ABSTRACT

A simple electrochemical technique is described, which images and quantitatively measures the distribution and severity of fatigue damage in aluminum alloys. The technique is based upon (i) the creation of microcracks in a surface anodic oxide film during fatigue of the underlying metal, and (ii) the detection of these microcracks by contacting the surface with a gel electrode. When a voltage pulse is applied, current passes through the fatigue-induced microcracks in the oxide film, and an image of the sites of current flow is retained in the surface of the gel. The capabilities of the technique are illustrated by measurements on 6061-T6, 7075-T6 and 2024-T4 aluminum. The electrochemically formed images correlated directly with scanning electron micrographs of the specimens. Hairline fatigue cracks \( >10 \ \mu\text{m} \) long are easily imaged, while the charge flow during the formation of the image is a quantitative measure of the crack length. The accumulation of fatigue deformation prior to the appearance of a fatigue crack is also detected, and in this regard the sensitivity of the gel electrode exceeds that of a scanning electron microscope. The distribution of fatigue deformation may be mapped as early as 1% of the fatigue life, and the charge flow to the regions of most severe damage increases systematically with fatigue cycling as the density of microcracks in the oxide increases. The simplicity of this electrochemical gel electrode method renders it directly applicable to field investigations, and provides a new tool for quantitatively assessing the distribution of fatigue damage.
INTRODUCTION

The inability to measure quantitatively the extent of fatigue damage in metallic structures, is an old problem which continues to resist extensive research efforts with an ever increasing variety of techniques. Traditionally, nondestructive evaluation techniques have focussed on the detection of fatigue cracks, and have been evaluated in terms of the smallest crack detectable. While this in itself has proven to be a difficult enough problem, it is nevertheless easier than detecting the more subtle processes associated with persistent slip band formation, that occur during the earlier so-called crack initiation stage of metal fatigue. This paper describes a simple electrochemical technique, hereafter referred to as the gel electrode, which not only can detect and quantitatively assess the extent of damage during the crack initiation stage, but also can provide images of minute fatigue cracks which develop later in life.

The gel electrode technique relies upon the detection of fatigue induced microcracks in a thin surface oxide film. This rupturing of surface oxide films by the accumulation of fatigue damage in the underlying metal was first studied extensively by the exoelectron method, that is by the photoelectrons which are emitted preferentially from the freshly exposed metal surfaces. However, the technique was restricted by the need to perform the fatigue test in a vacuum, to prevent reoxidation of the microcracks.

The gel electrode method does not suffer from this drawback, so fatigue testing may be performed under normal atmospheric conditions. The procedure is a modified version of a technique developed by Klein for the study of defective sites in anodic oxide films. The microcracks in the oxide are detected by contacting the specimen with a dried surface film on a gel-electrolyte, containing potassium iodide (KI) and starch, and applying a voltage pulse to stimulate a corrosion current which flows preferentially to the microcracks. This current anodically oxidizes the KI to release iodine ions, which react with the starch to form a black complex. These complexes are retained in the skin of the gel and provide an accurate spatial representation of the sites of the microcracks in the oxide film, i.e. the sites of fatigue damage.

The utility of the gel electrode is illustrated by experiments on 1100-0, 2024-T3, 6061-T6 and 7075-T6 aluminum. Fatigue deformation is measured as early as 0.2% of the fatigue life, with a sensitivity that surpasses the capabilities of optical and scanning electron microscopy. In addition, the gel electrode provides high contrast images of fatigue cracks, some of which approach only 10 μm in length.
EXPERIMENTAL PROCEDURE

The procedure consists of three steps: (i) specimen preparation including anodization to produce a surface oxide film 14 nm thick, (ii) fatigue cycling of the specimen, and (iii) gel electrode printing of the microcracks in the oxide film.

Specimen Preparation

The specimens of 7075-T6, 6061-T6, 2024-T3 and 1100-0 aluminum were machined from sheet material 1.5 mm thick to produce a conventional tapered bending fatigue specimen. When the sole experimental objective was to image fatigue cracks, a small notch was filed in one edge to accelerate the process of crack initiation. A few specimens were mechanically polished to aid subsequent examination by optical and scanning electron microscopy, but polishing is not necessary for successful gel electrode imaging. The other specimens were cleaned in chromic acid at 70°C. All specimens were then anodized in a 3% solution of tartaric acid at a potential of 10 volts to form a surface oxide film 14 nm thick.

Fatigue Cycling

The flat sheet specimens were fatigued by reverse bending, and required $\approx 10^5$ cycles to initiate a fatigue crack at the notch. In the absence of a notch the strain amplitude was increased to cause failure after $\approx 10^5$ cycles.

Gel Electrode Imaging

The electrolyte consists of an agar gel containing 0.2 molar potassium iodide, 0.05 molar borax and 30 grams of starch per liter. This warm fluid mixture was dispensed into small lengths ($\approx 3$ cm) of plastic tube 6 mm in diameter. The plastic tube was over-filled so that, upon cooling, the liquid formed a smooth hemispherical gel tip at one end of the tube as shown in Fig. 1. A piece of aluminum wire was sealed into the other end of the tube to serve as a cathode. In approximately five minutes a flexible skin formed on the gel tip. This tip was then pressed gently against the surface of the specimen and a pulse of negative potential was applied to the cathode.

The images formed in the skin of the gel were viewed and photographed with an optical microscope. These images were then correlated with observations of the specimens in a scanning electron microscope (SEM). The latter also provided the basis for independent measurements of the length of any fatigue cracks. The flow of current during image formation was recorded on a Nicolet Digital oscilloscope and stored on magnetic discs. This information was later displayed on an X-Y recorder, and the total charge
flow during imaging was obtained by measuring the area under the curve.

OXIDE FILM RUPTURE

The oxide film thickness of 14 nm was selected for the following reasons. (i) It is thicker than the natural oxide (4 nm) that spontaneously reforms in the fatigue induced microcracks. This ensures that during gel electrode imaging, the electrically driven corrosion currents pass primarily through the thin oxide in the microcracks. (ii) It is thin enough to be ruptured by the fatigue induced emergence of persistent slip bands. This is illustrated by the photoelectron micrograph of a fatigued specimen of 6061-T6 aluminum in Fig. 2, where the microcracks in the oxide are imaged by the exoelectron emission (the white regions). This mode of film rupture is essential for the quantitative measurement of the early stages of fatigue deformation by the gel electrode.
MEASUREMENT AND IMAGING OF METAL FATIGUE

Fig. 2. Photoelectron micrograph of a specimen of 6061-T6 aluminum coated with a 14 nm oxide and fatigued for 10^6 cycles at a cyclic strain of 6.8 x 10^{-3}. The white regions of exoelectron emission define microcracks in the oxide film produced by slip band extrusion from the substrate metal.

MEASUREMENT OF FATIGUE DEFORMATION

In these experiments a 5 volt, 5 sec pulse was applied to the gel electrode, to provide good sensitivity for the detection of microcracks in the oxide film. (The criterion adopted was that relatively little current should pass through the intact regions of the oxide film.) The flow of current produced by such a long voltage pulse overexposed the gel electrode image, and produced arrays of black spots on the tip of the gel. These spots serve as a useful visual indicator of the extent and location of microcracking of the oxide film, as well as providing a measure of the area of contact (√0.12 cm²). Each specimen was surveyed along the length of each side by contacting a series of gel tips at intervals of √5 mm along the center line. The appearance of the gel tips, and the measured charge flow, from such a survey of a fatigued specimen of 6061-T6 aluminum is shown in Fig. 3. This specimen had been fatigued for 10^6 cycles (√7% fatigue life) at a surface cyclic strain of ± 3.1 x 10^{-3}. The gel electrode has clearly detected a well-developed distribution of fatigue damage. The density of
Fig. 3. The flow of charge during gel electrode imaging as a function of distance along the center line of a specimen. Photographs of the gel tips (enlarged 50%) show a correlation between the spot density and the charge density. Specimen at ~7% of fatigue life.
spots on the gel tip correlate with the measured charge flow, and both confirm the disruption of the surface oxide to be the most severe at a location about 2.5 cm from the large shoulder of the specimen. This maximum value of the charge flow is \( \sqrt{100} \times \) greater than the charge which flows to a non-fatigued specimen.

The flow of current to a specimen of 6061-T6 aluminum is illustrated by the photograph of two oscilloscope traces in Fig. 4. The lower trace was obtained before fatigue cycling, while the upper one corresponds to the location of maximum charge flow after only 700 fatigue cycles, or 0.5% of fatigue life. The wide separation between the two current traces vividly demonstrates the high sensitivity attainable with the gel electrode.

![Fig. 4. Photograph of oscilloscope traces of current flow to a specimen of 6061-T6 aluminum. Lower trace 0 cycles, upper trace 700 cycles (0.5% life).](image)

The density of charge flow increases substantially with additional fatigue cycling. This is also true for the density of spots that develop on the gel tips, which provide a good visual indication of the severity of the fatigue. But from a quantitative viewpoint, the significant parameter is the maximum value of charge density observed to flow during imaging. The effect of fatigue cycling on the observed maximum charge density, for a large number of specimens of 6061-T6 and 1100-0 aluminum, is summarized in Fig. 5. Each data point represents a different specimen. Note that the abscissa in Fig. 5 is expressed in terms of the fraction of
fatigue life expended rather than fatigue cycles. This compensates for a slight difference in the fatigue life for the two sets of experiments: the average life of similarly prepared specimens of 6061-T6 aluminum was $1.4 \times 10^7$ cycles, while that for 1100-0 aluminum was $1.0 \times 10^6$ cycles. The range of charge density observed to flow to non-fatigued regions is also indicated. While this background limits the ultimate sensitivity of the gel electrode technique, fatigue damage is still readily detected as early as $\approx 0.2\%$ of the fatigue life.

Fig. 5. The density of charge which flows to specimens of 1100-0 and 6061-T6 aluminum as a function of the fraction of fatigue life, during the application of a 5V 5s pulse.
During the early stages of fatigue the charge flow to the 6061-T6 aluminum was an order of magnitude larger than that to the 1100-0 (Fig. 5). However, this does not denote a greater sensitivity for fatigue detection in 6061-T6, because the initial background of charge flow in the absence of fatigue is also an order of magnitude larger (Fig. 5). This higher background for the 6061-T6 material is attributed to a larger concentration of defects in the oxide film formed on such an alloy, as opposed to that which forms on the much higher purity (99%) 1100-0 aluminum. Even the latter contains some defects, which have been shown to control not only the current flow from an electrolyte, but also the resistance to rupture during tensile deformation. Similarly, during the early stages of fatigue, the highly defective oxide on 6061-T6 aluminum will rupture more readily than the less defective oxide on 1100-0 aluminum, resulting in the observed marked difference in charge flow (Fig. 5). However, with continued fatigue cycling the low strength 1100-0 material develops much more severe surface deformation than does the much stronger 6061-T6 alloy, imposing much greater localized stresses on its oxide film. These higher stresses rupture the film at more locations so that eventually the charge flow to the 1100-0 aluminum equals that to the 6061-T6 alloy.

The scatter bands in Fig. 5 have been drawn to encompass all data points. Most of this scatter is considered to originate from specimen to specimen variation. For example, the width of the band for 6061-T6 aluminum is about a factor of 4, while the range of fatigue lives for similar specimens was found to be a factor of 3. Thus the quantitative relationship evident in Fig. 5 provides a basis for measuring the severity of the fatigue damage, and for estimating the fatigue life. For example, if a charge flow of $5 \times 10^2$ coulombs/cm$^2$ is measured, then a specimen of 6061-T6 aluminum has consumed between 0.5% and 2% of its life, while a specimen of 1100-0 aluminum is between 3% and 7% of life.

This ability to detect fatigue damage earlier than 1% of the fatigue life, demonstrates that the development of microcracks in a surface oxide film (in conjunction with the gel electrode imaging) provides the basis of a very sensitive and quantitative tool for the assessment of fatigue damage. During subsequent microscopic examination of many of the specimens, it was found that this sensitivity for the detection of surface fatigue deformation surpassed the capabilities of optical and scanning electron microscopy. The only features encountered in the SEM, for example, were corrosion artifacts produced by the gel electrode imaging process, and these only occurred in regions where the current flow had indicated the presence of fatigue-induced microcracks in the oxide.
IMAGING OF FATIGUE CRACKS

The detection and imaging of fatigue cracks with the gel electrode is very straightforward, and does not require the high sensitivity described above. So in these experiments a 10 volt pulse of much shorter duration was applied to the gel electrode, to produce images which could be compared with direct examination of the cracks by conventional forms of microscopy. The sensitivity and spatial resolution attainable with the gel electrode are determined to some extent by the charge flow, and this can be controlled by the duration of the voltage pulse. For a 10 volt pulse, a duration of 100 ms consistently produced an image which is clearly visible to the unaided eye, but subsequent examination of the gel with an optical microscope reveals that the image is overexposed, so that some of the more subtle features are not resolvable. This is illustrated in Fig. 6 by the enlarged view of an image of a crack, which was grown from a notch in a specimen of 2024-T3 aluminum. Subsequent examination of this specimen in a scanning electron microscope showed that, while the width of the crack is greatly exaggerated in this gel electrode image, the important parameter of total crack length (1.7 mm) is very clearly defined. In addition, the feature at 'A' in Fig. 6 was correlated with a second crack, which as shown by the scanning electron micrograph in Fig. 7 has a total length of only \( \sim \)100 \( \mu \)m.

Fig. 6. Optical micrograph of a gel electrode image formed by a 10V, 100 ms pulse, showing two fatigue cracks (A and B) in 2024-T3 aluminum.
If a 10 volt pulse of 1 ms duration is applied to the gel electrode, the image produced has good spatial resolution, but it is so faint that it is difficult to photograph with an optical microscope. A 10 volt pulse of 10 ms duration is in general the best compromise, providing an image with sufficient spatial resolution and contrast to permit direction correlation with scanning electron micrographs of the specimen itself. This is illustrated in Fig. 8 by the distinctive geometry of the tip of a crack in 2024 aluminum. (Note that the gel electrode always produces a mirror image.) The gel electrode has faithfully imaged all the features of the crack visible in the SEM, and some of these are only \( \sqrt{30} \) \( \mu \text{m} \) in length. Even shorter cracks can be imaged, but if a crack is only \( \sqrt{10} \) \( \mu \text{m} \) in length, the image formed by a 10 ms pulse is often simply a spot rather than a linear feature. While this represents the limit of resolution of an image formed in this way, it is not the limit of sensitivity, because on several occasions the fatigue damage associated with spots in the gel image could not be identified with the SEM. Presumably these spots represented detection of fatigue deformation as discussed in the previous section.

Thus the gel electrode images provide a very simple and direct measure of the length of a fatigue crack, while at the same time revealing their often complex geometries. A second measure of crack...
Fig. 8. (a) Optical micrograph of a gel electrode image formed by a 10V, 10 ms pulse showing the tip of a fatigue crack in 2024 aluminum. (b) Scanning electron micrograph of the fatigue crack imaged in (a). Note the top to bottom mirror imaging by the gel electrode.
length is provided by the flow of charge during the formation of the image. As shown in Fig. 9 the charge is proportional to crack length, and within the limits of experimental scatter this result is independent of alloy composition. Note that even in the absence of a crack the charge flow can be as large as $2.5 \times 10^{-6}$ coulombs. This is due to two factors: (a) $1 \times 10^{-6}$ coulombs are required to charge up the capacitance formed by the conductive gel and the oxide coated metal, and (b) $< 1.5 \times 10^{-6}$ coulombs 'leak' through defects in the oxide film. This latter contribution varies rather unpredictably and limits the sensitivity of charge flow measurements for the detection of fatigue cracks. Thus, experience to date indicates that cracks of length $> 200 \mu m$ can be reliably detected by charge flow measurements.

![Fig. 9. Effect of crack length on the charge flow during the formation of a gel electrode image with a 10V, 10 ms pulse.](image-url)
On the other hand, the images themselves do not suffer from this limitation, since a small uniform charge flow does not create a visible image. Thus, image inspection by optical microscopy can definitely reveal the presence of fatigue cracks as short as 10 μm.

SUMMARY

The gel electrode is a new tool for the study and assessment of surface fatigue damage in metals. The technique is very simple, requires minimal surface preparation, and yet provides a sensitivity that exceeds the capabilities of optical or scanning electron microscopy. Experiments on a range of aluminum alloys have demonstrated the following capabilities.

1. Fatigue cracks in the metal, some as short as only \( \sim 10 \) μm in length, may be consistently detected and located.

2. The flow of charge during imaging of a fatigue crack is proportional to the crack length, and independent of the composition of all alloys studied to date. This charge flow provides an alternative measure of crack length, and can detect cracks as short as 100 μm.

3. Fatigue deformation may be detected as early as 0.2% of the fatigue life.

4. The flow of charge is a measure of the severity of the fatigue deformation, and provides a basis for the early prediction of fatigue life.

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REFERENCES


