. Measurement of the coating thickness was done via optical microscopy with an Olympus GX51 (Olympus Deutschland GmbH, Germany). Hardness measurements were performed on a Fischerscope HM 2000XYm (Helmut Fischer GmbH + Co. KG, Germany). Afterwards, the polished samples were analyzed by scanning electron microscopy (SEM) on a LEO 1455VP (LEO Elektronenmikroskopie GmbH, Germany) to examine coating damage through fatigue loading. The quadrant backscattering detector (QBSD) was used for coating characterization and visualization of coating irregularities and cracks.

3. Results and Discussion

Table 3 lists the measured coating thickness and hardness. A coating thickness of about 45 µm was achieved. The non-uniformity in oxide film thickness, which shows in minimum and maximum values of 14.1 and 103.9 µm, respectively, was also observed in [4]. The large deviation of the coating thickness is owed to the PEO process and the short oxidation time, which was chosen in order to generate a thin oxide film and therefore to reduce the negative effect on fatigue strength. With longer PEO process time and therefore a thicker oxide film, the waviness of the PEO film can be reduced but the negative influence on fatigue strength would be enhanced. The coating exhibits a hardness of about 7880 MPa.

| Table 3. Properties of the PEO coating on the 6082 aluminum alloy. |
|---------------------------------------------------------------|
|                  | Average | Minimum | Maximum   |
| Coating thickness in µm | 47.3    | 14.1    | 103.9     |
| Martens hardness in MPa | 7881.3  | 5840.3  | 9661.1    |

The fatigue behavior of the aluminum alloy in uncoated and plasma electrolytic oxidized condition and its dependence on the load ratio is shown in figure 2. In table 4 the fatigue limit for N₀ = 10⁷ cycles is listed. The uncoated aluminum substrate exhibits a fatigue strength amplitude of 120 MPa at alternating tension-compression stresses. With increasing load ratio to R = 0.1 and therefore increased mean stress, the stress amplitude is reduced to 90 MPa. This significant influence of the mean stress on the fatigue life time for the aluminum substrate is in good accordance to the investigations from Zhao et al. [10] and Mayer et al. [11].

Plasma electrolytic oxidation leads in general to a decreased fatigue life time, which is explained by the internal compressive stresses in the PEO film, which, in turn, results in tensile stresses in the adjacent substrate [4–7]. Our investigation confirms this effect. For R = -1 the fatigue limit is decreased by 50 %, as compared to the uncoated substrate. Contrary to the uncoated substrate, the load ratio and therefore the applied mean stress does not influence the fatigue life time of the PEO coated
substrate. The fatigue strength amplitudes at the load ratios $R = 0$ and $R = 0.1$ are just as high as for alternating compression-tension stresses.

![Figure 2](image)

**Figure 2.** Fatigue behavior of the 6082 aluminum alloy depending on the load ratio: a) uncoated, b) plasma electrolytic oxidized (PEO).

**Table 4.** Fatigue limit at $N_D = 10^7$ cycles of the uncoated and PEO coated 6082 aluminum alloy at the tested load ratios.

| load ratio $R$ | Maximum stress $\sigma_o$ in MPa | Minimum stress $\sigma_u$ in MPa | Stress amplitude $\sigma_a$ in MPa | Mean stress $\sigma_m$ in MPa |
|---------------|----------------------------------|----------------------------------|-----------------------------------|-----------------------------|
| uncoated      | -1                               | 120                              | 120                               | 0                           |
|               | 0                                | 180                              | 90                                | 90                          |
|               | 0.1                              | 200                              | 20                                | 90                          |
| plasma        | -1                               | 55                               | 55                                | 0                           |
| electrolytic  | 0                                | 120                              | 0                                 | 60                          |
| oxidized      | 0.1                              | 120                              | 12                                | 54                          |

The Haigh diagram depicted in figure 3 presents the fatigue limit at $N_D = 10^7$ cycles depending on the mean stress for the uncoated substrate and the PEO treated condition. The mean stress sensitivity factor $M$ is indicated by the slope of the secant of the data points for the tested conditions. It is clearly visible, that the mean stress influence on the fatigue strength is diminished by PEO. The uncoated substrate exhibits a mean stress sensitivity of $M = 0.2$, calculated according to equation 1, which is typically for aluminum alloys [8,13].

By the PEO treatment the mean stress influence is neutralized to $M = 0$. For this condition our results show a fatigue stress amplitude which is unattached by the load ratio and therefore independent from the applied mean stress. This decrease in mean stress sensitivity through PEO treatment is firstly shown in these findings and for this effect no explanation can be found in literature. We cannot conclude with certainty, but supposedly the neutralized mean stress sensitivity is linked to the oxide film characteristic, especially the present micro-cracks and the residual stresses in the PEO film. It is well-known, that PEO films exhibit internal compressive stresses [4,14,15] and we assume these have direct influence on crack initiation and growth from the oxide film through the interface and in the substrate.
Figure 3. Haigh diagram representing the fatigue strength amplitudes at $N_D = 10^7$ cycles for the uncoated and the PEO coated condition at the tested load ratios. The slope of the secant indicates the mean stress sensitivity factor $M$.

Figure 4. SEM micrographs (QBSD) of the PEO treated 6082 aluminum alloy after fatigue testing at load ratio $R = -1$: a) runout (non-failure), b) fatigued specimen.

Figure 5. SEM micrographs (QBSD) of the PEO treated 6082 aluminum alloy after fatigue testing at load ratio $R = 0$: a) runout (non-failure), b) fatigued specimen.
Figure 6. SEM micrographs (QBSD) of the PEO treated 6082 aluminum alloy after fatigue testing at load ratio $R = 0.1$: a) runout (non-failure), b) fatigued specimen.

SEM micrographs (QBSD) in the figures 4-6 show the PEO film after fatigue testing at three different load ratios. The oxide film has a porous nature and branched micro-cracks are clearly visible in all tested specimens, even despite failure through fatigue did not occur (see figures 4-6 a)). The appearance of the oxide film for the fatigued specimen differs, dependent on the load ratio. At alternating tension-compression stresses a large amount of fatigue-induced radial cracks are present, which grow from the oxide film just into the substrate. A few major cracks grow long-ranging in the substrate and lead to failure (shown in figure 4 b)). With increased load ratio and therefore higher applied tension stresses, the number of cracks is still high, but, contrary to the fatigued PEO treated specimens at $R = -1$, only short-ranging cracks are present. It cannot be concluded with certainty if this crack length effect is fatigue-induced or originated by preparation and the considered sample planes. But under fully reversed tension-compression loading at $R = -1$, the oxide film exhibits major shattering due to additional fatigue damage through the compression part of the load cycle. That is why we expect that the majority of these cracks originates from the fatigue testing.

4. Summary and Conclusions

The effect of plasma electrolytic oxidation on the mean stress sensitivity of the fatigue lifetime of the 6082 aluminum alloy was investigated. An uncoated and a PEO treated condition and their fatigue strength at three different load ratios was compared. The following conclusions can be drawn:

(1) The fatigue strength is drastically reduced by PEO treatment at all tested load ratios. The loss in fatigue performance can be attributed to the porous nature of the oxide film, which promotes early crack initiation.

(2) The PEO treatment neutralizes the mean stress influence on the fatigue lifetime and therefore the fatigue strength is unattached by the load ratio. These results are firstly shown in this paper and no explanation for this effect can be found in literature. Supposedly the neutralized mean stress sensitivity is linked to the present micro-cracks and the internal compressive stresses in the PEO film which have direct influence on crack initiation and growth from the oxide film through the interface and in the substrate.

(3) The susceptibility for fatigue induced cracking of the oxide film is influenced by the load ratio. Fatigue loading at $R = 0$ and $R = 0.1$ causes a high amount of radial cracks in the coating, which grow partially just in the substrate. Under alternating tension-compression stresses, the number of cracks and their length increases. Shattering of the oxide film is more pronounced then, due to the fatigue damage through the tension and additionally through the compression part of the load cycle.
Acknowledgements
This work was financially supported by the German Research Foundation (Deutsche Forschungsgemeinschaft, DFG) in the framework of Collaborative Research Centre (Sonderforschungsbereich, SFB) 692 “High-strength aluminium-based lightweight materials for safety components”. The assistance of Dagmar Dietrich, Christel Gläser, Juliane Mehnert, Karla Muhr and Gabriele Tauchmann is gratefully acknowledged.

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