Fatigue behaviour and fracture mechanism of a 316 stainless steel hardened by carburizing

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Abstract

Fatigue behaviour and fracture mechanism of a carburized 316 stainless steel were studied under rotary bending in laboratory air and in 3\% NaCl aqueous solution using materials with two different case depths. In laboratory air, the carburized materials showed considerably higher fatigue strength compared with the untreated material, and the fatigue lives of the material with thick case were longer than those of the material with thin case, but the fatigue limits were the same, which increased by 30\% with respect to the fatigue limit of the untreated material. In 3\% NaCl aqueous solution, the carburized materials exhibited no reduction in fatigue strength, indicating the excellent corrosion resistance of the carburized case. Regardless of environment and stress level, fatigue cracks always initiated underneath the carburized case at a very early stage of fatigue life and subsequently grew predominantly into the core material. Small crack growth behaviour was also similar in both environments, thus the fracture mechanism of the carburized materials was unaffected by corrosive environment.

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1. Introduction

In recent years, it has been strongly required to extend the service life of machine components and structures due to economic and environmental reasons. To meet this demand, various surface engineering techniques have become major interest because they can provide additional surface properties such as high strength, thermal barrier, corrosion and wear resistance to structural materials.

Austenitic stainless steels have excellent corrosion resistance, but they possess relatively low strength and poor wear resistance. Therefore, it is very important to improve those properties by surface treatment and when surface-modified materials are applied to load bearing components, the evaluation of the fatigue properties is necessary. In austenitic stainless steels, the fatigue behaviour of materials treated by shot peening [1–5], laser [6], dynamic ion mixing [7] and coating [8] has been reported, but so many studies have not been done so far, because of very limited surface modification techniques that can apply to austenitic stainless steels without any loss of their advantages such as corrosion resistance and ductility.

A modified carburizing technique has been developed, which can improve significantly wear resistance and strength [9], but fatigue behaviour has not been studied. The purpose of the present study is to understand the fatigue behaviour of a type 316 austenitic stainless steel hardened by the modified carburizing. Rotary bending fatigue tests were conducted in laboratory air and in 3\% NaCl aqueous solution using materials with two different case depths. Fatigue strength was evaluated and fracture mechanism was discussed on the basis of crack initiation and small crack growth behaviour and fracture surface analysis.
2. Experimental details

2.1. Material and specimen

The material used in the present study is a 316 austenitic stainless steel of 16 mm diameter whose chemical composition (wt.%) is C 0.05, Si 0.35, Mn 1.35, P 0.033, S 0.025, Ni 10.1, Cr 16.9, Mo 2.11. The material was solution treated at 1353 K for 1 h followed by oil cooling. After solution treatment, fatigue specimens with a reduced section of 5.5 mm diameter were machined and then mechanically polished by emery paper. Subsequently, the following surface treatment was applied.

2.2. Surface modification technique

A modified gas-carburizing technique, which is called pionite treatment [9], was performed at a temperature below 773 K in a CO and H$_2$ gas mixture. During this process, carbon is diffused into the material and thus carbon-diffused zone is formed at the surface region without any Cr carbide formation where hardness is remarkably increased. It is believed that this treatment can improve wear resistance and strength without any loss of corrosion resistance, ductility and toughness of austenitic stainless steels.

In order to produce materials with two different case depths, the treatment times, $t_p$, of 15 and 35 h were employed. Hereafter, the materials treated for 15 and 35 h are denoted as the 15 h treated material and the 35 h treated material, respectively, and the material not subjected to the surface modification is referred to as the untreated material.

2.3. Procedures

Fatigue tests were carried out using cantilever-type rotary bending fatigue testing machines operating at a frequency of 19 Hz in laboratory air and in 3% NaCl aqueous solution. The solution was dropped continuously onto the specimen surface by a pump from a reserved tank. The temperature of the solution was not controlled.

Crack initiation and small crack growth were monitored with replication technique. Fatigue testing was periodically interrupted and replicas were taken, and then crack length was measured by laser microscope.

After experiment, fracture surfaces were examined in detail by scanning electron microscope (SEM).

3. Results

3.1. Microstructure characterization

SEM micrographs of the microstructures after etching are shown in Fig. 1. As well known, the untreated material has the microstructure consisted of austenitic grains (Fig. 1(a)). In the carburized materials, the surface region that is clearly distinguished from the core material can be recognized, which is the case formed by carburizing (Fig. 1(b),(c)). As can be seen in the figure, the carburized case depths are approximately 20 and 40 µm for the 15 and 35 h treated materials, respectively. It has been indicated that the microstructure underneath the carburized case is the same austenitic structure as the untreated material and the austenitic structure was maintained and no Cr carbides were formed in the carburized case [9].

3.2. Hardness profile

Vickers hardness profiles measured on the cross-section of the untreated and carburized materials are represented in Fig. 2, where the applied load was 9.8 N for the untreated material and 0.098 N for the carburized materials. It can be seen that hardness at or near the surface attains to more than approximately 800HV and 1000HV in the 15 and 35 h treated materials, respectively. Such a high hardness cannot be obtained by mechanical surface treatments such as shot peening that resulted in the maximum value of approximately 300HV [2–5].

Hardness rapidly decreases with increasing the distance from the surface and then reaches a constant value of approximately 172HV that is the hardness of the core material, i.e. the untreated material. The region of the increased hardness is approximately 40 µm regardless of treatment time. As can be seen in Fig. 1, however, the carburized case depth depended on treatment time, thus the carburized case depth, $d_p$, in

![Fig. 1. SEM micrographs showing microstructures: (a) untreated, (b) $t_p = 15$ h, (c) $t_p = 35$ h.](image-url)
the present study was defined as the depth established from the microstructure characterization.

3.3. Mechanical properties

The tensile properties are listed in Table 1. In the carburized materials, tensile strength increases and ductility decreases compared with the untreated material and with increasing treatment time. It can be seen that the differences in tensile property between the untreated and carburized materials are small, this being due to very thin case depths of 20–40 μm.

Residual stress at the surface was measured by the X-ray diffraction method under the conditions of the characteristic X-ray of CrKα, the tube voltage of 35 kV, the tube current of 200 mA and (220) plane reflection. Consequently, the compressive residual stresses of approximately 1450 and 1500 MPa were measured in the 15 and 35 h treated materials, respectively.

3.4. Fatigue behaviour in laboratory air

3.4.1. Fatigue strength

The S–N diagram in laboratory air is shown in Fig. 3. The fatigue strength of the carburized materials increases considerably compared with the untreated material. The dependence of fatigue strength on case depth can be seen in the finite life region, where the 35 h treated material exhibits slightly longer fatigue lives than the 15 h treated material. However, there is no discernible difference in the fatigue limit that is 390 MPa for both carburized materials. The fatigue limit of the untreated material is 300 MPa, thus the improvement by 30% is achieved with the modified carburizing employed in the present study.

Fatigue tests at the fatigue limit were continued to \( N = 5 \times 10^7 \) cycles, because it has been indicated that subsurface fracture occurred in long life regime in surface-modified materials [10–14] and high strength steels [15–20]. However, no fatigue failure took place, thus the carburized materials would not show fatigue failure in much longer life regime.

3.4.2. Crack initiation

Fig. 4 reveals typical examples of SEM micrographs of fracture surface near the crack initiation site. In the untreated material (Fig. 4(a)), the crack is generated at the specimen surface due to cyclic slip deformation, because a stage I like facet was seen at the crack initiation site regardless of stress level. After crack initiation, the crack grows radially into the interior of the specimen.

In the carburized materials (Fig. 4(b),(c)), it should be noted that the cracks initiate underneath the carburized case i.e. at or near the boundary between the carburized case and the core material independent of stress level and there exists no nonmetallic inclusion at the subsurface crack initiation site that is usually seen in high strength steels. Similar subsurface crack initiation was also observed in austenitic stainless steels treated by shot peening [2–4].

A close examination reveals a smooth facet in the carburized case just above the subsurface crack initiation site, particularly remarkable in the 35 h
treated material. It is also worth noting that there exists a fish-eye like pattern that extends predominantly into the core material and the sizes are approximately 150 μm in the radial direction.

### 3.4.3. Small crack growth

As seen in Fig. 4(b),(c), the crack initiation site of the carburized materials was subsurface at or near the boundary between the carburized case and the core material. Since the case depths are very shallow and a brittle facet was seen in the carburized case, it was thought that the crack initiated reaches the specimen surface immediately. In order to confirm this hypothesis, cracks were monitored on the specimen surface by replication technique. Consequently, it was possible to measure the crack growth on the surface of the carburized materials.

The surface crack length, 2c, is represented in Fig. 5 as a function of cycle ratio, N/N_f (N_f: fatigue life). In the untreated material, cracks initiated at an early stage of fatigue life and grew gradually with increasing number of cycles. In the carburized materials, cracks were also detected at an early stage of fatigue life independent of case depth and stress level, this indicates that the subsurface crack initiation underneath the carburized case has occurred at much earlier stage of fatigue cycling. Therefore, the fatigue life is dominated by small crack growth. It should be noted that cracks grew very slowly in discontinuous manner in the region of crack length of 2c < 0.2 – 0.3 mm. Subsequently, they grew rapidly and continuously to final fracture with increasing number of cycles.

Fig. 6 shows the relationship between crack growth rate at the specimen surface, dc/dN, and 2c in the untreated and carburized materials. It should be noted that in the carburized materials, temporary decreases in dc/dN often take place in the region of crack length of 2c < 0.3 mm. This corresponds to the discontinuous crack growth seen in Fig. 5. Such a crack growth seems to have occurred due to intermittent brittle fracture of very hard carburized case. Therefore, Fig. 6 does not
3.5.1. Fatigue strength

Fig. 7 shows the $S$–$N$ diagram in 3% NaCl aqueous solution. For comparison, the $S$–$N$ curves in laboratory air are also included without experimental data points. In the untreated material, the fatigue strength in 3% NaCl aqueous solution is lower than that in laboratory air and the reduction increases gradually with decreasing stress level. This is the well-known corrosion fatigue behaviour. On the contrary, the carburized materials exhibit longer fatigue lives at higher stress levels than in laboratory air, but tend to show nearly the same fatigue lives as in laboratory air with decreasing stress level. In addition, the fatigue limit seems to exist in the corrosive environment within the range of experiment.

The increase of fatigue life at higher stress levels in 3% NaCl aqueous solution is believed to be due to suppression of temperature raise resulted in during fatigue cycling. The surface temperature was not measured in the present study, but significant temperature raise has also been indicated in austenitic stainless steel [2,21,22]. Thus, a few additional fatigue tests were performed in laboratory air using the carburized specimens that are forced to cool by air. The obtained data are plotted in Fig. 7. The fatigue lives are slightly longer than in laboratory air, but shorter than in 3% NaCl aqueous solution. These results seem reasonable, because the cooling effect by the solution is much larger than by air.

As indicated above, it should be emphasized that the materials hardened by the present carburizing showed no reduction of fatigue strength in aqueous corrosive environment.

3.5.2. Crack initiation

Typical examples of SEM micrographs of fracture surface near the crack initiation site in 3% NaCl aqueous solution are shown in Fig. 8. In the untreated material (Fig. 8(a)), the crack initiates at the surface and subsequent small crack growth seems to be affected significantly by corrosive environment because of brittle nature of the fracture surface. In the carburized materials (Fig. 8(b),(c)), it is worth noting that the cracks always initiate underneath the carburized case even in 3% NaCl aqueous solution and a brittle facet can be seen in the case just above the subsurface crack initiation site, particularly in Fig. 8(c), which has occurred due to crack initiation and growth in the interior of the specimens.

The depth to the crack initiation site from the surface, $d_c$, is represented in Fig. 9 as a function of stress amplitude in both environments. It can be seen that the subsurface crack initiation site is roughly equal to the case depth regardless of environment and stress level.

3.5.3. Crack growth

As well as in laboratory air, crack initiation and small crack growth were monitored with replication technique in 3% NaCl aqueous solution. Fig. 10 shows $2c$ as a function of $N/N_f$. As described previously, the crack initiation site was subsurface, but cracks are
detected at a relatively early stage of fatigue cycling. Subsequent small crack growth behaviour is also similar to that in laboratory air. These results suggest that the fracture mechanism operated is basically the same in both environments.

4. Discussion

4.1. Improvement of fatigue strength in laboratory air

As shown in Fig. 3, the fatigue strength of the carburized materials was improved considerably compared with the untreated material and the fatigue limit increased by 30% by the modified carburizing. This is due to suppression of slip deformation at the specimen surface because of remarkable hardness increase, i.e. the resistance to crack initiation is significantly enhanced in the carburized case. Therefore, crack initiation becomes difficult to occur at the surface and then moves from the surface to the subsurface between the
carburized case and the core material. The fatigue limit of the carburized materials was 390 MPa that is considerably higher than the proof stress (299 MPa) and the fatigue limit (300 MPa) of the untreated material. This is because the limiting stress for crack initiation could be enhanced due to constraint of deformation by the hard carburized case.

In the finite life region, the effect of treatment time, i.e. case depth on fatigue strength was slightly seen where the fatigue lives of the 35 h treated material were longer than those of the 15 h treated material. This also may be due to enhancement of the crack initiation resistance and constraint of the carburized case to small crack growth because of increased hardness of the carburized case with increasing treatment time.

4.2. Effect of corrosive environment on fatigue behaviour

As shown in Fig. 7, the carburized materials showed no reduction of fatigue strength in 3% NaCl aqueous solution. Since the fatigue strength of the untreated material decreased significantly in the corrosive environment, the present modified carburizing can prevent the decrease of corrosion fatigue strength.

In the untreated material, the early small crack growth was affected by 3% NaCl aqueous solution and brittle fracture surface was observed, which seems to suggest the effect of hydrogen embrittlement. The authors have indicated the same behaviour, in which the early small crack growth of a 304 austenitic stainless steel was enhanced in 3% NaCl aqueous solution compared with laboratory air [23,24]. On the other hand, such a brittle appearance was not seen in the carburized materials (Fig. 8(b),(c)). It is supposed that the presence of the carburized case would exert some influence on the corrosion reaction within the crack.

4.3. Subsurface crack initiation

The hardness of the carburized case was increased remarkably by carburizing as shown in Fig. 2. As discussed previously, the strength of the carburized case increases remarkably compared with the core material, thus crack initiation at the surface becomes difficult and then moves to the subsurface near the boundary between the carburized case and the core material.

It has been well known that there exists the following correlation between fatigue strength (fatigue limit) and Vickers hardness [25].

$$\sigma_W = 1.6HV$$

The strengths of the carburized materials estimated from Eq. (1) are shown in Fig. 11 as a function of the distance from the surface. Also included are the applied stress distributions at $\sigma = 500$ and 400 MPa and the depth to the crack initiation site plotted against applied stress. It is easily understood that surface-related fracture would not take place, because the strengths of the carburized case are considerably higher than applied stresses. It can also be seen in the figure that the distance from the surface where applied stresses become higher than the strengths of the carburized materials is approximately 20–40 $\mu$m, which is nearly the same as the case depth and the subsurface crack initiation site of 30–50 $\mu$m. Therefore, the subsurface crack initiation behaviour in the present carburized materials can be realized due to such a simple strength-applied stress model.

As described above, the carburized case can endure much higher applied stresses. Even though the stress gradient is considered, applied stresses at the subsurface crack initiation site, i.e. the boundary between the carburized case and the core material, are still significantly higher than the fatigue limit and the proof stress of the untreated material. This is the cause of the observed early crack initiation, and subsurface cracks can generate due to slip deformation at the higher applied stresses that exceed the constraint of deformation by the carburized case. Fig. 12 shows a matching fracture surface near
the crack initiation site in laboratory air. It should be noted that there exists a stage I facet at the subsurface crack initiation site in both fracture surfaces.

4.4. Fracture process or mechanism

Based on the fracture surface analysis and the observation of crack growth at the surface, the fracture process or fracture mechanism of the carburized materials is illustrated schematically in Fig. 13. The crack initiated at or near the boundary between the carburized case and the core material immediately appears on the surface (Fig. 13(a)). This is due to brittle fracture of the carburized case because the hard case cannot accommodate large deformation due to crack initiation and subsequent small crack growth. In fact, the crack size observed with the first replica was in good agreement with the surface length of a brittle facet in the carburized case seen on the fracture surfaces. Subsequently, the crack extends predominantly into the core material with a fish-eye like pattern because of the constraint and the compressive residual stress in the carburized case (Fig. 13(b)). When the crack grows to some extent, brittle fracture in the carburized case occurs again. These crack growth processes result in the discontinuous or intermittent crack growth observed at the specimen surface. When the crack length reaches 0.2–0.4 mm, the crack shape becomes semicircular and the continuous crack growth takes place (Fig. 13(c)).

As an additional note, although there was no discernible difference in the crack initiation behaviour between the 15 and 35 h treated materials, the latter material had a strong propensity of discontinuous crack growth. This is because more brittle nature of the carburized case in the 35 h treated material.

5. Conclusions

In the present study, rotary bending fatigue tests were performed in laboratory air and in 3% NaCl aqueous solution using materials with two different case depths of a carburized 316 austenitic stainless steel. Fatigue strength was evaluated and fracture mechanism was discussed on the basis of observation of crack initiation and small crack growth and fracture surface analysis. The conclusions can be made as follows:

1. The surface hardness in the carburized case attained to more than 800HV and 1000HV for the 15 and 35 h treated materials, respectively. Hardness decreased rapidly with increasing the distance from the surface and then reached the core hardness at approximately 40 \( \mu \)m from the surface regardless of treatment time. The case depths established by the microstructure characterization were 20 and 40 \( \mu \)m for the 15 and 35 h treated materials, respectively.
2. The tensile strength and ductility of the carburized materials slightly increased and decreased, respectively, compared with the untreated material and with increasing treatment time.
3. In laboratory air, the fatigue strength of the carburized materials increased considerably compared with the untreated material. The case depth dependence of fatigue strength was seen in the finite life region where the fatigue lives of the material with thick
case were slightly longer than those of the material with thin case, while there was no discernible difference in the fatigue limit (390 MPa) that was 30% increase with respect to the fatigue limit (300 MPa) of the untreated material.

4. In 3% NaCl aqueous solution, the untreated material showed significant decrease in fatigue strength compared with laboratory air, while the carburized materials exhibited no reduction of fatigue strength, indicating excellent corrosion resistance of the carburized case.

5. Fatigue cracks initiated at a very early stage of fatigue life near the boundary between the carburized case and the core material due to cyclic slip deformation and thus fatigue life was dominated by small crack growth.

6. The crack growth at the specimen surface was discontinuous. This was due to intermittent brittle fracture of the carburized case because of preceded crack growth in the subsurface.

7. Crack initiation and small crack growth behaviour were the same in both environments, thus the fracture mechanism of the carburized materials was unaffected by corrosive environment.

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