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The effect of excimer laser surface treatment on the pitting corrosion fatigue behaviour of aluminium alloy 7075

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An excimer laser (KrF) operating at a wavelength of 248 nm was used to modify the surface microstructure of 7075-T651 aluminium alloy. The aim was to improve both the corrosion resistance and the pitting corrosion fatigue resistance of the alloy by means of laser surface melting (LSM). The microstructure and the phases of the modified surface structure were analysed, and the corrosion behaviour of the untreated and the laser-treated specimens were evaluated by immersion test. The fatigue resistance of the 7075 alloy has been presented in the form of S/N curves.

A microscopical examination and the transmission electron microscopy (TEM) study revealed that LSM caused a reduction both in number and size of constituent particles and a refinement of the grain structure within the laser melted zone. As a result, the corrosion resistance of the aluminium alloy was improved. There was a significant reduction in the number of corrosion pits and shallow attack occurred. The fatigue test results showed that under dry fatigue conditions, the total fatigue life of the laser treated specimens, in which the crack initiation period is of considerable significance, was lower than that of the untreated specimens. However, after shot peening, the fatigue life of the laser treated specimens was recovered. This was primarily attributed to the elimination of surface defects, but also be in part, due to the introduction of compressive residual stresses in the surface layer of the specimen. The fatigue resistance of the shot peened laser-treated specimens, tested in 3.5 wt% NaCl solution with 48 hrs prior immersion, was greater than the untreated specimens with an increase of two orders of magnitude in fatigue life. This was primarily due to the elimination of surface defects and the reduction of corrosion pits.

1. Introduction

The high strength aluminium alloy (HSAL) AA7075, based on the Al—Zn—Mg—Cu system, has been widely used for aircraft construction for the past forty years, but unfortunately, the alloy is susceptible to pitting corrosion, intergranular corrosion cracking (IGC) and stress corrosion cracking (SCC) [1, 2]. These problems have persisted for over half a century. Although advances in heat treatment, surface treatment and alloy development have overcome some of the major corrosion problems with such HSAL [3, 4], this has not been without penalties, some quite heavy. For example, cladding a HSAL with an appropriate anodically behaving alloy of aluminium or even pure aluminium itself can increase SCC initiation resistance; however, cladding causes a significant reduction in fatigue strength. One of the non-traditional surface engineering techniques, namely laser surface melting (LSM) has attracted growing interest in recent years for the improvement of the corrosion performance of aluminium alloys. LSM is a versatile and promising technique which could be used to modify the surface properties of a material without affecting its bulk properties.

It is well known that corrosion pitting in HSAL, like the 7075 alloy is usually associated with the constituent particles, commonly referred to 'insolubles'. And such pitting could have a marked effect on the fatigue properties of HSAL. Work by Sankaran [5] showed that corrosion pitting significantly reduces the fatigue lives of the 7075-T6 alloy. Pao [6] has further pointed out that within a notch-root surface where there were no pre-existing corrosion pits, fatigue cracks generally formed at large constituent particles. Furthermore, the work of Ludtka [7] unequivocally demonstrated that with such an alloy in the T6 condition, intergranular fracture initiated at grain boundary Cr-containing

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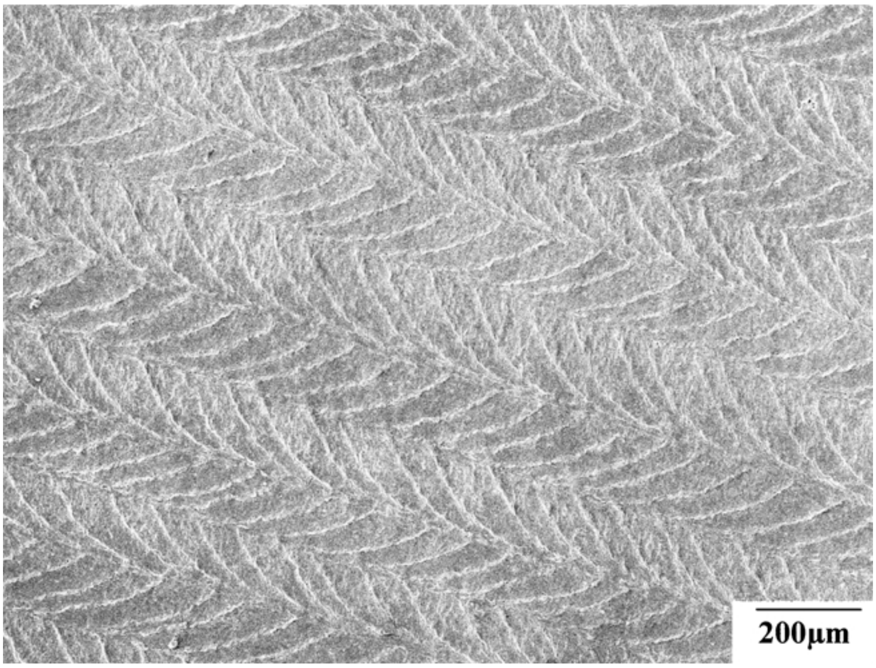


Figure 1 Surface morphology of the laser treated specimen.

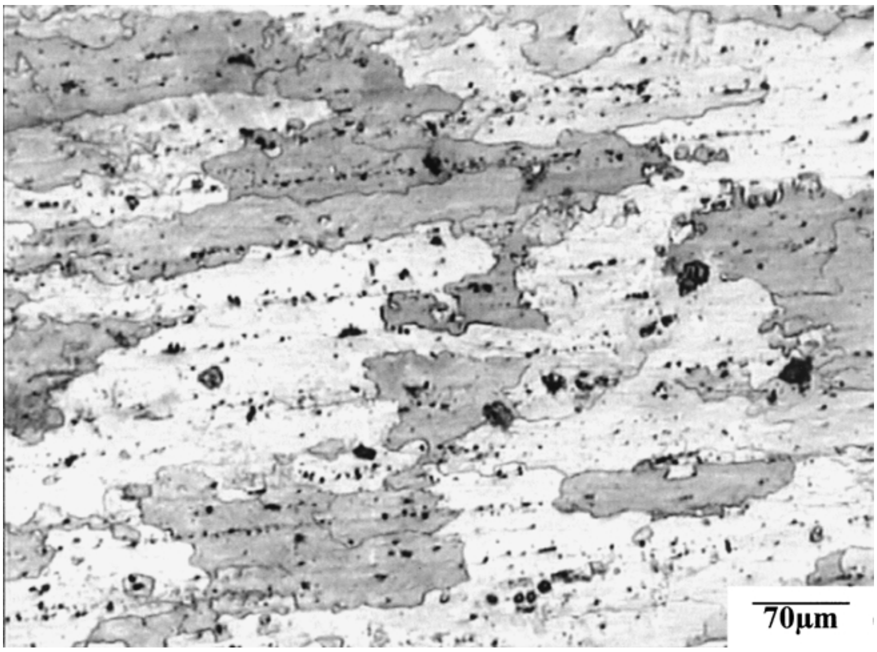


Figure 2 Microstructure of the untreated 7075 alloy.

dispersoids. Most recently, the work of Forsyth [8] on the study of corrosion attack of machined surfaces of the 7010 alloy has shown that the 'retained ingotism' from the cast state has a significant effect on corrosion behaviour of the alloy. He has shown that tested in seawater, corrosion attack was concentrated in the interdendritic regions when incompletely homogenised structures were present. The potential for using lasers to improve corrosion resistance of metals lies in the virtue of the extremely high cooling rate of the surface of the metal when subjected to laser surface treatment: micro-crystalline structures with homogenised composition occur and this would be expected to improve both corrosion and pitting corrosion fatigue resistance of HSAL.

So far, most of the corrosion studies of laser treated aluminium alloys have concentrated on the general and pitting corrosion behaviour [9–12], and mostly CO_2 lasers were employed. The results of similar studies of the corrosion fatigue behaviour of these materials have not been reported. The few investigations of the general corrosion behaviour of laser treated HSAL have revealed that a modest improvement in corrosion properties was obtained after LSM. It is anticipated that excimer laser surface treatment should give an even more pronounced improvement because the short pulse duration of the UV radiation, in the range of nanoseconds, would result in extremely fast cooling rates and thus even more homogenised surface structures would form. In fact, the cooling rates in excimer laser processing (in

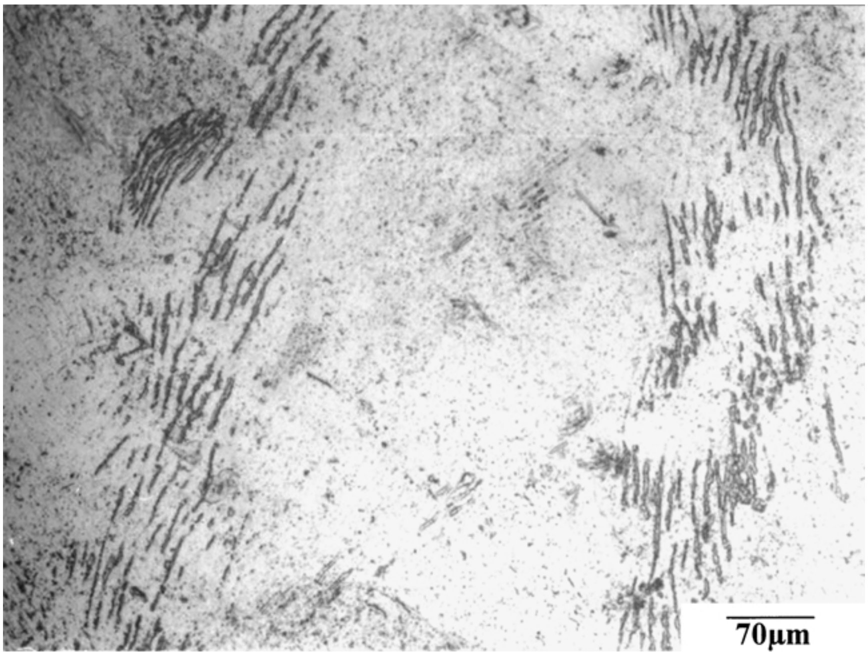


Figure 3 Microstructure of the laser treated surface.

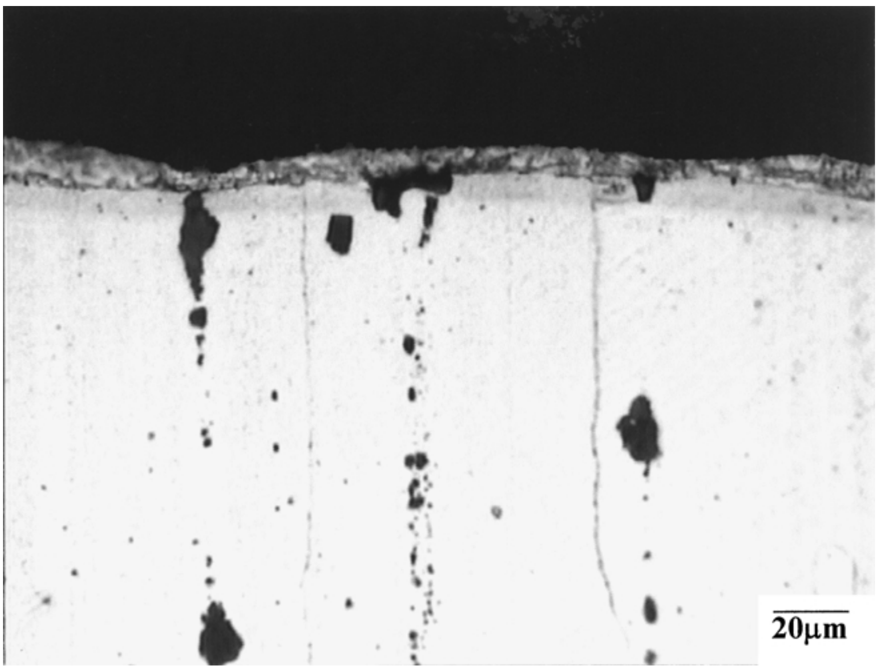


Figure 4 A microsection showing stringers of particles terminated at the fusion boundary.

the order of 10^9°C/s) are somewhat two orders of magnitude higher than that for either CO_2 or YAG laser treatment. Also, the characteristic of limited heat diffusion of excimer laser processing has virtually eliminated any heat affected zone. Based on our previous work on laser surface treatment of aluminium alloys and their composites [13, 14], the aim of the present study is to investigate the effects of LSM on the corrosion fatigue initiation resistance of HSAL. The study, however, does not intend to address the fundamental problem of corrosion fatigue in its classical sense, but focuses on the problem of pitting corrosion induced fatigue.

2. Experimental method

The material used for this study was aluminium alloy 7075-T651 supplied by Alcoa in the form of 19.6 mm thick plates. Specimens of the alloy in the form of discs of 16 mm diameter and 3 mm thickness were machined from the plate for laser treatment. The specimens were prepared by cutting along the short-transverse and longitudinal directions to provide L-ST faces for testing. Prior to laser treatment, all the specimens were finely ground and finished on 2400-grit emery paper, then cleaned with distilled water and alcohol.

Laser surface treatment was conducted using a Lambda Physik excimer laser LPX315i with the laser

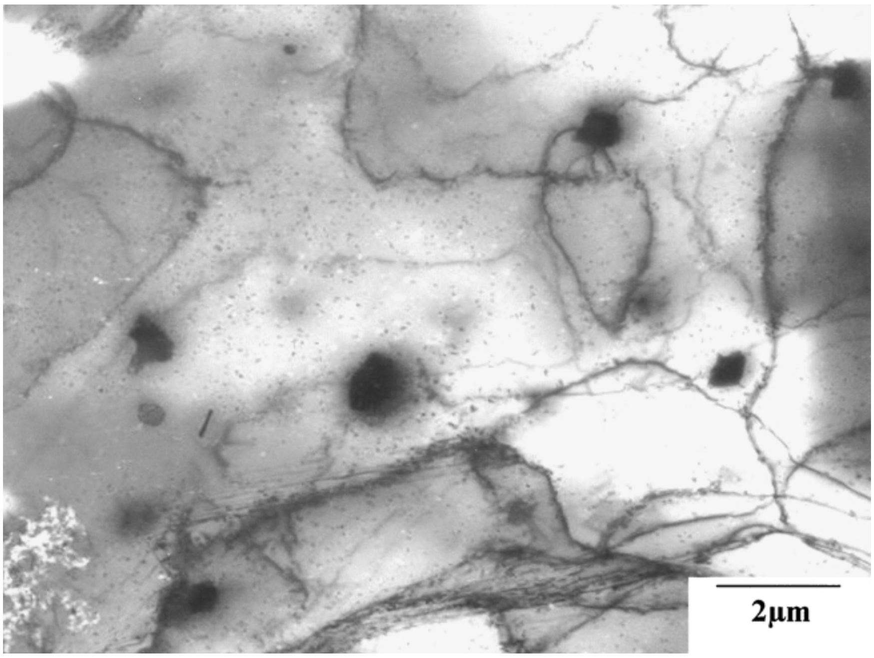


Figure 5 TEM micrograph of the untreated material showing Al—Cu—Fe—Zn particles.

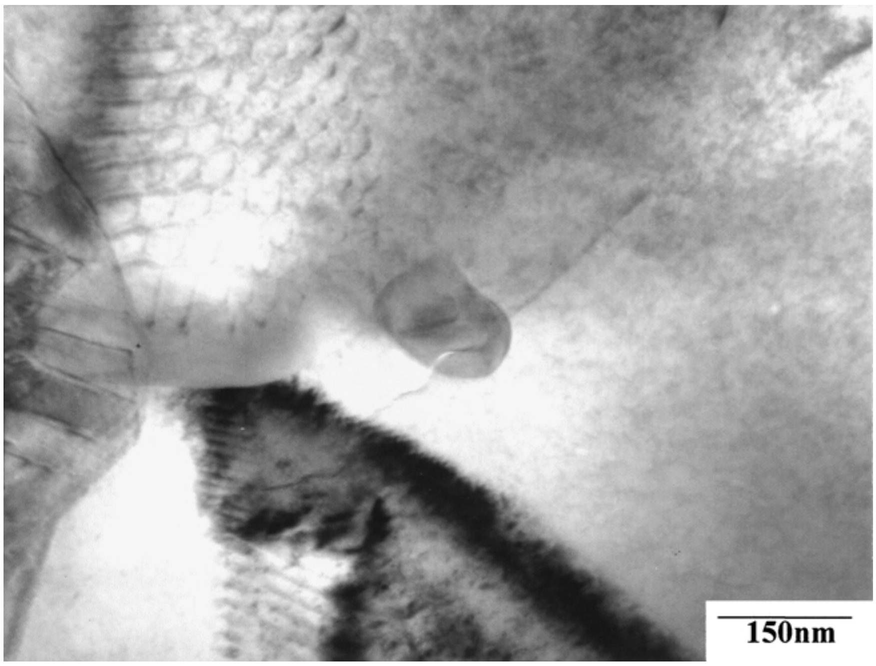


Figure 6 TEM micrograph of a laser treated specimen showing a particle free matrix.

operated at a wavelength of 248 nm (laser active medium KrF) and pulse duration of 25 ns. The beam delivery system was so designed that a circular aperture was irradiated by a homogeneous laser beam from the rear and imaged down to a 0.6 mm spot size on the surface of the specimen. Laser surface treatment was performed in air. The laser pulsing frequency was fixed at 10 Hz, and the scanning speed was set at 2 mm/sec. The laser pulse energy was varied from 3.3 J cm^{-2} to 15.4 J cm^{-2}. The laser energy was measured by using an energy meter at a position below the final focusing lens. A 50% track overlapping condition was employed.

Intergranular corrosion immersion testing according to ASTM G110 was used to evaluate the corrosion resistance of the laser treated and untreated specimens. Tests were performed in $NaCl/H_2O_2$ solution which has been prepared using analytical grade reagents: 57 gm NaCl and 10 ml H_2O_2 (30%) per liter of distilled water.

The effects of laser surface modification on the pitting corrosion fatigue behaviour of the Al-alloy were studied using a rotating cantilever Wöhler type fatigue machine. Total fatigue life, in which the crack initiation period is of considerable significance was determined on plain fatigue specimens under reversed loading cycles, i.e. the stress ratio $R = -1$ [15]. A corrosion attachment that allows a constant circulation of corrosion solution around the fatigue specimen is fitted to the fatigue machine. As for the untreated specimens, they

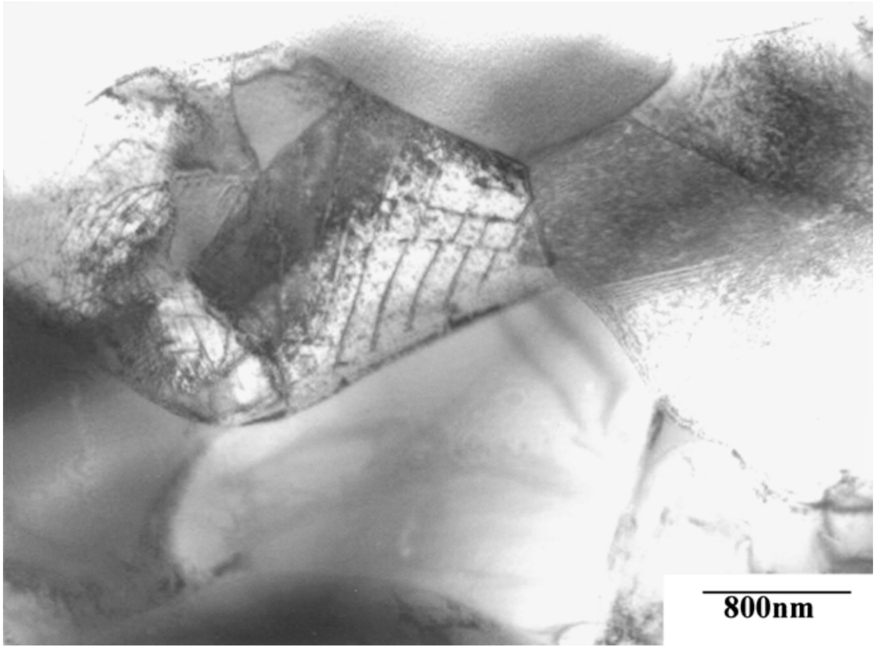


Figure 7 TEM micrograph of a laser treated specimen showing a refined grain structure.

were ground in the lengthwise direction with 2400-grit emery papers so as to remove circumferencial machine marks. Laser treatment on the fatigue specimens was carried out using pulse energy density of 9.3 J cm^{-2}. A thermal analysis showed that using this energy density, a surface layer of about 0.3 μm would be vaporised. The specimens were rotated in steps, and the laser scan was performed in the specimen's longitudinal direction. The fatigue test was carried out under two conditions: (i) dry fatigue, and (ii) fatigue in 3.5 wt% NaCl solution with prior immersion (specimens were soaked in NaCl/H_2O_2 solution for 48 h before the fatigue test).

3. Results and discussion

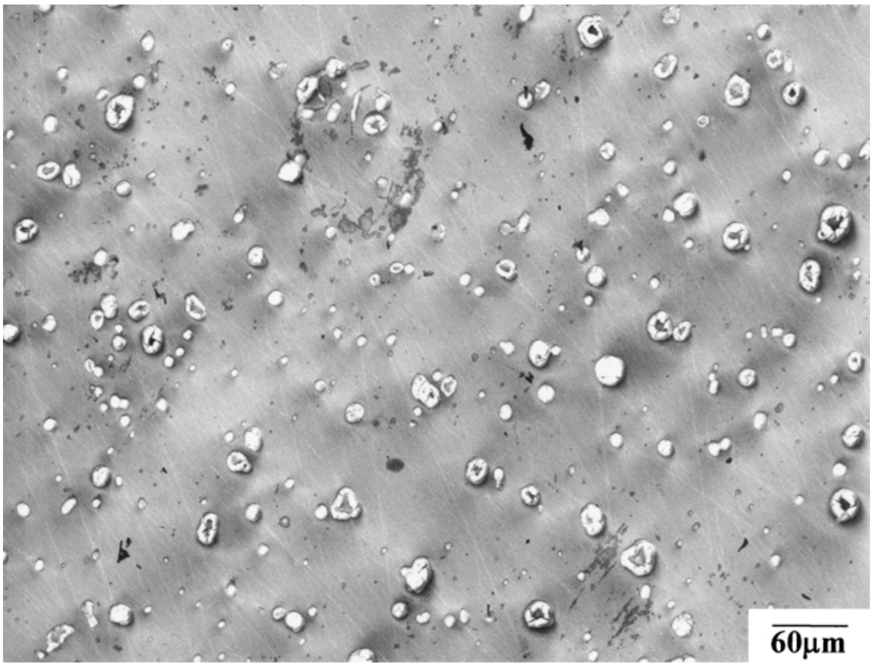
3.1. Microstructural changes

All the laser treated specimens were found to exhibit a wavy topography in the form of surface ripples (Fig. 1). To reveal the changes of microstructure after laser treatment, specimens were lightly polished back using 1 μm diamond paste, then etched with Keller's reagent. Figs 2 and 3 show the microstructure of the alloy before and after the laser treatment. It is apparent that most of the large out-of-solution particles that were present in the untreated material have been removed. Because only light polishing was applied, some wavy uneven surface marks still remained on the surface. It is anticipated that the pitting corrosion resistance of the alloy would be improved after laser treatment due to the elimination of large constituent particles.

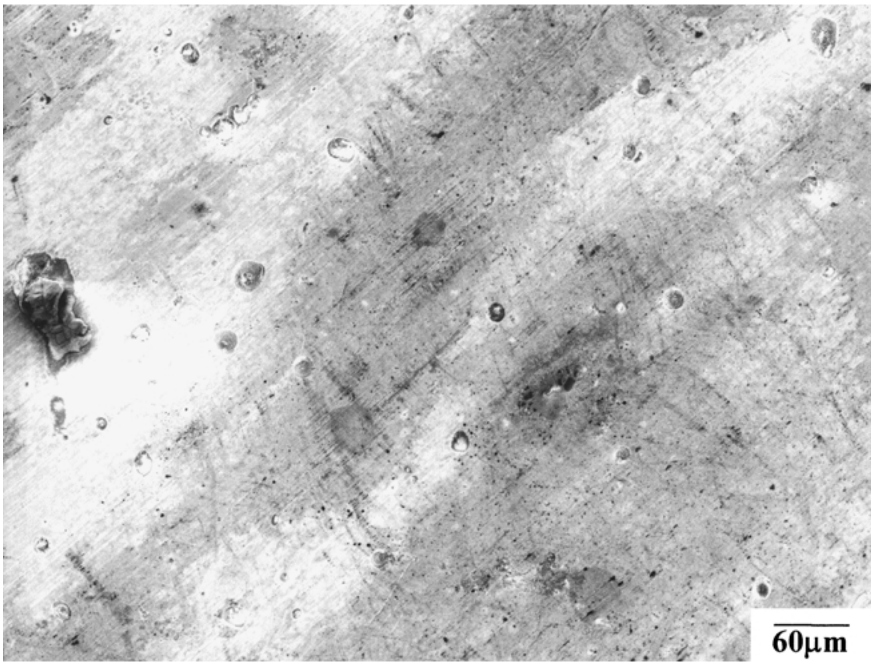
A transverse section (Fig. 4) of the laser treated specimen shows a re-solidified layer of about 8 μm thick had been formed at the surface, where stringers of particles were terminated at the laser-melted boundary. This figure also shows that a distinct boundary was obtained between the fusion layer and the parent material, and no pronounced heat affected zone (HAZ) was observed. This was believed to be due to the short pulse length of the excimer laser, which minimises thermal diffusion, also the absorption depth of UV photons in metals is relatively shallow. The TEM study of the laser treated specimens showed that after laser treatment most Al—Cu—Fe—Zn particles in the matrix were eliminated (cf. Figs 5 and 6). They were likely taken into solution during laser melting and stayed in solution during resolidification. This was accomplished due to the extremely fast cooling rate of excimer laser processing, which has been calculated to be in excess of 10^{9}°C/s. Also, grain reinforcement was observed in some regions after excimer laser treatment (Fig. 7).

3.2. Immersion test

To reveal the degree of attack, all the immersion tested specimens were lightly polished by using 1 μm diamond paste. The corroded surfaces and the cross sections of the immersion tested 7075 specimens are shown in Figs 8 and 9 respectively. Fig. 8 clearly shows that the untreated specimen (Fig. 8a) was found to exhibit serious pitting corrosion, while few pits were observed in the laser treated specimens (Fig. 8b). In the untreated specimen, the results of EDX confirmed that corrosion pits were initiated at the cathodic Al—Cu—Fe—Zn particles (Fig. 10) as well as the anodic Al—Mg—Zn particles. The former tends to promote the surrounding matrix dissolution whilst the latter is itself attacked. To reveal the depth of pits, the immersed specimens were cross-sectioned and examined. It is clear that corrosion attack of the untreated specimen had advanced along the stringers of second phase particles to a depth of about 250 μm (Fig. 9a). The active corrosion path was formed as a result of the dissolution of anodic particles themselves and also the aluminium matrix. In contrast, only relatively shallow corrosion pits were found in the laser treated specimen (Fig. 9b). This must be due to the elimination of large second phase particles



(a)



(b)

Figure 8 Corroded surfaces of the immersion tested specimens: (a) untreated and (b) laser treated at 15.4 J cm^{-2}.

in the laser remelted layer and the stopping of second phase stringers emerging at the surface of the specimen. The elimination of second phase particles at the surface also allows a continuous passivation film to form thus providing better overall corrosion resistance. In fact, a similar effect of sealing second phase particles against the corrodent also happens in mechanically machined surface of high strength aluminium alloy 7010 [16] where the flow zone envelops the emergent particles that would otherwise corrode. However, because the flow zone produced by machining is itself reactive, corrosion attack through it down to the particles beneath is quickly achieved. Thus in this case, the benefit of submerging the particles is counteracted by this reactivity and the overall effect is that there is no particular advantage or improved corrosion resistance. However, in the present case with laser surface treatment, the treated layer did not seem to be particular reactive, therefore the benefit obtained from eliminating the particles could be fully realised.

From the results of the immersion test, it is obvious that the initial corrosion attack in the 7075-T6 alloy was primarily by pitting. The general improvement in corrosion resistance obtained for the laser treated 7075 specimens is therefore considered to be primarily due to the elimination of harmful second phase particles, those being the primary sources for the nucleation of corrosion pits. After laser treatment, most of the anodic and cathodic types of particles were removed from the surface of the matrix material; therefore the degree

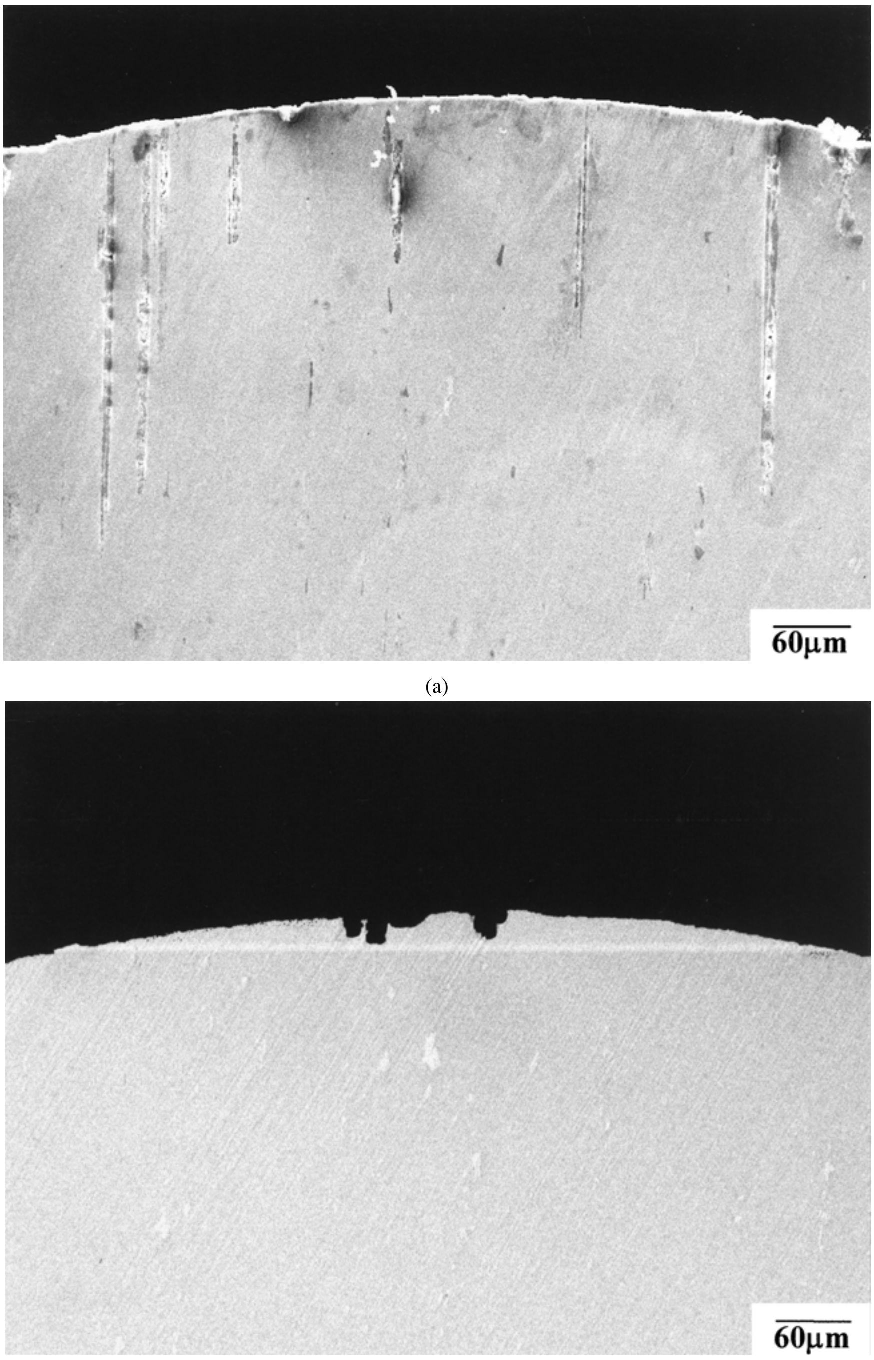


Figure 9 Microsections of the immersion tested specimens: (a) untreated and (b) laser treated at 15.4 J cm^{-2}.

of preferential corrosion attack on the matrix and particle dissolution itself was significantly reduced. The absence of Cu-bearing particles in the surface of the laser treated specimens also leads to a reduction in corrosion current because it is well known that they are active sites for galvanic corrosion [17]. Apart from this, laser surface treatment provided a homogenised surface layer and therefore compositional gradients between the matrix and grain boundaries were reduced. Such a homogenised surface structure could be advantageous where subsequent anodising is required. Since the presence of 'insolubles' causes a reduction in the quality of sulphuric acid anodise coatings, in the worse cases providing damaging defects that cannot be sealed.

3.3. Dry fatigue behaviour

The results of the dry fatigue test of the untreated and the laser treated specimens are presented in Fig. 11. A comparison of the fatigue strength of the untreated and laser treated specimens with shot peening was made at a stress level of about 280 MPa. The results showed that the fatigue life of the alloy was significantly reduced after laser treatment, a reduction of two orders of magnitude in the number of cycles to failure being recorded. Low glancing angle XRD was employed to determine whether laser treatment had caused the presence of tensile residual stresses at the surface of the specimen. Any such stresses appear in the surface layer would certainly reduce the fatigue strength of the specimen.

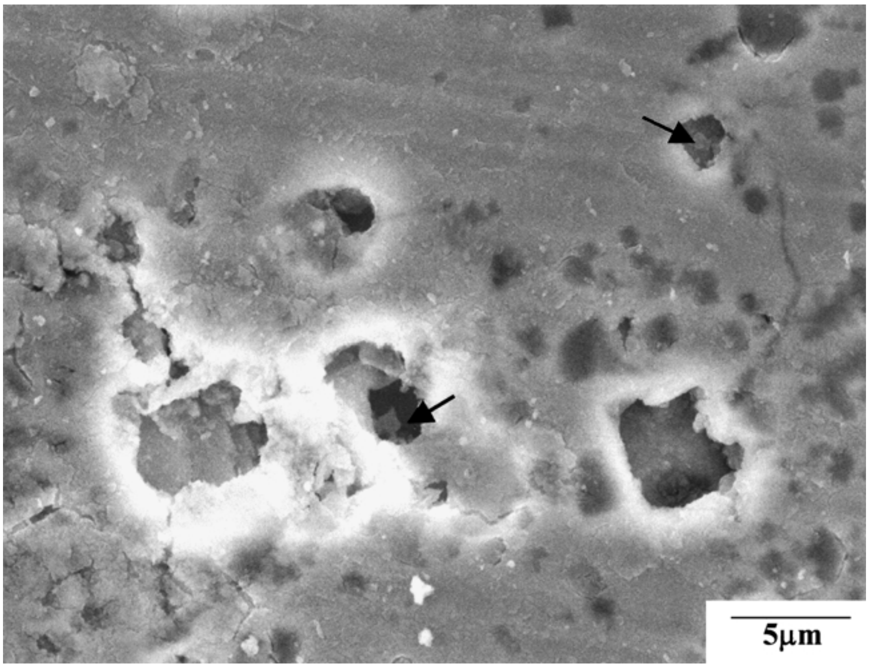


Figure 10 Corroded 7075 surface showing Al—Cu—Fe—Zn particles.

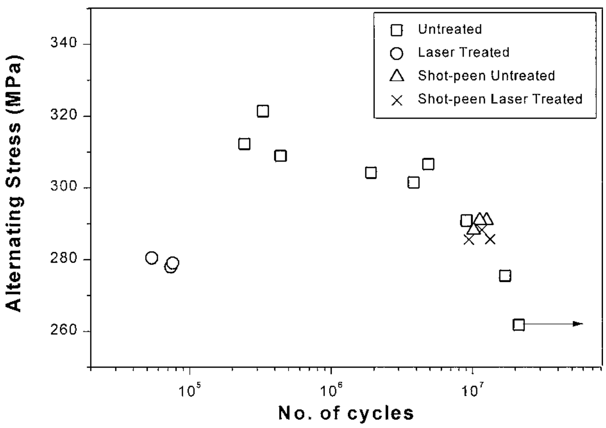


Figure 11 Dry fatigue S/N curves for the untreated and laser treated specimens.

The results showed that the stress at the surface was virtually unchanged after laser treatment. On the other hand, it might be expected that the grain refinement that resulted from laser treatment would improve the fatigue initiation resistance compared with that of the untreated material. The fact that fatigue crack initiation is more difficult in fine-grain material is well established and is in keeping with our presently accepted understanding of grain size effects on plastic deformation and would fit in with a Hall-Petch approach to flow strength. The reverse result found in this study was probably due to the presence of surface defects in laser treated specimens and there is no doubt that fatigue crack initiation resistance, as represented by the uncracked test life under the conventional S/N type testing, is strongly influenced by the presence of defects. Where crack initiation at defects occurs, then direct Stage II growth ensues with no evidence of a Stage I period. Mazal [18] reported that the fatigue initiation resistance of a grey cast iron was much reduced after CO_2 laser treatment. He concluded that this was due to the presence of an inhomogeneous surface caused by laser tracks overlapping, in this case overlaps could be acting as fatigue initiation sites. In the present study, although no obvious surface cracks or defects were detected, a close examination of the laser treated surface revealed that the ripples formed in the surface resembled a series of fold lines (Fig. 12). It is considered that these lines could act as fatigue initiation sites and cause early crack initiation. In addition, a small amount of minute gas porosity (Fig. 13) was found within the laser treated zone. It is possible that, due to the inherent extremely fast cooling rate of excimer laser processing, that gas would have insufficient time to escape and would be trapped within the melt zone. Skallerud [19] and Couper [20] have suggested that almost all the pores lead to early crack initiation are located close to surface of the material. Buffière [21] showed that as small as 1% volume fraction of porosity can lead to a reduction of 50% of the fatigue life and 20% of the endurance limit in cast aluminium alloys. The fatigued surface of the laser treated specimens was examined, and it was found that many microcracks were present, and it is apparent that some of these cracks were initiated at those ripple features (Fig. 14). It is therefore believed that the inferior fatigue properties of the laser treated specimens were primarily due to poor fatigue initiation resistance.

Shot peening was applied to both the laser treated and the untreated fatigue specimens. The process was performed using aluminium oxide particles which have a mesh size of 100. A pressure of 6 bars and a standing distance of 450 mm were used. The aim was to test whether shot peening could restore the fatigue strength of the laser treated material by virtue of its ability to close up any near surface defects and at the same time induce compressive stresses in the surface. The fatigue results (Fig. 11) showed that after shot peening, the dry fatigue lives of the laser treated specimens were recovered and were practically equal to those of the untreated

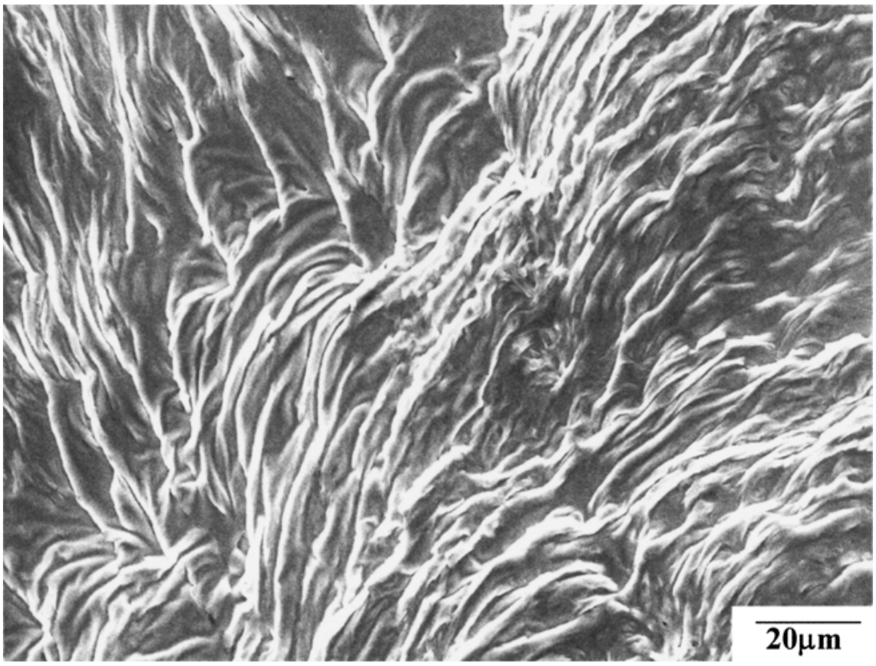


Figure 12 Ripple features in laser treated surface.

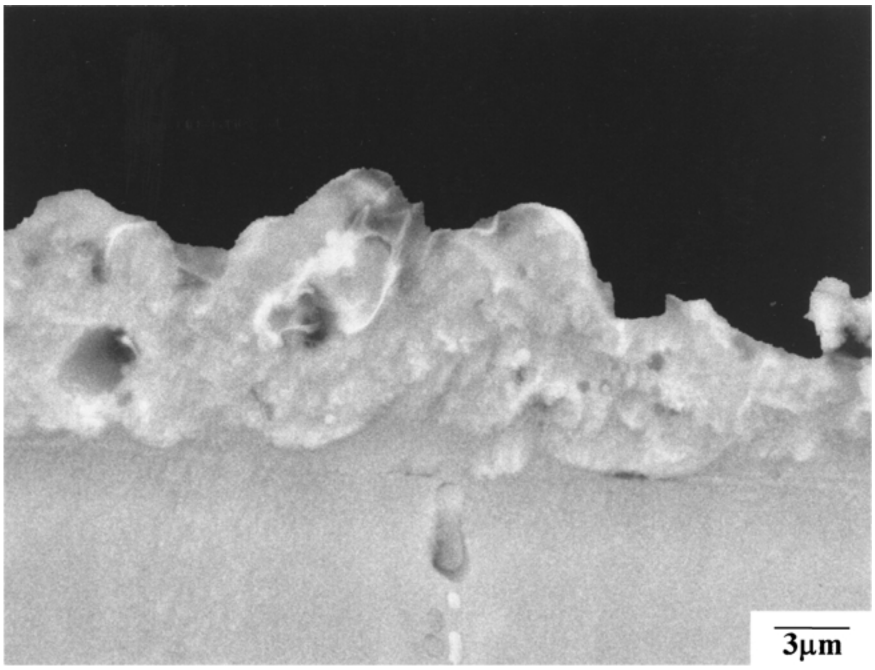


Figure 13 Porosity within the laser treated zone.

specimens with shot peening. A microsection of a laser treated specimen with shot peening revealed that porosity in the melt zone has been eliminated (Fig. 15). In addition, the regular ripple features have been removed (cf. Figs 1 and 16) after shot peening.

However, the fatigue results also showed that the fatigue strength of the untreated specimens was not increased after shot peening. Eftekhari [22] and Filho [23] have shown that the effect of shot peening on the fatigue lives of aluminium alloys and aluminium composite was insignificant. The study of Shaw [24] on the fatigue properties of 7075 found that if the magnitude of deformation resulted from shot peening is not large enough, no improvement on fatigue crack resistance was obtained. This suggests that in the present study, the amount of work done on the untreated specimens by shot peening was insufficient to create a high degree of work hardening in the surface. This was confirmed by the results of the nano-hardness tests of the untreated specimens, where the hardness values measured before and after shot peening were similar, which was about 2.8 GPa for both cases. In fact, the results were not unexpected, since for Duralumin in the fully aged condition work hardening effects were found to be small. Now, for the laser treated specimens, the modified layer is in a solutionised condition which was evident from the results of the TEM work where a precipitate free matrix was found (Fig. 6). This was also in agreement

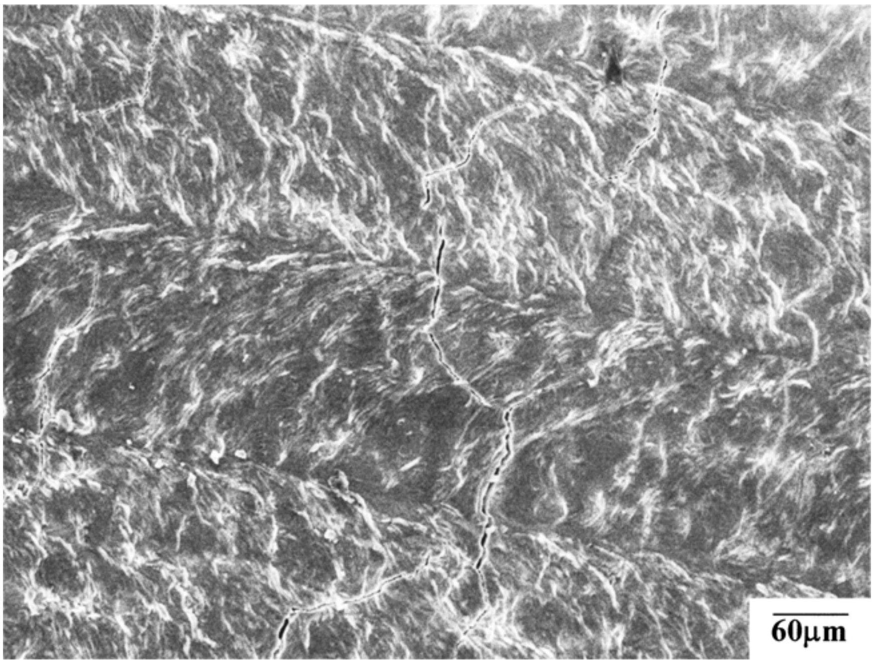


Figure 14 Microcracks in the surface of a laser treated specimen after being fatigued.

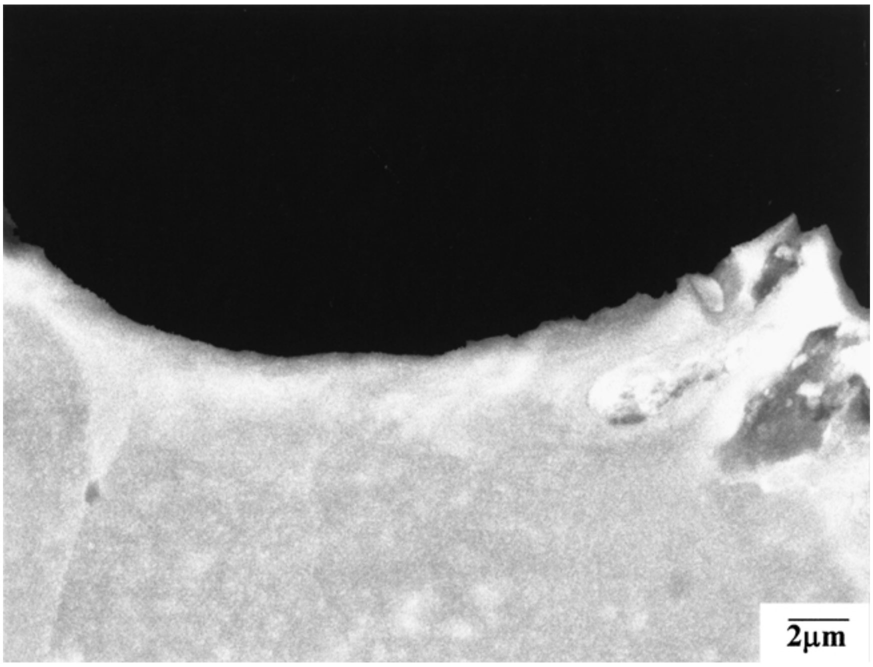


Figure 15 A microsection of a laser treated specimen after shot peening.

with the results of the nano-hardness tests: after laser treatment the hardness at the surface was reduced to about 1.7 GPa. But shot peening of the specimen did not show any significant changes in the hardness value. The results of low glancing angle XRD showed that compressive residual stresses were introduced in the surface layer after shot peening for both the untreated and laser treated specimens. Based on these results, it is therefore considered that the beneficial effects of shot peening on laser treated specimens are twofold: one is the elimination of surface defects, such as micro-porosity and fold lines; the other is the modification of residual surface stresses. However, the former may play a more significant role, since it had been found that residual surface stresses fade more quickly in aluminium alloys than in steels with the application of cyclic strain. As for the untreated specimens, despite the fact that compressive stresses were introduced in the surface layer, the fatigue strength was not increased by shot peening. This was very likely due to the roughening of the surface by shot peening; the surface roughness, in terms of R_a, measured by a form talysurf laser interferometer before and after shot peening were found to be 0.04 μm and 2.3 μm, respectively.

3.4. Pitting corrosion fatigue behaviour

The results of corrosion fatigue testing are presented in Fig. 17. To make a comparison of the pitting corrosion fatigue resistance of the laser treated and the

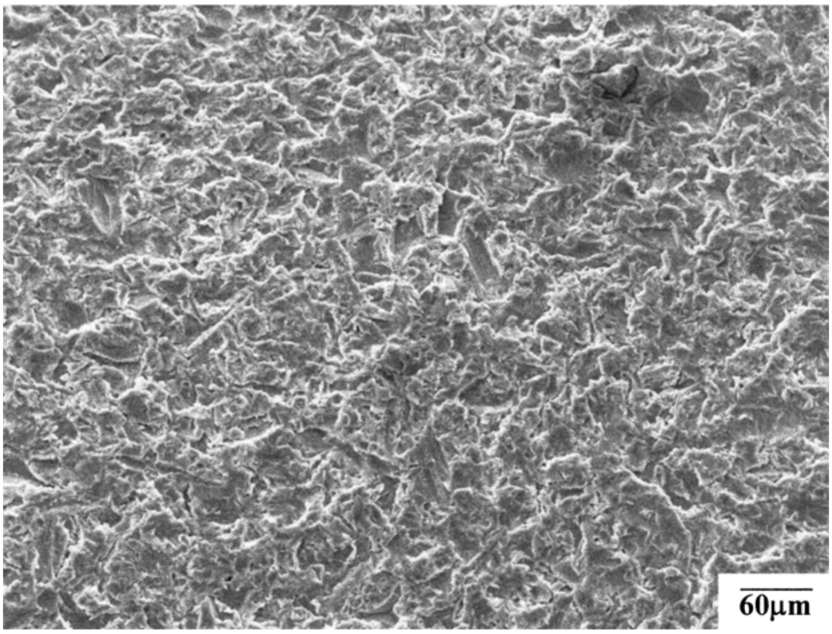


Figure 16 Surface morphology of a laser treated specimen after shot peening.

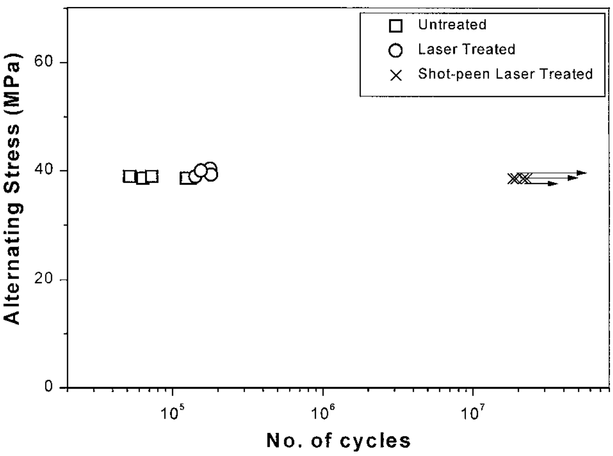


Figure 17 Corrosion fatigue S/N curves of the untreated and the laser treated specimens.

untreated materials, all the pre-corroded specimens were tested at a stress level of 40 MPa. The results indicated that the pitting corrosion fatigue resistance of the laser treated specimens was slightly better than the untreated specimens. This shows that despite the presence of microporosity in the melted layer, the lives of the laser treated specimens were some 30% to 50% longer than those of the untreated specimens. One of the reasons must be due to the greater pitting resistance of the laser treated material as shown in the immersion tests. Unlike the laser treated specimens, numerous corrosion pits were found in the surface of the untreated fatigue specimens (cf. Figs 18 and 19), and there is no doubt that these defects would act as fatigue initiation sites and cause early fatigue crack growth. Such an effect had caused a large difference in fatigue strength between the dry fatigue and the pre-corroded

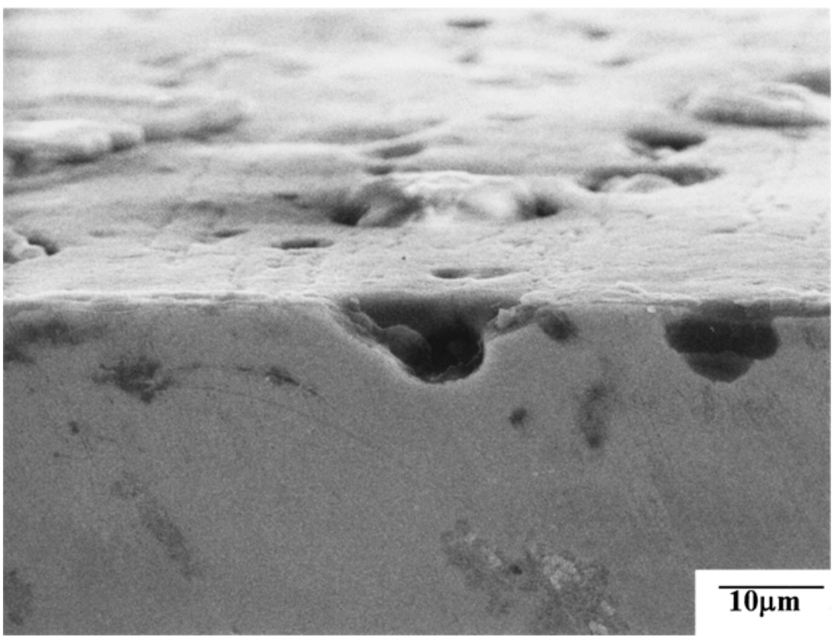


Figure 18 An untreated fatigue specimen showing corrosion pits present in the surface.

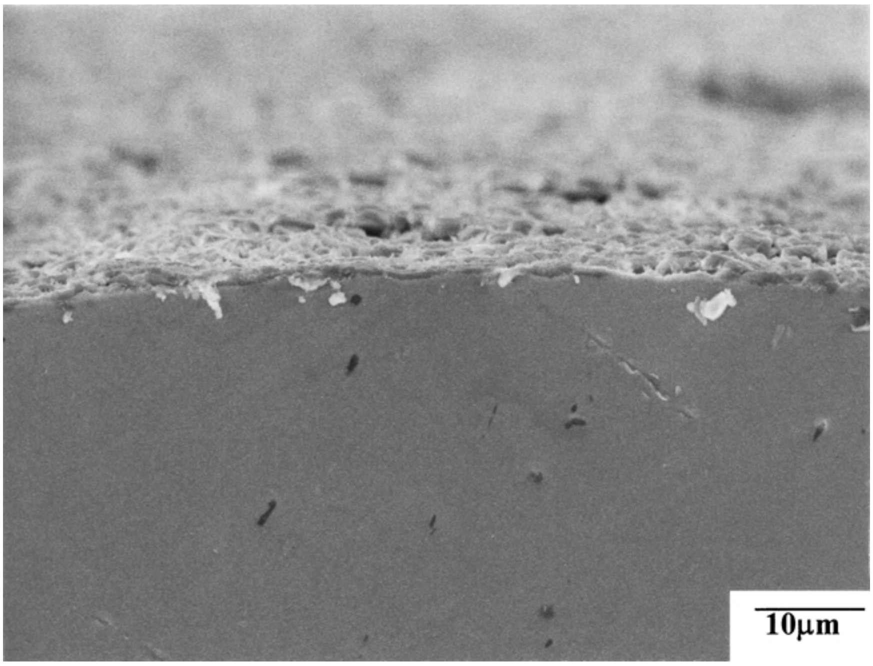


Figure 19 A laser treated specimen showing corrosion pits were largely absent from the surface.

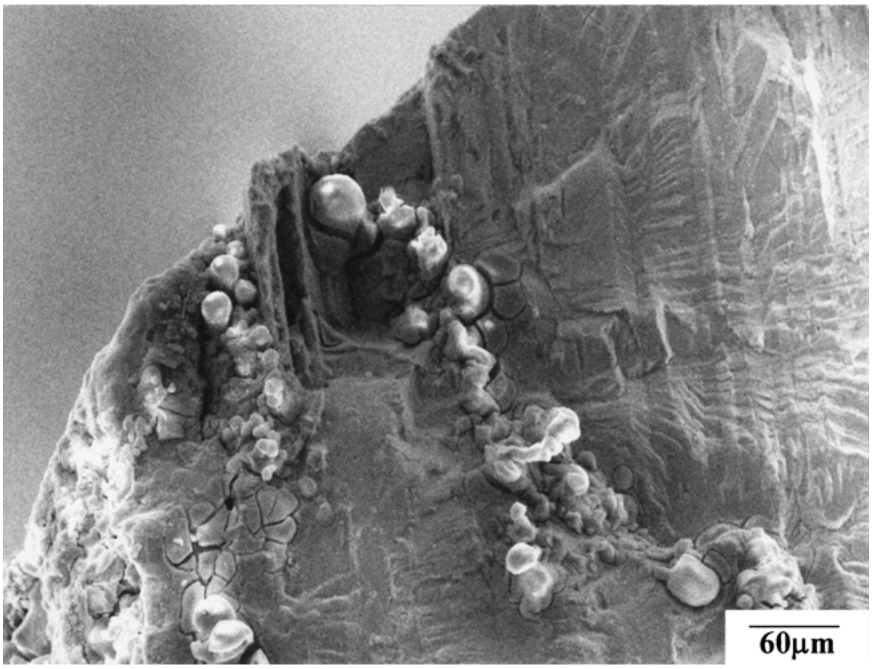


Figure 20 Fracture surface of an untreated specimen, showing initiation site was associated with corrosion pits.

fatigue conditions; a reduction in fatigue strength of about 8 times was recorded for the latter. In fact, a similar result was reported by Dolley [25] that a reduction in fatigue life of more than one order of magnitude was obtained for the pre-corroded specimens of aluminium alloy 2024. An examination of the fracture surface of an untreated specimen showed that pits and corrosion products were present at the fatigue crack origin (Fig. 20). Although very frequently, some relatively long planar corrosion cracks (Fig. 21) were found in the fracture surface, they did not seem to develop into main fatigue cracks. It is apparent that these planar cracks are lying perpendicular to the main fatigue crack propagation plane, where they are operating in the so-called crack stopping mode [26]. However, the situation might have been totally different if the specimens had been tested in the short-transverse direction.

A significant improvement in fatigue life was obtained after the laser treated specimens were shot peened. An increase of two orders of magnitude in the number of cycles to failure was obtained as compared to untreated specimens. The substantially improvement in fatigue strength obtained after shot peening is believed to be due to the elimination of surface defects as well as to the induced compressive residual stresses.

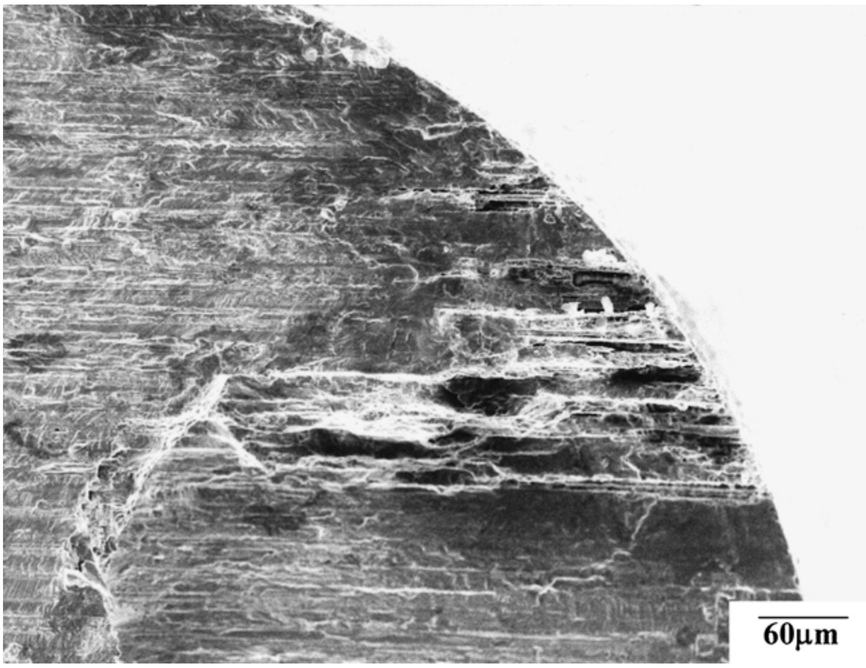


Figure 21 Fracture surface of an untreated specimen showing planar fatigue corrosion cracks.

4. Conclusions

(i) Excimer laser surface treatment has significantly improved the corrosion resistance of 7075 alloy when subjected to an immersion test in NaCl solution. In general, the improvement in corrosion pitting resistance of the alloy is considered to be due to the reduction of constituent particles in the laser modified layer as well as the chemical homogenisation of the matrix material.

(ii) Excimer laser surface treatment has significantly reduced the fatigue strength of the alloy when tested in air due to laser induced defects, however, the lost fatigue life was recovered after shot peening.

(iii) The corrosion fatigue lives of the pre-corroded laser treated specimens were some 30%–50% greater than that of the untreated specimens. This was mainly attributed to the better corrosion pitting resistance of the laser treated material.

(iv) An even greater improvement in corrosion fatigue life was achieved when the laser treated specimens were shot peened, the increase being two orders of magnitude. Shot peening eliminated surface defects in the laser treated specimens and also introduced compressive residual stresses in the surface layer of the specimen.

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