Effect of Flaws in Coating Film on Fatigue Strength of Steel Coated with Titanium Nitride*

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To clarify the effect of flaws in the coating film on fatigue strength, cantilever-type rotating-bending fatigue tests were conducted in air and in saline solution (3.0% NaCl) using specimens of 0.37%C steel with flaws in the coating of titanium nitride (TiN) thin film coated by PVD and CVD methods. Flaws in the coating film on the specimen surface were introduced by the application of 1.1-1.6% static tensile strain before the test. An obvious decrease in fatigue life of the specimen with flawed coating film was observed in both environments, as compared with that of an uncoated specimen and a specimen with unflawed coating film. This behavior was marked for fatigue in air, that is, the decrease of fatigue life was 90-75% in air as opposed to 70-50% in saline solution. Although film thickness was 3-5 μm, the flaw in the film had the same effect as a notch at which cracks initiate on the substrate. Many cracks were induced in the substrate directly under a flaw and coalesced into a large crack at an early stage of fatigue in air. In corrosion fatigue, corrosion pits at which cracks initiate occur on the substrate under a flaw at an early stage of the fatigue process, and the incubation period prior to the formation of pits does not occur.

Key Words: Fatigue, Corrosion Fatigue, Coating, Surface Treatment, Titanium Nitride, Thin Film, Flaw, Chemical Vapor Deposition, Physical Vapor Deposition, Carbon Steel

1. Introduction

The fabrication of a thin layer of titanium nitride (TiN) on the surface of various engineering components by a variety of techniques has received considerable attention in the past few years. A number of superior properties of TiN thin film produced by physical vapor deposition (PVD) or chemical vapor deposition (CVD), such as, high hardness, high resistance to wear, chemical stability, corrosion resistance, relatively good adhesion of the films and an attractive color, may be attributed to surface improvement of metals. Coating technology on materials will be utilized more widely for various kinds of machine components and structures which require high wear resistance, high corrosion resistance and cavitation-erosion resistance.

Another interesting application of hard thin films coated on metals is to improve the fatigue strength of metals in various environments. A hard coating layer well-adhered to a substrate material will affect the mechanisms of plastic deformation and crack initiation during the fatigue process, but so far there is very little information available about the effect of a coating film on the fatigue behavior of metals[(1)-(6)]. One of the authors has previously reported that the fatigue life of TiN-coated carbon steel was greater in air and in 3% saline solution as compared with that of an uncoated specimen[(7)-(9)]. This is due to the fact that TiN coating film improves crack initiation life in air, and protects the substrate from a corrosive environment. The influence of the applied stress ratio on the fatigue strength of carbon steel coated with TiN has also been studied, and it has been clarified that fatigue life is affected by the fracture behavior of the coating film on the specimen surface[(10)]. Under a testing condition where large deformation occurs or accumulates during fatigue, TiN coating film is fractured at

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an early stage of the fatigue process because it is too brittle to accommodate the deformation of substrate metal. Flaws in the coating film induce the initiation of fatigue cracks on the substrate.

These experimental results indicate that a hard coating film on the specimen surface can act as a barrier to the egress of dislocations and thus delay fatigue crack initiation. Flaws in the coating film act as notches at which cracks initiate on the substrate. It will be important for practical use to clarify the fatigue behavior of coated components which have flaws or defects in the film, because there is a likelihood that flaws or defects will form during the deposition process and during use. The purpose of this investigation is to clarify the fatigue behavior of steel coated with TiN, in order to apply ceramics coatings to machine components and structures.

In this study, cantilever-type rotating-bending fatigue tests were conducted in air and in saline solution using specimens of 0.37 wt% carbon steel with flaws artificially induced in TiN thin film coated by PVD and CVD methods. The effect of flaws in coating film on fatigue life is discussed through the observation of crack initiation sites and fracture surfaces.

2. Experimental Procedure

2.1 Testing material and coating conditions

The substrate metal used in this study was 0.37 wt% carbon steel, JIS S 35 C, normalized at 1138 K for 30 min. The chemical composition of this steel is shown in Table 1. Specimens were smooth and hour-glass-shaped with a minimum diameter of 10 mm for fatigue tests in air and of 8 mm for corrosion fatigue tests in saline solution. Figure 1 shows the geometry and size of specimens which were machined after heat treatment. Before TiN deposition, the substrate was polished with emery paper up to grade #1000 and electropolished to a depth of about 15 μm.

TiN coating was deposited onto the specimen surfaces by use of PVD or CVD processes. In CVD coating, specimens were inserted into a stream of mixed gases (H_2 : N_2 : $TiCl_4$ = 32.8 : 65.6 : 1.6 [vol %]) under a reduced pressure of 3.33×10^3 Pa at 1223 K for 3 h. Thickness and Vickers hardness of the CVD-coated layer were 5-6 μm and H_v(50 gf) = 2170, respectively. By observation using an optical microscope, the ferrite and pearlite structure of the CVD-coated specimen was formed to be larger than that of the uncoated one[10]. This is due to the high temperature of the CVD coating process. Also, decarburization of the substrate near the CVD-coated TiN layer was observed.

In PVD coating, the hollow cathode discharge process was employed in vacuum to generate a glow discharge in nitrogen into which titanium was evaporated at a constant substrate temperature of 623 K. The thickness of the coating film was 2 - 3 μm and Vickers hardness was H_v(15 gf) = 1888. Mechanical properties of the normalized, annealed, PVD- and CVD-coated specimens are shown in Table 2.

2.2 Method to make artificially flaws in coating film

The TiN layer coated by PVD and CVD is so brittle that it is easily fractured by small tensile strain. The experimental relationship between flaw density on the coating film and total tensile strain of the specimen has been reported by the authors[10]-[13]. It was found that flaws in the coating film appeared at the total tensile strain of 0.40% for PVD film and 0.87% for CVD film, and their amount increased with tensile strain.

Based on the experimental results, tensile strain of about 1.1 - 1.6% was applied to the coating film to introduce the desired flaw density, where flaw density was defined as the number of flaws per millimeter along the axial direction. The specimens with flaw density, ρ, of 20 - 24/mm for PVD film and 23 - 26/mm for CVD film were prepared in this experiment. Also, to examine the effect of flaw density on the corrosion fatigue behavior, different flaw densities of 10, 30 and 47/mm were introduced on the PVD-coated specimens. Figure 2 shows a typical example of the flaws on the CVD coated film obtained by

Table 1 Chemical composition of JIS S 35 C used

(wt%)

C	Si	Mn	P	S	Cu	Ni	Cr
0.37	0.24	0.77	0.019	0.023	0.1	0.2	0.4

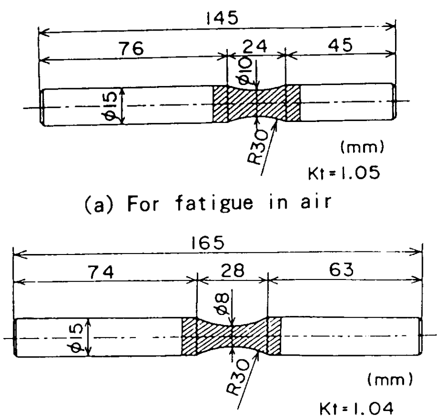

Fig. 1 Shape and dimensions of fatigue test specimens; (a) for fatigue tests in air, and (b) for corrosion fatigue tests in saline solution

Table 2 Mechanical properties of the tested materials

Materials	Upper yield stress σ_{SU} (MPa)	Lower yield stress σ_{SL} (MPa)	Tensile strength σ_B (MPa)	Young's modulus E (GPa)	Elongation δ (%)	Area reduction φ (%)	Hardness Hv (100gf)	Grain size d (μm)
S35C (N)	401	371	616	204	25.8	60.1	213	16
PVD-coating	389	354	615	206	25.1	63.2	219	18
CVD-coating	316	295	580	204	24.0	51.4	210	31
S35C (A)	-	-	-	-	-	-	195	35

(N) : Normalized at 1138K for 0.5h, air cool
(A) : Annealed at 1223K for 3h, furnace cool

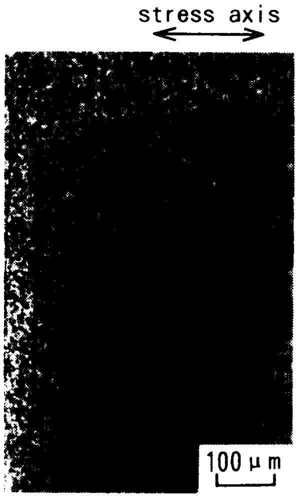

Fig. 2 An example of flaws on CVD-coated TiN film induced by static tension

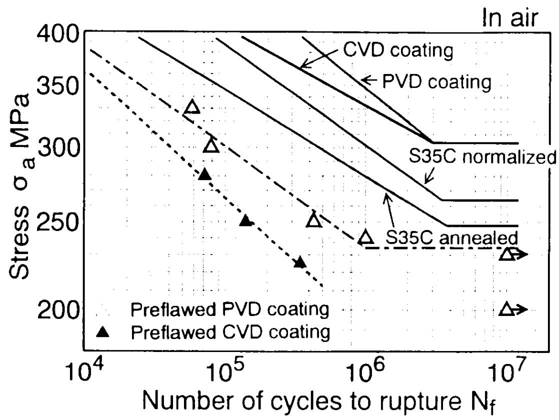

Fig. 3 S-N diagram for fatigue test in air using specimens with preflawed coating film

observation using an optical microscope of the plastic replica which was taken from the specimen surface after applying the tensile strain. Flaws of the coating film were aligned perpendicular to the specimen axis.

2.3 Testing method

Fatigue testing was conducted in air at room temperature and in a 3.0%NaCl aqueous solution environment, using a cantilever-type rotating-bending fatigue machine under a testing frequency of 29.7 Hz. In corrosion fatigue tests, saline solution controlled at 298±2 K was continuously circulated in a plastic reservoir through the cell at a flow rate of about 32 ml/min. The test was interrupted periodically to take plastic replicas from the specimen surface, which were examined using an optical microscope or a scanning electron microscope (SEM).

3. Experimental Results

3.1 S-N curve

Figure 3 shows the experimental results obtained from the fatigue tests in air, using the specimens with preflawed coating film. In this figure, S-N curves indicated by solid lines for CVD- and PVD-coated specimen, and uncoated normalized and annealed*[1] specimens are also shown for comparison[7],[9]. It can be seen in this figure that the fatigue life of the specimen with preflawed coating film clearly decreases, as compared with the specimen with unflawed coating film. It is of great importance that the fatigue strength of the specimen with preflawed coating film is less than that of an uncoated specimen. Preflaws in the coating film affect not only the finite fatigue life but also the endurance limit. The endurance limit of specimens with preflawed PVD coating film decreases about 11% and 22% as compared with the normalized and PVD-coated specimens without preflaws, respectively.

Figure 4 shows S-N curves of the specimens with preflawed PVD and CVD coating films obtained by the corrosion fatigue test. Solid lines in this figure indicate the experimental results obtained in a previous study from the PVD- and CVD-coated, and uncoated specimens[8],[9]. A decrease of corrosion fatigue life in

*[1] CVD coating was performed at high temperature and then the substrate was annealed. A specimen heat-treated under the same conditions as those in the CVD coating process was used for comparison of fatigue behavior of CVD-coated specimen.

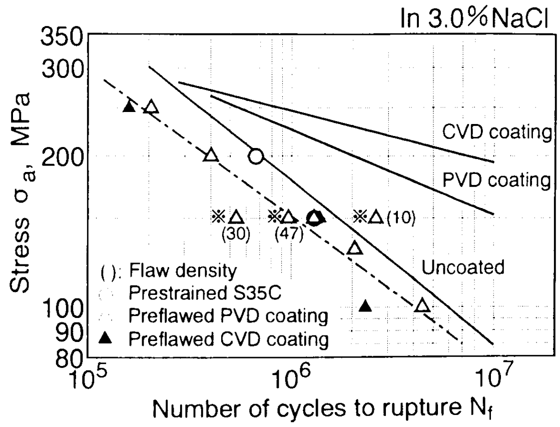

Fig. 4 S-N diagram for corrosion fatigue test in 3.0% NaCl saline solution using specimens with preflawed coating film

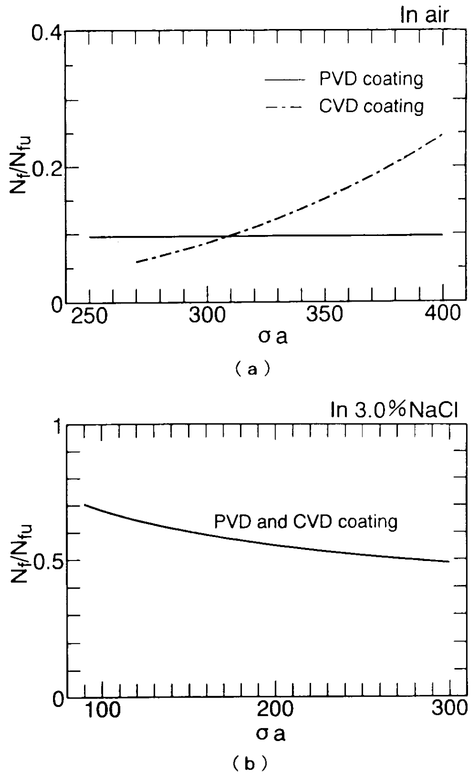

Fig. 5 Decrease in fatigue life of specimens with preflawed coating film: (a) fatigue in air, and (b) corrosion fatigue in saline solution

the specimens with preflawed coating film was also observed. Since no effect of the application of tensile prestrain of 1.5% to the uncoated specimen on corrosion fatigue strength was observed, as shown in this figure, the decrease in corrosion fatigue strength of specimens with preflawed coating film may be due to the preflaws in the coating film. It can be seen from this figure that there was no difference in corrosion fatigue life between specimens with preflawed PVD coating film and those with preflawed CVD coating film, just as heat treatment condition, annealing and normalizing had no effect on corrosion fatigue life of uncoated specimens.

The relationship between stress amplitude, σ_a, and N_f/N_{fu} is shown in Fig. 5, where N_f is the fatigue life of the specimens with preflawed coating film and N_{fu} is that of the uncoated specimen. In the case of fatigue in air, N_f/N_{fu} for the PVD-coated specimen is about 0.1 independent of stress amplitude, whereas for the CVD-coated specimen, it varies between 0.1 - 0.25 depending on stress amplitude. On the other hand, N_f/N_{fu} of corrosion fatigue is about 0.5 - 0.7 for PVD- and CVD-coated specimens. Therefore, the effect of preflaws in coating film on the decrease of fatigue life is greater in air than in saline solution. This is due mainly to the difference in fatigue crack initiation mechanisms which will be discussed in a subsequent section.

In order to discuss the effect of preflaw density in coating film on corrosion fatigue life, corrosion fatigue testing was conducted under $\sigma_a = 150$ MPa using the PVD-coated specimens having different preflaw densities of 47, 30 and 10/mm introduced by applying static tensile strain. The results obtained are plotted in the S-N diagram of Fig. 4 and also shown in Fig. 6 as the relationship between the density of

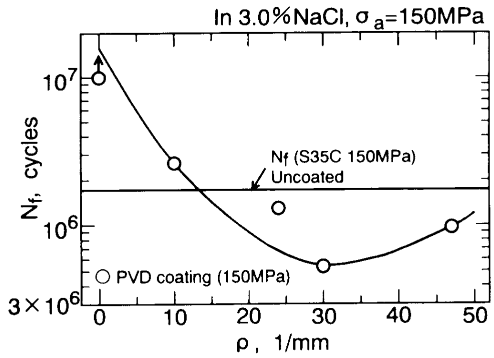

Fig. 6 Relationship between corrosion fatigue life in PVD-coated specimen and preflaw density in coating film determined from experiment ($\sigma_a = 150$ MPa)

preflaws in coating film, ρ, and corrosion fatigue life, N_f. N_f of specimens with preflawed coating film decreases as ρ is increased. There is clearly a trend wherein corrosion fatigue life is the shortest at certain density of preflaws, and for larger ρ N_f increases and approaches the corrosion fatigue life of uncoated specimen as ρ is increased.

3.2 Observation of specimen surface

Figure 7 shows optical microscopic observations of plastic replicas which were taken from the specimen surface after corrosion fatigue testing. It was observed that the flaws in the PVD coating film were perpendicular to the specimen axis and the exfoliation of the coating film was occurred. From the comparison of Figs. 7(a) and (b), the area of exfoliation of the coating film on the specimen surface depends on the preflaw density, that is, exfoliation occurred more easily during fatigue with higher density of preflaws in film on the specimen surface.

SEM observation results of PVD-coated specimen surfaces are shown in Fig. 8. It can be clearly seen from the micrographs that the coating film was peeled off from the substrate metal along the straight flaws of film, and corrosion pits were formed on the exposed substrate by dissolution.

3.3 Fractography

Figure 9 shows typical scanning electron micrographs of corrosion pits on the fracture surface beneath the PVD or CVD coating film. Small corrosion pits were initiated on the substrate metal along the straight flaws of the coating film, through which saline solution penetrated. Corrosion fatigue cracks of coated specimens were initiated at these corrosion

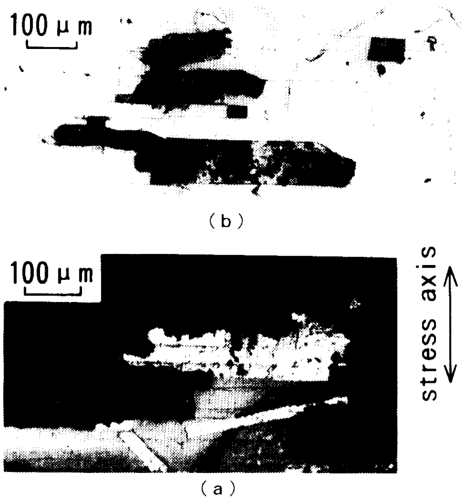

Fig. 7 Surface morphology of specimen with preflawed PVD coating film after corrosion fatigue testing: (a) preflaw density ρ = 47/mm, σ_a = 150 MPa, and (b) ρ = 24/mm, σ_a = 250 MPa

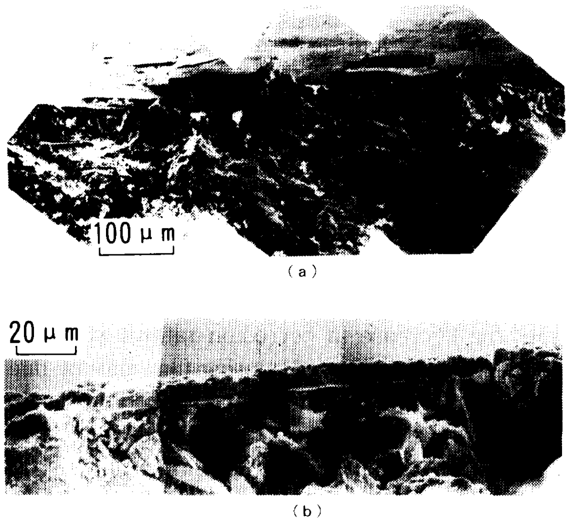

Fig. 9 Typical examples of fracture surface for (a) PVD coated specimen and (b) CVD coated specimen tested under corrosion fatigue

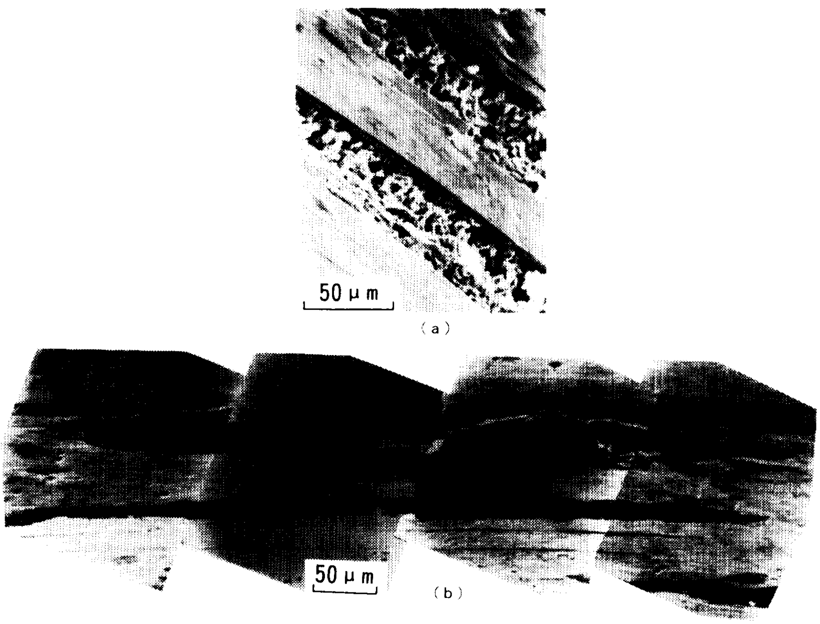

Fig. 8 SEM observation of surface of specimen with preflawed PVD coating film under corrosion fatigue testing

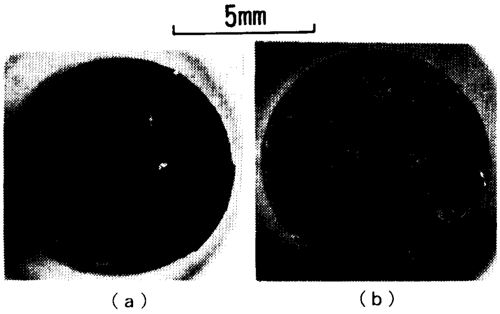

Fig. 10 Macroscopic observation of fracture surface for specimens with preflawed (a) PVD and (b) CVD coating films under corrosion fatigue test ($\sigma_a = 150$ MPa)

pits and propagated.

Figure 10 shows macroscopic observation of the fracture surface of the specimens tested under corrosion fatigue. Fracture surface observation of specimens with preflawed PVD and CVD coating films revealed many ratchet marks which are the origins of multiple fatigue cracks, each of which produce a separate fatigue crack zone. Corrosion fatigue cracks propagated from small corrosion pits which were caused by corrosive solution. This morphology was the same as that of uncoated specimens tested in saline solution, although the number of ratchet marks on the fracture surface of the coated specimen was less than that of the uncoated specimen since the coating improved the corrosion fatigue strength[8],[9]. It is concluded that the number of cracks on the fracture surface initiated at corrosion pits is inversely correlated with corrosion fatigue life of the specimen with preflawed coating film.

4. Discussion

From the experimental results of the specimen with preflawed coating film under fatigue tests in air and in saline solution, it was clearly found that the fatigue life of the specimen with preflawed coating film drastically decreased as compared with that of an uncoated specimen or one with unflawed coating film. It can be easily understood that corrosion fatigue strength in the specimen with flawed coating film is less than that of the specimen with unflawed coating film, because the substrate surface is exposed to a corrosive environment. The finding that the fatigue life in both environments is less for a specimen with preflawed coating film than for an uncoated specimen is unfortunate for surface improvement technology and cannot be explained by common sense. In this section, possible mechanisms of the decrease of fatigue strength due to flaws in coating film will be discussed.

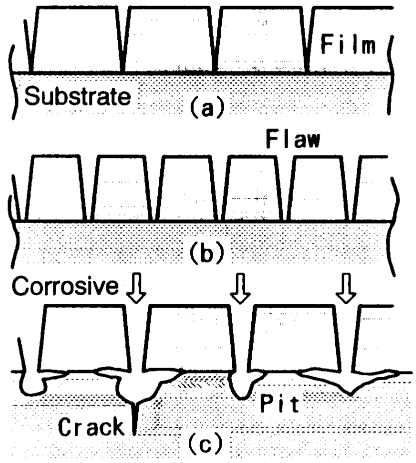

Fig. 11 Schematic illustration to explain the formation of corrosion pits and crack initiation of specimen with preflawed TiN coating film

4.1 Mechanism of decrease in corrosion fatigue strength

The corrosion fatigue crack initiation mechanism of specimens with flawed coating film is summarized in Fig. 11. Corrosion pits are likely to form on the substrate metal due to saline solution penetrating throughout the flaws in the coating film. Dissolution of substrate metal may be accelerated not only by the galvanic action of local corrosion cells composed of the coating (cathode) and substrate (anode), but also by the existence of residual tensile stresses on the substrate which are formed by the balance of residual compressive stresses in the coating film during the deposition process[11],[12]. The experimental results presented in the previous section are explained with due consideration of the model shown in Fig. 11:

(1) The reason that corrosion fatigue life in the specimen with preflawed coating film is shorter than that in the uncoated specimen is that the corrosion fatigue process of metal consists of the nucleation and growth of corrosion pits, crack initiation at corrosion pits, and crack growth. Since TiN is electrochemically nobler than steels, the substrate area exposed to saline solution through the flaws acts as an anodic area and as a nucleus for the formation of corrosion pits. Therefore, it is suggested that the incubation period prior to the formation of corrosion pits does not occur and crack initiation at the pits occurs on the substrate at an early stage of the corrosion fatigue process in the specimens with preflawed coating film. Figure 12 shows a typical example of crack initiation at a corrosion pit which is under a flaw in the coating film. The SEM micrograph was taken in an axial cross section of a CVD-coated specimen.

The ratio of incubation period for pit formation

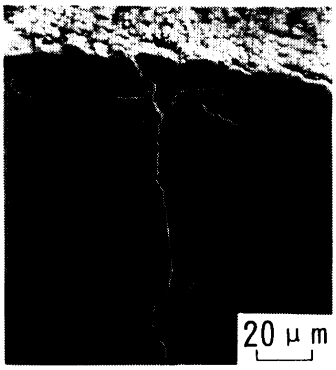

Fig. 12 SEM observation of the cross section of a CVD-coated specimen showing corrosion pits and a crack just below flaws in the coating film

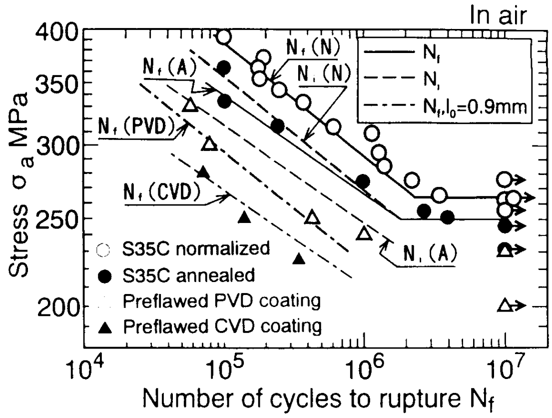

Fig. 13 Estimation results of fatigue life in air for specimen with preflawed coating film

and initiation of cracks to corrosion fatigue life is usually about 10 - 20 % for steel. The unexpected decrease of 50 - 70 % in corrosion fatigue life in specimens with preflawed coating film cannot be explained by only the decrease of crack initiation life. It is suggested that a complete explanation must take into account the initiation of many cracks induced at corrosion pits directly under a flaw and the formation of a large crack by coalescence at an early stage of fatigue, in addition to the disappearance of an incubation period.

(2) Dependence on preflaw density of corrosion fatigue life: The opening crevice of a flaw in coating film caused by deformation of the substrate may be proportional to the flaw density which is related to tensile strain, as shown in Figs. 11(a) and (b). The corrosive solution penetrates more easily to the substrate metal through the opening crevice, and the probability of pit formation along a flaw increases as flaw density increases. After increasing past a certain preflaw density, the coating film peels off from the substrate surface because of dissolution at the interface, and then residual tensile stresses on the substrate are released. In this situation, the effect of flaws in the coating film on the formation of corrosion pits disappears.

4.2 Mechanism of decrease in fatigue strength in air

Preflaws in the coating film have the same effect as notches for crack initiation on substrates and decrease the fatigue life in air, even though the film thickness is 3 - 5 μm which is quite shallow compared to a notch. The fatigue life, N_f, in air is controlled by the crack initiation life, N_i, and the crack growth life, N_p. It is generally recognized that N_i/N_f is about 1/2 - 1/3 for a smooth specimen of steel[14]. The experimental finding in this study that the fatigue life in a specimen with preflawed coating film is about 10% that of an uncoated specimen cannot explain the decrease of crack initiation life due to the notch effect of flaws, even though N_i is negligible. The effect of flaws in the coating film in decreasing fatigue life will be examined by predicting the fatigue life using the small fatigue crack growth law.

Fatigue crack growth life, N_p, is estimated using the following small fatigue crack growth law proposed by Nishitani and coworkers[15],[16]:

$$N_p=\frac{1}{C_3}\left(\frac{\sigma_B}{\sigma_a}\right)^n\left\{\ln\left(\frac{l_1}{l_0}\right)+\ln\left(\frac{d}{5}\right)\right\}, \quad (1)$$

where C_3 and n are material constants, σ_B is ultimate tensile strength, d is diameter of the specimen, and l_0 and l_1 are initial and final crack lengths, respectively. C_3 and n were determined from the experimental results reported by Nishitani et al.[15]: that is, $C_3=1.2\times10^{-2}$ and $n=9.5$ for annealed carbon steel, and $C_3=3.4\times10^{-3}$ and $n=8.23$ for normalized carbon steel. For estimation of fatigue life in uncoated steel, it was assumed that l_0 and l_1 are 0.05 mm and 1 mm, respectively, and $N_f=N_i+N_p=2N_p$ ($N_i/N_f=0.5$). Estimated results are shown as solid lines in Fig. 13. Solid lines denoted by $N_f(N)$*2 and $N_f(A)$*2 are in good agreement with the experimental results plotted in this figure. Broken lines denoted by $N_i(N)$ and $N_i(A)$ in this figure indicate the crack growth life predicted for normalized and annealed steel, respectively. The fatigue life of specimens with preflawed coating film is expected to coincide with this line under the assumption that cracks will initiate just behind the flaw and grow immediately after fatigue loading, and based on the experimental finding that the fatigue crack growth rate is not affected by the coating[7],[9]. However this

*2 N and A in parentheses mean that estimation was made using Eq.(1) with the parameters of normalized and annealed steel, respectively.

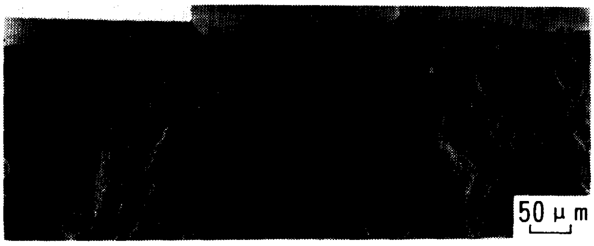

Fig. 14 SEM observation of the region around the origin of fatigue crack in specimen with preflawed CVD coating film tested in air

hypothesis is incorrect. Dot-dashed lines indicated by N_f (PVD) and N_f (CVD) in Fig. 13 show the predicted results obtained from Eq.(1) using l_0=0.9 mm. They are in good agreement with the experimental results obtained from the specimens with preflawed PVD and CVD coating films. This means that the specimen with preflawed coating film has a crack of 0.9 mm length in an early stage of the fatigue process, namely the size of preflaws in coating film is equivalent to that of a crack in the specimen.

Figure 14 shows a typical example of observation by SEM in the region around the origin of fatigue cracks in a specimen with preflawed CVD coating film. It can be seen from the figure that many cracks initiate at the substrate along the straight flaw of the coating film, and these cracks propagate independently and coalesce at an early stage of the fatigue process. The mechanism of crack initiation and propagation in a specimen with flawed coating film has already been proposed in other papers by the authors[11],[13]. It is indicated from the above discussion that a shallow flaw in the coating film on the specimen surface acts as both a notch at which crack initiate and an initial crack in the specimen, and this then degrades fatigue strength.

5. Conclusions

The effect of flaws in TiN coating film introduced by the application of static tensile strain, on fatigue strength in air and in saline solution was discussed through the cantilever-type rotating-bending fatigue tests using specimens of carbon steel coated by PVD and CVD processes, and the following conclusions were obtained.

(1) Fatigue life of the specimen with preflawed coating film obviously decreased in both environment, as compared with those of an uncoated specimen and one with unflawed coating film. The effect of preflaws in coating film on fatigue life was more pronounced in air than in saline solution.

(2) Decrease in corrosion fatigue life depended on the preflaw density in the coating film, and the shortest fatigue life was found to occur at a certain flaw density.

(3) In corrosion fatigue, crack initiation on the substrate which occurred at corrosion pits was accelerated by crevice corrosion due to the penetration of corrosive solution into the substrate through the flaws, and by the disappearance of an incubation period prior to the formation of pits.

(4) Decrease in fatigue life in air caused by preflaws in the coating film was due to the facts that the flaws have the same effect as a notch for crack initiation on substrate, and many cracks are induced in the substrate under a flaw and form a large crack by coalescence at an early stage of fatigue.

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