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Effect of a commercial hard anodizing on the fatigue property of a 2014-T6 aluminium alloy

R. Sadeler

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Abstract The effect of a commercial hard anodizing on the fatigue property of 2014 Al alloy which has been solution heat treated and age hardened prior to the hard anodizing of approximately 10 μm in thickness have been investigated. The results indicated that fatigue life in high stress region for aluminium alloy samples hard anodized was shorter than that of the other material condition which has been solution heat treated and then age hardened (T6 heat treated), while the life in low stress region was longer than that of the material (T6 heat treated). However, such a coating to the aluminium substrate gives rise to a significant increase in fatigue strength of 10^7 cycle in comparison with the as cast condition, but a much less increase in comparison with T6 heat treated samples. From the microscopic point of view, it has been observed that fatigue fracture of samples hard anodized initiated in the coating in high stress region in opposition to low stress region in which fatigue fracture initiation started on the interface between coating and substrate.

Introduction

The low density of aluminium leads to high specific properties which, in turn, favour the selection of aluminium alloys as the primary material for applications in aerospace and allied fields [1]. The 2000 series aluminium alloys are frequently used for making a wide variety of structural components for aircraft where damage tolerance and relatively high strength are paramount. The strength of the alloys is achieved through heat treatment, which develops fine strengthening precipitates of various structures and compositions, in addition to the relatively coarse equilibrium material formed through casting and thermo mechanical processing [2]. Sadeler et al. [3] reported a increase of approximately 43% in the fatigue strength of 2014 Al alloy (4.40 Cu, 0.68 Mg, 0.58 Mn, and 0.83 Si) in the solution treatment followed by the aging.

High resistance of metals against wear, fatigue and corrosion can be achieved by several different treatments, like mechanical, thermochemical and coating processes in addition to thermal. Hard anodizing is a prime requirement for many of above mentioned characteristics for a variety of applications. It is used, for example, for deployment mechanism components, aircraft undercarriage legs, naval magazine ammunition guides and hydraulic gears [4]. Hard anodic coatings prevent cold welding in space conditions [4]. Hard anodizing also provides an excellent base for dry lubricants [5].

The hard anodizing differs from the normal anodizing in the sense that utilizing lower electrolyte temperatures and higher current densities. For both processes, however, the fundamental mechanisms of formation and growth of anodic coating are the same, and are almost joined with the metallurgical history of the alloy production [6–10].

The present article has been conducted in order to accomplish two different purposes. Firstly, to study the fatigue behaviour of 2014 Al alloy which has been solution heat treated and age hardened prior to the hard anodizing of approximately 10 μm in thickness in

R. Sadeler (✉)
Department of Mechanical Engineering, Faculty of Engineering, Atatürk University, Erzurum, Turkey
e-mail: receps@atauni.edu.tr

addition to as-cast and T6 heat treated conditions. Secondly, to conduct a fractographic analysis of the fracture surfaces in order to obtain some information regarding the processes of crack initiation and propagation as well as the behaviour of the coating during the fatigue test.

Experimental details

Material, aging and hard anodizing treatments

The chemical composition of 2014 aluminium alloy, which was produced by direct chill continues, is shown in Table 1. After being treated in T6 condition (solution temperature at 520 °C for 2 h, aging 170 °C for 10 h), all the specimens were carefully ground with successive SiC papers grit 800–1,200 in order to eliminate the circumferential notches and scratches, and polished to with diamond paste to a grit size of 0.3 μm, then cleaned by ultrasonic cleaner prior to the hard anodizing. The same surface finishing procedures were applied to as-cast condition as well. The surface roughness within the gage length of the specimens was maintained below approximately 0.5 μm for as-cast and T6 heat treatment conditions while it increased to approximately 0.8 μm by hard anodizing condition.

All the samples were hard anodized at TINKAP Industries Inc. The specific properties of the anodic oxide will be dependent upon the specific alloy being treated and the process parameters being applied. Hard anodizing is produced by immersing the aluminium components into an electrolyte solution. Hard anodizing may be provided in thickness ranging from 10 μm to 50 μm, depending on the alloy and the application. For hard anodizing coating, the bath was selected which has the 50% sulphuric acid and 50% water. The process took place under the following conditions: bath temperature with range of –5/15 °C, current density with 1 $A/dm^{2,}$ process time with 1 h. A thickness of approximately 10 μm, which was corroborated by means of the image analysis, was attained.

The microhardness on the cross-section of the coating was measured with Vickers hardness, using a PC controlled Buehler–Omnimet tester. Vickers hardness numbers were obtained by averaging eight measurements on each specimen with a load with 200 g. Microstructure and hard anodized substrates were characterised using a Jeol 6400 Scanning Electron Microscope (SEM).

Table 1 Chemical composition (wt%) of 2014 aluminium alloy

	Cu	Mg	Mn	Si	Fe	Cr	Zn	Al
(Wt%)	4.51	0.39	0.60	0.68	0.33	0.05	0.090	Balance

Fatigue tests

Fatigue test were carried out under rotating bending conditions ($R = -1$), employing a R.R Moore equipment, at 50 Hz with specimens shown in Fig. 1 machined to dimensions by a CNC machine. All the tests were carried out in air at room temperature (23 °C). In this type of test, the specimen is subjected to a dead-weight load while bearings allow the rotation. At the mid point of the circular test section surface, the material is subjected to sinusoidal stress amplitude from tension on the top to compression on the bottom with each rotation. The bending moment applied to the specimens was determined as a function of the alternating stress and diameter of the sample by means of simple relationship:

$$M = \frac{\pi}{32}\sigma_a d^3 \tag{1}$$

where, M represents the bending moment, σ the maximum alternating stress and d the specimen diameter. In this type of test, the maximum number of cycles to failure is obtained at a given stress level. The stress σ is continually reduced, and the number of cycles to failure N_f, increases. Finally, the limiting value of stress (endurance/fatigue limit) is reached where fatigue failure will not occur [11, 12]. However, for aluminium alloys as most nonferrous, the σ–N_f curve does not approach an asymptote. In general, a fatigue limit is often arbitrarily defined as the fatigue strength at 10^7 cycles and should be experimentally determined [13].

The portion of the uncoated specimens having continuous radius was polished to get very small surface roughness. This was done to avoid the most likely influence of surface roughness on fatigue strength.

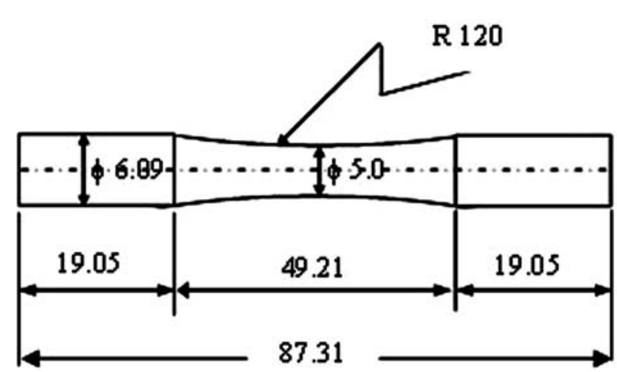

Fig. 1 Rotating bending fatigue specimens (dimensions in mm)

Tests were terminated by complete fracture of all the specimens. A total of 24 samples were employed for evaluating the fatigue properties of the material under the three different conditions investigated. The relationship between the alternating stress and the number of cycles to failure for the all the condition analyzed is defined by the Basquin equation [14];

$$\sigma_a = \sigma'_f (N_f)^b \quad (2)$$

where, σ_a = stress amplitude; σ'_f = fatigue strength coefficient; b = fatigue strength exponent; N_f = number of cycles to failure.

Results

Characteristics of hard anodic oxide films

As shown in Fig. 2, the hardness of 2014 Al alloy was increased by T6 heat treatment because the solubility of copper in aluminium increased markedly with solution treating temperature. During quenching, this Cu was retained in solution. Thus, Al-rich phase would contain Cu in a supersaturated solid solution at room temperature. In the aging, fine particles of $CuAl_2$ formed and precipitated in solution. Thus, the hardness of alloy increased by means of artificially age hardening [15].

Besides, hard anodizing coating to aluminium alloy T6 heat treated gives rise to a significant increase of hardness in comparison with other material conditions because hard anodizing, which is generally made by anodizing in sulphuric acid at low temperature, produces a hardness coating with large cells and small diameter pores. The yielding strength of the base-material (T6 condition) was approximately 415 MPa in internet [16]. The standard deviations (SD) for as-cast, T6 heat treated and hard anodized conditions were obtained as 4.6, 4.8 and 8.5, respectively.

Figure 3 reveals a view of the interface between hard anodizing coating (HA) and substrate before fatigue test. The anodic film seems to be uniform and has evidently adequate adhesion characteristics due to the absence of visible cracks along the interface.

Fatigue tests

The stress amplitude (σ_a) for the fatigue tests of the samples T6 heat treated and hard anodized was appointed in the range of about 216–327 MPa, whereas for the as-cast samples in the range of about 166–254 MPa. Tables 2–4 present the σ_a in terms of the number of cycles to failure N_f data concerning all the conditions investigated. Fig. 4 illustrates their graphical representation. As mentioned earlier, each experimental point on the graphs represents a mean value of three samples.

Each group of experimental data have been represented by means of curve line on a normal plot considering the validity of the Eq. 2 earlier represented. Both values of each conditions analysed were determined by means of the least square fitting the Eq. 2 to experimental values of σ_a and N_f. Table 5 summarizes the values of the parameters σ'_f and b for the three set of data represented in Fig. 4.Such parameters would be of upmost importance for the design of structural components and parts could fail by high cycle fatigue under service.

The results obtained, regarding the fatigue properties of the hard anodized and other material conditions, have been plotted in Fig. 4. At raised alternating stress

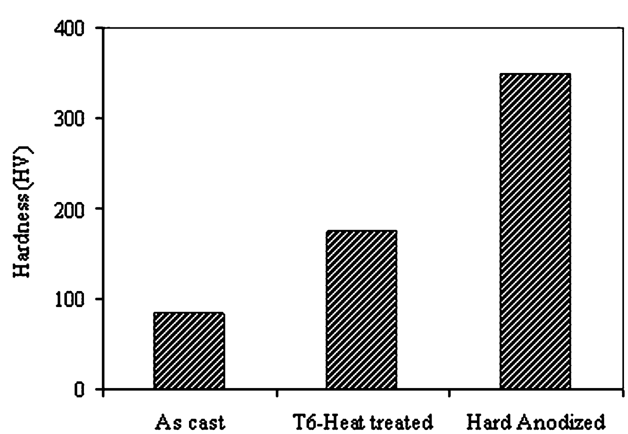

Fig. 2 Influence of T6 heat treatment and hard anodizing process on hardness of 2014 Al alloy

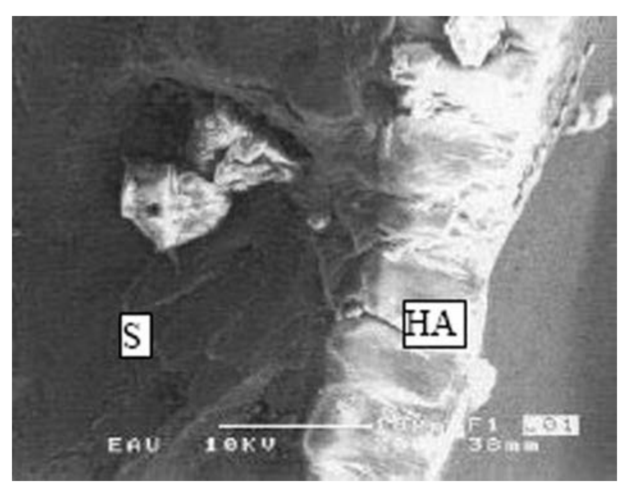

Fig. 3 SEM views of the interface between the hard anodizing coating and substrate before fatigue test. The hard anodizing has been identified as (HA) and the substrate as (S)

Table 2 Stress amplitude in terms of the number of cycles to failure for as-cast samples

Material condition	Stress amplitude (MPa)	Mean number of cycles to failure (×10^3)	Standard deviation (SD)
As cast	166.0	30626	4907915
	179.0	18918	3667430
	191.6	16442	2915224
	204.2	1443	302223
	216.7	790	273846
	229.3	615	151555
	242.0	495	102327
	254.0	330	55677

Table 3 Stress amplitude in terms of the number of cycles to failure for T6-heat treated samples

Material condition	Stress amplitude (MPa)	Mean number of cycles to failure (×10^3)	Standard deviation (SD)
T6-Heat treated	216.0	74150	9297296
	231.8	68434	5507510
	237.7	1500	275189
	244.3	1085	228164
	259.4	997	102220
	271.9	595	65795
	299.4	187	12529
	327.0	50	6244

Table 4 Stress amplitude in terms of the number of cycles to failure for hard anodized samples

Material condition	Stress amplitude (MPa)	Mean number of cycles to failure (×10^3)	Standard deviation (SD)
Hard anodized	216.0	38074	7953582
	231.8	17012	5574358
	237.7	3402	849987
	244.3	2139	387828
	259.4	1243	252168
	271.9	466	108018
	299.4	320	78479
	327.0	135	53563

level (300 MPa), the curve obtained for the hard anodized samples indicates a reduction in fatigue life, in comparison with the T6 heat treated samples, of approximately 2.4-fold, whereas at low stresses (216 MPa) the hard anodizing coating gives rise to an increase in the fatigue life about a 2.2-fold increase.

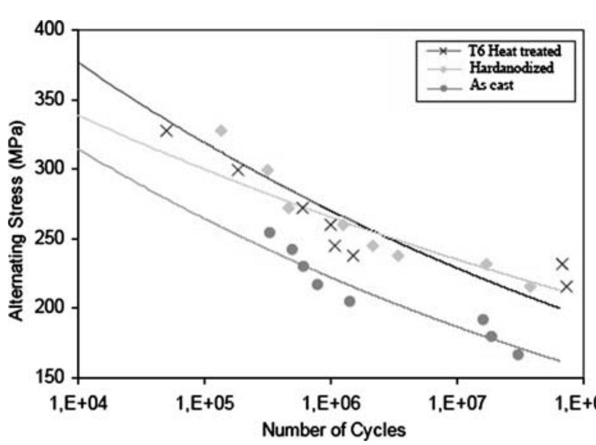

Fig. 4 Mean number of cycles prior to failure N_f a function of the alternating stress to applied to material σ_a for as cast, T6 heat treated and hard anodized samples

Briefly, these observations obtained from Fig. 4. exhibit some significant facts. Firstly, fatigue life in high stress region for aluminium alloy samples hard anodized was shorter than that of the other material condition which has been solution heat treated and then age hardened (T6 heat treatment), while the life in low stress region was longer than that of T6 heat treatment. Secondly, a anodic film to the aluminium substrate gives rise to a significant increase in fatigue strength of 10^7 cycle in comparison with the as cast condition, whereas a much less increase in comparison with T6 heat treated samples.

In order to analyses further this variation in fatigue life, Fig. 5 illustrates the change in the fraction of cycles to failure in T6 heat treatment and hard anodizing conditions, and cycles to failure of the as cast as a function of the stress amplitude. However, since the as

Table 5 Parameters involved in Eq. 2 for the description of the σ_a in terms of N_f for the conditions tested

Material and condition	σ_f' (MPa)	b	Correlation coefficient (R) (%)
2014 Aluminium, as cast	630.29	–0.07547	94
2014 Aluminium, T6-heat treated	730.86	–0.07204	95
2014 Aluminium, hard anodized	550.28	–0.05276	92

cast samples displayed lower stress amplitude than T6 heat treated and hard anodized samples, it was necessary to recalculate the cycles to failure at the stress amplitudes to which the as cast material was subjected by means of Eq. 2 describing the experimental data of the T6 heat treated and hard anodized materials. Thus, it can be observed that in high stress amplitude (310 MPa), fatigue life of hard anodized specimens increased by approximately 3 times in relation to behaviour of the as cast material, while in low stress amplitude (190 MPa) maximum increase was obtained by approximately 63 times. At large stress amplitudes the curve tend to intercept to stress axis which reveals that under high stress conditions the hard anodizing coating would not have any beneficial influence. Similarly, for T6 heat treated samples, it is clear that increase in fatigue life appear almost constant (approximately 16 times). At approximate 250 MPa, both curve coincided each other.

According to results presented in Table 5, the change in absolute value of the fatigue strength exponent (b) in Eq. 2 for hard anodized samples is more evident than T6-het treated material condition. Particularly, in as cast and hard anodized conditions, curves tend to appear as almost parallel caused by the relatively small change in the slopes. Nevertheless, correlation coefficients of the curve lines considered were found between 92% and 95%. This indicates that this equation is applicable to the fatigue behaviour of all the conditions for 2014 aluminium alloy.

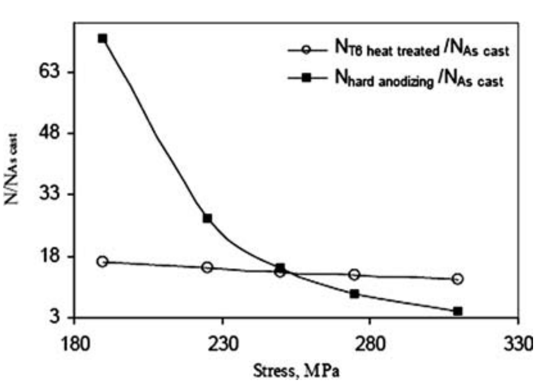

Fig. 5 Number of cycles to failure for the T6 heat treatment and hard anodizing conditions by corresponding number of cycles to failure of the as cast as a function of the alternating stress

Evaluation of the fracture surfaces of the fatigued samples

Several specimens tested at different alternating stresses were examined after failure by SEM in order to study more closely the sites of crack initiation for as cast and T6 heat treatment conditions analysed. Fig. 6a illustrate a typical micrograph of the general view of the fracture surface of a sample in the as cast condition, tested 254 MPa. The fracture surface is not flat, revealing the presence of a number of fracture steps, which points out that fracture has occurred as a consequence of the propagation of several cracks that initiated from surface of the specimen. Fig. 6b represents the site initiation of the main crack on the surface of the sample. The zone of final rupture is in the cross section of the specimen. Fig. 7a illustrates a typical fracture surface for T6 heat treatment condition when the specimens are tested at 216 MPa. In this case, it is possible to identify the initiation site of the one dominant crack which gives rise to the presence of a step on the fracture surface (Fig. 7b).

On the other hand, a number of specimens tested under different alternating stress conditions were closely examined after failure by SEM in order to explain reason of the shorter fatigue life in the hard anodized specimens at high stress region in comparison with T6 heat treated samples.

Figure 8 reveals a view of the fracture surface of a hard anodized sample, tested at 271.9 and 231.8 MPa. Though the fatigue cracks of the sample hard anodized are initiated within coating and propagates towards the substrate in higher alternating stress, it seems that the fatigue cracks of the samples hard anodized are initiated from the interface between the coating and substrate in lower alternating stress.

Conclusion

A number of fatigue experiments were conducted to evaluate the effect of the hard anodizing coating of

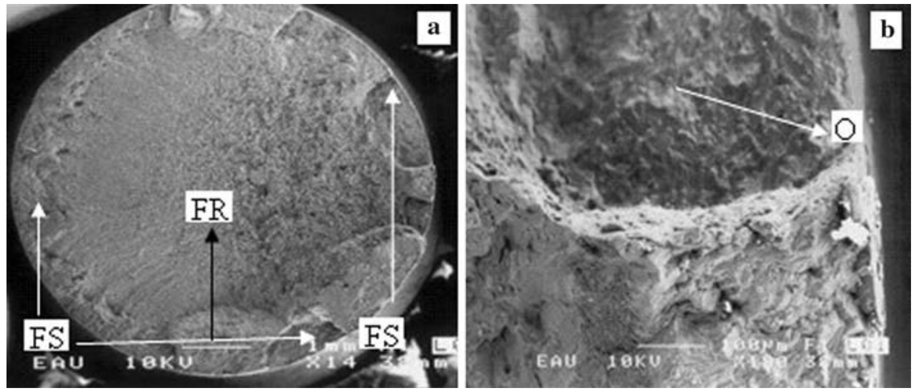

Fig. 6 (**a**) General view of the fracture surface of a sample in the as cast condition, tested at 254 MPa. The arrows reveal the presence of a number of fracture steps (FS); while the black arrow shows the zone of final rupture (FR); (**b**) Detailed view of the initiation site of the crack on the surface of the sample. The arrow presents the origin (O) of the dominant crack

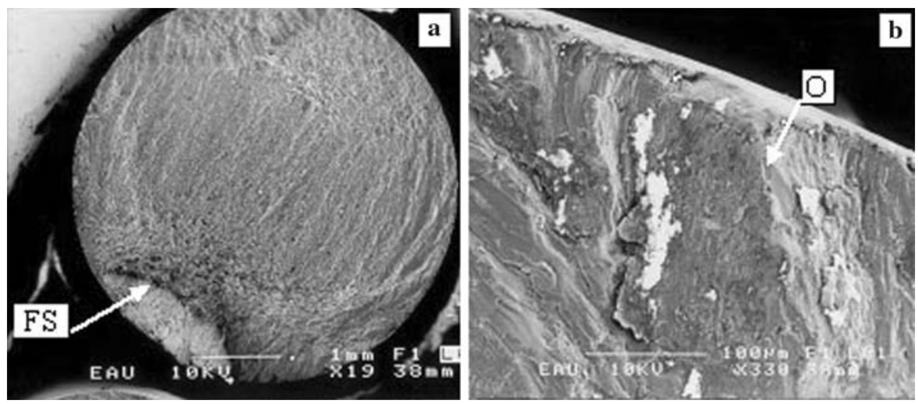

Fig. 7 (**a**) General view of the fracture surface of a sample in the T6 heat treatment condition, tested at 216 MPa. The arrow reveals the presence of only one of fracture step; (**b**) Detailed view of the initiation site of the crack on the surface of the sample. The crack initiation site is identified with (O)

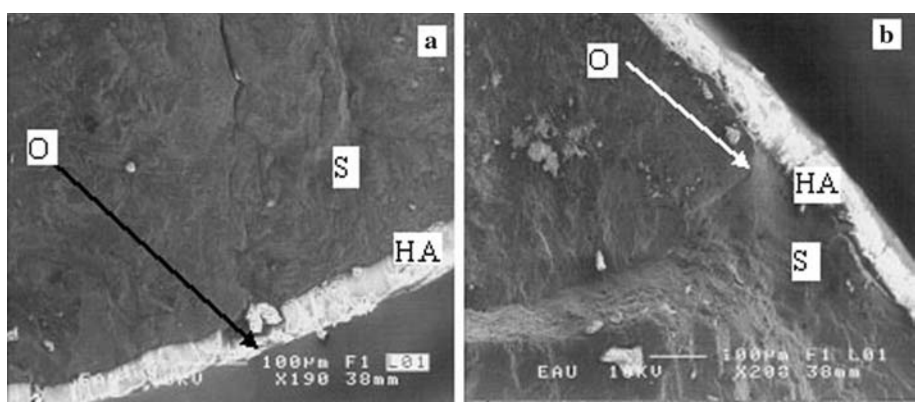

Fig. 8 (**a**) SEM view of the fracture surface of a hard anodized sample, tested at 271.9 MPa. The fatigue cracks have nucleated within the hard anodizing coating and propagate towards the substrate. The hard anodizing coating has been identified as (HA) and the substrate as (S). (**b**) SEM view of the fracture surface of a hard anodized sample, tested at 231.8 MPa. The crack initiation point is observed on the substrate

approximately 10 μm thick on samples of 2014-T6 aluminium alloy.

The main results obtained are summarized as follows:

1. The hardness of 2014-T6 aluminium alloy is significantly improved by the hard anodizing process because hard anodizing produces a hardness coating with large cells and small diameter pores.
2. Both T6 heat treatment and hard anodizing conditions presented total fatigue life superior to the as-cast condition.
3. The effect of anodising on fatigue properties depends on the stress level. The increase in the fatigue life of the hard anodized samples is reached at lower alternating stresses applied during the fatigue tests, whereas at elevated stresses the reduction in the fatigue life is observed in comparison to T6 heat treated condition.
4. The analysis of the fracture surface of all the conditions tested, revealed that at low alternating stresses, the dominant crack responsible for the fracture process is developed at the interface of the

substrate and coating and usually propagated into metal since the hard anodic film adhered so strongly to the substrate metal, whereas the crack started within film at high alternating stresses. Therefore, it is concluded that the decrease in fatigue life in hard anodized samples occurs as a result of the passage of the fatigue cracks from the coating to the substrate.

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