Some affecting factors for fatigue strength of ceramics coating steel
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Abstract
Effect of TiN and CrN coating films deposited by PVD and CVD methods on fatigue behavior of carbon steel in air and in 3% saline solution was discussed. Fatigue life of metal was improved by coating, but this was affected by some factors, such as applied stress ratio, stress or strain amplitude, preflaws or flaws in the coating film occurred during fatigue process, coating film thickness and defects in the coating film.

1 Introduction
Numerous methods of surface treatment have been developed in the past for the purpose of the increase strength and extension of lifetime of the engineering components. For strength and failure resistance, the most sensitive part of component is its surface for reasons that the surface is directly exposed to unfavorable action of the environment and also the place with the best conditions for nucleation and growth of cracks. The resistance in the individual cases can be increased by various means depending on the substrate material and on the character of loading and environment. A desired properties of the surface layer can be achieved by two principal methods of surface treatment: One is that the surface layer on the substrate is modified with changing the composition or microstructure by chemical, thermochemical, thermal or mechanical action. The other is that the surface layer is created on the substrate surface by coating from other material [1].

The fabrication of a thin layer of ceramics 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 ceramics 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 and relatively good adhesion of the films and an attractive color, may be attributed to surface improvement of metals. Coating technology on materials will be
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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. It will be considered that the surface treatment using ceramics coating with some superior properties brings an improvement in one respect, and worsening in another.

The purpose of this investigation is to clarify the fatigue behavior of steel coated with ceramics, in order to apply ceramics coatings to machine components and structures. In this paper, through the some fatigue test results obtained in air and in 3% saline solution using specimen of 0.37 wt% carbon steel coated with TiN or CrN by PVD or CVD methods, the effect of coating film on fatigue strength is discussed from some points of view as loading conditions, environments and microstructure of coating film.

2 Testing material and coating conditions

The substrate metal used in this study was middle carbon steel, normalized at 1138K for 30 min. The chemical composition of this steel (wt%) is 0.37C, 0.24Si, 0.77Mn, 0.019P, 0.023S, 0.1Cu, 0.2Ni and 0.4Cr. 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. Specimens were machined after heat treatment and the surface was polished with emery paper up to grade #1000 and electropolished to a depth of about 15 µm, before ceramics deposition.

Ceramics coating was deposited onto the specimen surfaces by use of PVD or CVD processes. In PVD coating of TiN or CrN, the hollow cathode discharge process was employed in vacuum to generate a glow discharge in nitrogen into which titanium or chromium was evaporated at a constant substrate temperature of 623K for TiN and 723K for CrN. The thickness of the TiN-coating film was about 3 µm and Vickers hardness was Hv(15gf)=1888. Knoop hardness of CrN film was about HK 1900-2000. In CVD coating of TiN, specimens were inserted into a stream of mixed gases (H₂:N₂:TiCl₄=32.8 :65.6:1.6[vol.%]) under a reduced pressure of 3.33x10³Pa at 1223K for 3 h. Thickness and Vickers hardness of the CVD-coated layer were 5-6 µm and Hv(50gf)=2170, respectively.

3 High cycle fatigue behavior in air

Experimental results obtained by the cantilever-type rotating-bending fatigue tests in air at testing frequency of 30 Hz, using the smooth specimens coated with TiN by PVD and CVD methods are summarized in Fig.1. Results obtained using the specimens annealed at 1228K for 3 h as same as heat treatment of CVD process are also shown in Fig.1(a), in order to discuss the effect of structural change by coating condition on fatigue life. An increase in fatigue strength was obtained in the coating specimens, as compared with that of uncoated specimen. Fatigue limit
of the uncoated specimen normalized is 263 MPa and that annealed 248 MPa. Fatigue limits of TiN-coating specimen using PVD and CVD method are same in each other and are 304 MPa. The improvement in fatigue limit by coating was about 16–23%. The same result was obtained from the specimen coated by CrN.

Because the CVD coating was treated at high temperature as compared with PVD coating, microstructural change of substrate will occur during deposition process. Figure 2 shows the experimental relationship between Vickers hardness and distance from substrate surface to center of specimen coated with TiN by CVD and PVD. It can be seen from this figure that the hardness distribution of PVD-coating specimen does not changed but that of CVD-coating specimen decreases below coating film as compared with normalized specimen. This is due to the decarburization of the substrate near the CVD-coated TiN layer. From X-ray diffraction pattern of the CVD-coating film, it was suggested that the film is composed of TiN$_x$Cy formed by diffusion of carbon during deposition process [2]. And also, by observation using an optical microscope, the ferrite and pearlite structure of the CVD-coated specimen was formed to be larger than that of PVD-coated one [3]. Therefore, it is noticed for an evaluation of fatigue strength on coating materials to consider the structural change in substrate during deposition process.

It has been sometimes pointed out that tensile residual stresses on the substrate near the coating film generated by the balance of compressive residual stresses in the coating film may decrease the fatigue strength of the coating materials. In this study, large compressive stresses of about 0.87–1.14 GPa for the CVD-coated TiN film and 1.53–3.65 GPa for the PVD-coated one, which depend on diffraction plane, were obtained by X-ray stress measurement and small tensile residual stress of 0.045 GPa was measured on the substrate surface below the TiN-coating film deposited by PVD method. From the fatigue test results mentioned above, it can be concluded that the tensile residual stresses on the substrate is too small to affect the fatigue strength in this coating specimen.

A mechanism for the increase of fatigue strength in air is that hard surface layer can act as barriers to the egress of dislocations, causing dislocation pile-ups and the formation of crack nuclei beneath the film, and results in stable equilibrium position of dislocation at some distance from the layer. Figure 3 shows the analytical results of the elastic interaction between the edge dislocation and the
surface layer followed by the method of R. Weeks et al.[4]. Repulsive force acts on the edge dislocation in the substrate metal having lower elastic modulus than the film. Recently, it was shown experimentally by T. Matsue et al.[5] that plastic deformation of the substrate during the repeated bending fatigue was restricted by the TiN- or TiC-coating film, from the X-ray study showing that residual stress did not change in the substrate as compared with that of uncoated specimen.

4 Influence of applied stress ratio on fatigue strength

Figure 4 shows the experimental results obtained from the fatigue tests conducted under the condition of a stress ratio of $R(=\frac{\sigma_{\text{max}}}{\sigma_{\text{min}}})=0$, that is a repeated tension, at testing frequency of 20 Hz. It can be seen that the fatigue life of specimens coated with TiN by PVD increased in the region of the stress range below about 390 MPa, as compared with that of the uncoated specimen. On the other hand, fatigue life decreased for the region in the high-stress range. It is speculated that cyclic deformation behavior of the specimen affects the fatigue strength, because the value of the stress range (390 MPa) is similar to the yield stress for the PVD-coated specimen. From the experimental results obtained from the fatigue tests under $R=-1$, fatigue strength of coating specimens increased under all of the stress amplitude levels as compared with that of uncoated specimens [3], in contrast to the experimental results under $R=0$. 

Figure 4: S-N diagram under repeated tensile fatigue, $R=0$, in air.
In order to discuss the effects of stress ratio and stress level, cyclic deformation behavior of the specimen was measured by extensometer. The displacement during the fatigue process increased under testing conditions where $R=0$ and at stress range above 390 MPa, which were the conditions where fatigue strength decreased. On the other hand, under testing conditions where $R=0$ and at stress range below 390 MPa and $R=-1$, which were the conditions where fatigue strength increased, the displacement did not increase. From these results, it is clear that fatigue strength of coated specimens depends on deformation behavior and is closely related to flaws in the coating film, because flaws on the coating film occur at small strain level.

5 Low-cycle fatigue behavior

To make clear the relationship between deformation of coated specimen and fatigue strength, strain controlled low-cycle fatigue tests were conducted in air, using specimens of carbon steel coated with TiN by PVD method. Figure 5 shows the experimental results, where number of cycles to failure, $N_f$, was defined as the cycles which the tensile stress amplitude of the specimen decreases to 3/4 of the maximum value. In this figure, relationship between total strain range, $\Delta \varepsilon_t$, and $N_f$ is plotted, and also elastic strain range, $\Delta \varepsilon_e$, and plastic strain range, $\Delta \varepsilon_p$, obtained from hysteresis loop at $N_f/2$, are plotted against $N_f$.

It can be seen from the $\Delta \varepsilon_t$ - $N_f$ diagram that the fatigue strength of specimens coated with TiN increases in the region of low total strain amplitude below 0.7~0.8%, as compared with that of uncoated specimen. On the other hand, fatigue life decreased for region in high total strain range. This phenomenon is more clearly observed in $\Delta \varepsilon_p$ - $N_f$ diagram. It is speculated that cyclic deformation behavior of specimen affects the fatigue strength, because the value of the total strain range ($\Delta \varepsilon_t=0.7~0.8\%$) corresponds with the transition fatigue life in uncoated specimen.

![Figure 5: Strain amplitude vs. Life curves for specimens coated with TiN by PVD method and uncoated, obtained under low-cycle fatigue tests in air.](image-url)
From the optical observation of specimen surface after fatigue test, many flaws in coating film were perpendicular to the specimen axis and around crack under the testing conditions of high strain amplitude where fatigue strength decreased. The crack propagated on the straight along a flaw in comparison with that of uncoated specimen. On the other hand, any flaws could not be found on the specimen surface tested under low strain amplitude, which is the condition where fatigue strength increased [6]. It is suggested from the experimental evidences that fatigue strength of coating specimens is affected by the flaws of coating film occurred during fatigue process.

In order to discuss the rupture behavior of coating film, static tensile tests of coating specimens were performed in air. Figure 6 shows the experimental relation between flaw density on the coating film and total tensile strain of the specimen, where flaw density was defined as the number of flaws per millimeter along the axial direction. It was found that flaws on PVD coating films occurred at the total tensile strains of 0.34--0.40%, and that they increased with the strain. It is interest that the flaw initiation strain at tensile test coincides with the strain amplitude, $A \varepsilon_{1/2} = 0.35$--0.40%, under the low-cycle fatigue test, which is the condition where the reversal of fatigue strength occurs.

The fatigue crack initiation mechanism of coated material can be summarized as in Fig. 7. Under testing conditions where large deformation occurs or accumulates during fatigue, for examples, when high strain amplitude is applied under low-cycle fatigue tests or when $R=0$ and with high stress amplitude, TiN coating film is fractured at an early stage of the fatigue process, because it is too brittle to accommodate the substrate metal. Thus many cracks may be induced to initiate at the substrate by flaws of the coating film (Fig. 7(a)). On the other hand, under testing conditions without large cyclic deformation of the specimen, crack initiation is delayed by hard coating film on the specimen surface, which act as a barrier to the egress of dislocations (Fig. 7(b)).
6 Effect of flaws in coating film on fatigue strength

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. Cantilever-type rotating-bending fatigue tests were conducted in air using specimens of carbon steel with flaws artificially induced in 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.

Figure 8 shows the S-N curves 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 specimens are also shown for comparison. 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. Value of $N_f/N_{fu}$, where $N_f$ is the fatigue life of the specimens with preflawed coating film and $N_{fu}$ is that of the uncoated specimen, is about 0.1 independent of stress amplitude for the PVD-coated specimen, whereas it varies between 0.1~0.25 depending on stress amplitude for the CVD-coated specimen.

![Figure 8: S-N diagram for fatigue test in air using specimens with preflawed TiN coating film.](image)

Preflaws in the coating film have the same effect as notches for crack initiation on substrates and decrease the fatigue life, even though the film thickness is 3~5 μm which is quite shallow compared to a notch. The fatigue life, $N_f$, is controlled by the crack initiation life, $N_i$, and 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. The experimental finding in this study that 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 was explained by predicting the fatigue life using the small fatigue crack growth law [7]. As the results, it was estimated 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. 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 fatigue process.

7 Corrosion fatigue behavior

Because ceramics is a chemically stable compound, it is expected to increase a corrosion fatigue strength of substrate material by coating. Cantilever-type rotating-bending fatigue tests were conducted in 3.0% NaCl aqueous solution environment at testing frequency of about 30 Hz. The corrodent controlled at 298 ± 2K was continuously circulated in a plastic reservoir through the tank at a flow rate of about 32 ml/min.

Figure 9 shows the experimental results obtained using specimens coated with TiN by PVD and CVD methods, and with CrN by PVD method. Any differences of corrosion fatigue strength between normalized and annealed substrate metals can not be observed as shown in Fig.9(a). Obvious increase in corrosion fatigue
strength can be seen on the PVD- and CVD-coating specimens as compared with that of uncoated specimen. Improvement in corrosion fatigue strength at $10^7$ cycles is 87% for the specimen with PVD-coating TiN film and 126% for that with CVD-coating TiN film. Figure 9(b) shows S-N curves obtained using specimens coated with CrN by PVD method. In this figure, four S-N curves were plotted in order to discuss the effect of coating film thickness on corrosion fatigue strength. Corrosion fatigue strength increases with increasing the film thickness, except 1.5 μm thick coating film, but a significant difference does not find between 5 and 10 μm coating film. Fatigue strength at $10^7$ cycles is 84 MPa for uncoated specimen, 131MPa for 3 μm coating one, and 179 MPa for 5 and 10 μm coating one. Improvement of corrosion fatigue strength at $10^7$ cycles with coating was 56% and 112%, respectively.

Figure 10: Size distribution of pinhole defects in PVD coated CrN film formed during deposition process.

Fracture surface observation of uncoated specimens by SEM 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 coated 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 film protects substrate surface to generate corrosion pits. Small corrosion pits were initiated on the substrate surface below TiN or CrN coating film during corrosion fatigue. The corrosion pits are likely to form on the substrate metal due to saline solution penetrating throughout small pores and pinholes in the coating film, which are produced during coating process and act as a small anode area which accelerated the corrosion of substrate metal.

Figure 10 shows a typical example of defect size distribution in the CrN coating film obtained by SEM observation. There are a lot of small defects in the coating film, in which the greater part is under 5 μm diameter and a few is over 20 μm. These small defects are depended on the thickness of coating film and a number of small defects decreased with increasing film thickness. Therefore, it is pointed out that corrosion fatigue strength of coating metal is controlled by the defects in the coating.
8 Concluding remarks

Effect of ceramics coating film deposited by PVD and CVD methods on the fatigue behavior of metal was discussed based on some experimental results, in order to apply the ceramics coatings to various kinds of machine components and structures which require high functional characteristics, as one of a variety of techniques for surface improvement.

The fatigue life of TiN- or CrN-coated carbon steel was greater in air and in 3% saline solution as compared with that of an uncoated specimen, because that a hard coating layer well-adhered to a substrate material affects the mechanisms of plastic deformation and crack initiation during the fatigue process, and then improves crack initiation life in air and protects the substrate from a corrosive environment.

The influence of the applied stress ratio on the high cycle fatigue strength and of strain amplitude on low-cycle fatigue strength were discussed, and it was clarified that fatigue life is affected by the fracture behavior of the coating film on the specimen surface. Under a testing condition where large deformation occurs or accumulates during fatigue, coating film is fractured at an early stage of the fatigue process because it is too brittle to accommodate the deformation of substrate metal. Flaws in coating film induce the initiation of fatigue cracks on the substrate. Decrease in fatigue life in air caused by preflaws in the coating film induced artificially 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.

The improvement of corrosion fatigue strength depended on the thickness of coating film, because small pores and pinholes in the coating were produced during coating process and these defects decreased with increasing film thickness. A decrease in the number of the defects formed during deposition is required for improvement of corrosion resistance and corrosion fatigue strength of coating materials.

References