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## Effect of high-phosphorus electroless nickel coating on fatigue life of Al-Cu-Mg-Fe-Ni alloy

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The fatigue behaviour of Al-Cu-Mg-Fe-Ni alloy was evaluated in four different conditions: uncoated, after second zincating pre-treatment, coated with high phosphorus electroless nickel layer and coated with NiP and heat treated for hydrogen release. The results of the fatigue test indicated an increased fatigue life of the coated aluminium alloy up to 150%. This improvement was associated with the higher strength of the coating as compared to the substrate and with the development of compressive residual stresses in the coating during deposition.

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Electroless nickel plating is an effective method to increase the corrosion and wear resistance of structural materials such as steel and aluminium alloys [1,2]. One of the major parameters that influence the properties of the electroless nickel–phosphorus (NiP) coating is phosphorus content. For instance, NiP coatings with high phosphorus content (>8 wt.% P) have improved corrosion resistance and compressive stresses as compare to low- and medium-phosphorus containing coatings [2].

Very often NiP coated materials undergo cycling loading, which can induce fatigue fracture. The effect of the high-phosphorus electroless nickel coatings on fatigue behaviour of steel substrates has been investigated by several authors [3–6]. It was found that the ratio substrate/coating strength, coating thickness, coating internal residual stresses as well as the heat treatments applied for hydrogen release or coating hardening are among the factors which affect the fatigue properties of the NiP coated steel substrates. For example, Puchi et al. [3] have reported an increase in the fatigue life of two low strength (tensile strengths in the range of 220-440 MPa) carbon steels (i.e. AISI 1010 and 1045) coated with about 20–22 μm electroless Ni–10 wt.% P coating. The increase in the fatigue life was higher in the case of AISI 1010 as compared to the AISI 1045. However,

deposition of high-phosphorus electroless NiP coatings on high strength steels proved to be detrimental to their fatigue properties. Thus, Wu et al. [4] reported a reduction in the fatigue limit of about 39% for the quenched and tempered 30CrMoA steel coated with a 10 wt.% P electroless nickel coating. Berríos et al. [5] have studied the effect of NiP layer thickness on fatigue properties of the AISI 1045 plain carbon steel. It was found that the deposition of a 7 µm NiP layer did not affect the fatigue life of the coated steel whereas with the increase of layer thickness up to 37 µm the fatigue life decreases. On the other hand, Garcés and co-workers [6] reported that when 12–14 wt.% P is deposited on a quenched and tempered AISI 4340 (CrNiMo) steel, the fatigue life can decrease by almost 92% when the coating/substrate system is heat treated using a two steps heat treatment, i.e. 1 h at 200 °C followed by 1 h at 400 °C.

In a recent work, Puchi-Cabrera et al. [7] showed that approximately 18 wt.% P electroless nickel coating can improve significantly the fatigue and the corrosion-fatigue performance of 7075-T6 aluminium alloy. According to authors the higher mechanical properties of the NiP coating in comparison with the aluminium substrate and its very good adhesion contributed to better fatigue performance of the coated system. A few studies on the medium-phosphorus deposits (5–8 wt.% P) indicated that NiP coating can either increase [8] or decrease [9] the fatigue strength of aluminium alloys. There are no reported studies in the open literature with respect to fatigue behaviour of electroless NiP deposits on

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2618 aluminium substrates. In the present investigation the fatigue behaviour of the electroless high-phosphorus nickel deposit on aluminium 2618-T61 substrate has been studied.

The substrate used in the present study was a wrought aluminium alloy 2618 supplied as extruded bars with a diameter of 75 mm. The chemical composition of this alloy is (in wt.%) 2.35 Cu, 1.58 Mg, 1.07 Fe, 1.07 Ni, 0.17 Si, 0.052 Zn, 0.012 Cr, 0.075 Ti and Al balance. The material was further extruded to 22 mm diameter and heat treated to T61 temper condition (solutioning at 530 °C, water quenching and artificial aging at 200 °C for 20 h). To be used for axial fatigue testing the cylindrical specimens with a gauge diameter of 8 mm, a gauge length of 10 mm and a 50 mm fillet radius were subsequently machined and then polished to a surface roughness of  $R_a = 0.2 \, \mu m$ .

The deposition of the electroless NiP coatings was carried out using a proprietary electroless nickel solution for high phosphorus content. The deposition process was conducted at  $88 \pm 2$  °C and solution pH of  $4.8 \pm 0.1$ . Prior to electroless deposition all specimens underwent a double zincating pre-treatment that included 5 min degreasing at 60 °C, 2 min etching, 1 min first desmutting, 30 s first zincating, 30 s second desmutting, and 15 s second zincating. The coating thickness was measured by optical microscopy and was found to be  $17 \pm 1 \, \mu m$ .

A part of the coated specimens was subsequently heat-treated at 190 °C for 1.5 h to release hydrogen entrapped in the coating during electroless nickel deposition. After the heat treatment, the specimens were slowly cooled down in the furnace to minimize induced internal stresses.

The chemical composition of electroless NiP coating was determined by X-ray fluorescence (XRF) using a Philips PW1480 equipment.

The phase composition of electroless NiP coatings has been studied by X-ray diffraction (XRD) technique. The measurements were performed on a Bruker-AXS type D8 Advance series 2 diffractometer, equipped with diffracted beam graphite monochromator, using Cu K $\alpha$  radiation. The diffractometer scans were performed in  $\theta$ –2 $\theta$  geometry with a range of 15–120°2 $\theta$ , a stepsize of 0.1°2 $\theta$  and a counting time of 2 s per step.

The fatigue tests were performed using a 100 kN servohydraulic uniaxial Schenk testing machine under the load control condition and with a test frequency of 25 Hz in ambient air. Fully reversible push-pull (stress ratio, R = -1) sinusoidal load cycles were used. The tests were carried out according to ASTM E4689-90. The specimens were cycled at constant amplitude until failure or until at least  $10^7$  load cycles were reached.

The fatigue mechanisms were studied by examining the fracture surfaces with scanning electron microscopy (SEM). The analysis was conducted on a JEOL JSM-6400F microscope.

The chemical and phase composition of the electroless NiP coating has been studied using X-ray analyses. The X-ray fluorescence analysis indicates that the average phosphorus content was 13.2 wt.%. The coating has a high phosphorus content and therefore its asdeposited structure is expected to be amorphous. The

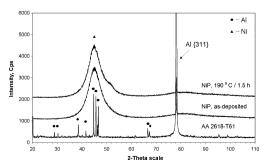
structure of the NiP coating was investigated by X-ray diffraction analysis (Fig. 1). A large diffraction peak at  $2\theta = 44.5^{\circ}$  followed by a second one at about  $2\theta = 81^{\circ}$  appears to be very similar to that reported for amorphous NiP coatings [10]. After annealing at 190 °C for 1 h the X-ray diffraction structure of NiP coating remained unchanged. The hardness of the coating (both in as-deposited and annealed conditions), measured with a Vickers diamond indenter under 100 g load, was about 550 HV<sub>0.1</sub> [10]. This is more than three times higher than that of substrate (160 HV<sub>0.1</sub>).

The maximum alternating stress as a function of number of cycles to failure (*S*–*N* curve) for the uncoated substrate, the substrate after double zincating pre-treatment, the substrate coated by electroless NiP in asdeposited and in heat-treated conditions is presented in Figure 2. From the Figure 2 it is followed that the double zincating pre-treatment decreases the fatigue life of the aluminium substrate. The magnitude of the reduction increases with the alternating stress decrease. For instance, at an alternating stress of 300 MPa the effect of the pre-treatment is almost negligible, whereas at 200 MPa the reduction in fatigue life reaches 75%.

Moreover, it can be observed from the Figure 2 that high-phosphorus electroless nickel deposit onto 2618-T61 substrate gives rise to the increase of the fatigue life of the substrate in relation to the uncoated aluminium alloy. The magnitude of the improvement depends on the applied alternating stress. Thus, at alternating stress >300 MPa the curves tend to converge. At alternating stress of 250 MPa the increase in fatigue life reaches about 100%, whereas at 200 MPa it reaches approximately 150%. At alternating stress <200 MPa the magnitude of the increase of fatigue life tends to decrease.

The hydrogen release heat treatment at 190 °C for 1.5 h did not change the fatigue behaviour of the electroless NiP coated samples, indicating improvement over the uncoated alloy.

Figures 3 and 4 represent the SEM micrographs of the fracture surface of the uncoated samples. Subsurface fatigue crack initiation sites, located at different depths ranging from near zero up to 4 mm were detected. Figure 3 shows the fracture surface of the sample tested at 250 MPa and fractured after 10<sup>5</sup> loading cycles. The fracture process started close to the specimen surface and was dominated by the propagation of a single crack. The radial lines on the fracture surface indicate the origin of the fatigue crack, pointed out by the arrow in the



**Figure 1.** X-ray diffraction patterns of the substrate and electroless NiP deposit in as-deposited state and after heat treatment at 190 °C for 1.5 h.

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