# The effect of TiN film on low-cycle fatigue behavior of steel coated by PVD method K. Shiozawa, L. Han

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# ABSTRACT

Strain controlled low-cycle fatigue test was conducted in air, using the specimen of 0.37% carbon steel coated with TiN by PVD method. Increase in fatigue life of coated specimen was observed under the region of low strain amplitude, below  $\Delta \varepsilon_{\pm}=0.35\sim0.40\%$ as compared with that of uncoated specimen. On the other hand, fatigue life of coated specimen decreased at the region of high strain amplitude, for reasons of many cracks induced to initiate at the substrate by rupture of coating film.

## INTRODUCTION

The production of thin layers of titanium nitride (TiN) on the surfaces 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 vapour deposition (PVD) or chemical vapour deposition (CVD) method, that is, high hardness, good 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-errosion resistance.

Another interesting application of hard thin film coated on metals will be to improve the fatigue strength of metals under various kinds of environment. Hard coating layer adhered well on

the substrate material will affect to the mechanisms of plastic deformation and crack initiation during fatigue process. But so far there is very little information available about the effect of coating film on the fatigue behavior of metals.

As one of the series of study on the influence of hard thin film on the fatigue strength of metals [1,2], strain controlled low-cycle fatigue test was conducted in air, using specimens of 0.37% carbon steel coated with TiN by PVD method. In this study, the dependency of strain amplitude on fatigue strength of coated materials was tried to explain by the experimental observation of specimen surface and fracture surface, and by the relationship between flaws occured during static tensile test and crack initiation behavior in fatigue process.

## EXPERIMENTAL PROCEDURE

#### Testing Material and Coating Condition

The substrate metal used in this investigation was 0.37% carbon steel, JIS S35C, normalized at 1138K for 30min. The chemical compositions of this steel is 0.37%C, 0.24%Si, 0.77%Mn, 0.019%P, 0.023%S, 0.1%Cu, 0.2%N and 0.4%Cr. Specimens for fatigue test were smooth round bars with a 8mm diameter and 8mm gauge length. Before TiN deposition, the substrate was polished with emery paper (grade #2000) and electropolished to a depth of about  $15\,\mu$  m.

TiN coating was deposited onto the specimen surface by use of PVD process. The hollow cathode discharge process was employed in vacuum to generate a glow discharge in nitrogen into which Ti was evaporated at a constant substrate temperature of Its thickness was  $2 \sim 3 \,\mu$  m and Vickers' hardness was Hv 623K. (15gf) 1888. It was suggested from the evaluation of X-ray diffraction patterns that the  $Ti_1N_1$  is formed on the PVD-coated layer which showed a goldish yellow colour. From the comparison with the actually measured and theoretical diffraction intensities of TiN film by X-ray, it was found to have (111) orientation components of texture in thin film.

#### Testing Method

Total strain controlled low-cycle fatigue tests were performed in air at room temperature using the electro-hydraulic fatigue testing machine. Strain wave shape was triangular with strain ratio of R (= $\varepsilon_{min}/\varepsilon_{max}$ )=-1 and equal-loading and reverseloading strain rate is 0.5%/sec.

# EXPERIMENTAL RESULTS AND DISCUSSION

#### **Results of Fatigue Tests**

Figure 1 shows the experimental results obtained from the low-

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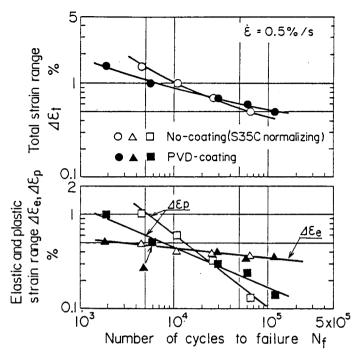


Figure 1 Strain amplitude-life curves for specimens coated with TiN by PVD method and uncoated obtained under low-cycle fatigue tests in air.

cycle fatigue tests under conditions of controlled total strain in air. Here number of cycles to failure, N<sub>t</sub>, was defined as the cycles at which the tensile stress amplitude of the specimen decreases to 3/4 of the maximum value. In this figure, relationship between total strain range,  $\Delta \varepsilon_{\pm}$ , and N<sub>t</sub> is plotted, and also the two strain range, one for elastic  $\Delta \varepsilon_{\pm}$  and one for plastic  $\Delta \varepsilon_{\pm}$  obtained from the hysteresis loops at N<sub>t</sub>/2, are plotted with N<sub>t</sub>.

It can be seen from the  $\Delta \varepsilon_{\pm}-N_t$  diagram that the fatigue life of specimens coated with TiN increased in the region of low total strain amplitude below  $\Delta \varepsilon_{\pm}=0.7\sim0.8\%$ , as compared with that of the uncoated one. On the other hand, fatigue life decreased for the region in high total strain range. This phenomenon is more clearly observed in  $\Delta \varepsilon_{\pm}-N_t$  diagram. It is speculated that cyclic deformation behavior of specimen affects the fatigue strength, because the value of the total strain range ( $\Delta \varepsilon_{\pm}=0.7\sim0.8\%$ ) corresponds with the transition fatigue life,N<sub>t</sub>, where  $\Delta \varepsilon_{\pm}=\Delta \varepsilon$ , in uncoated specimen. That is, at short lives, less than N<sub>t</sub>, plastic strain predominates, and at longer

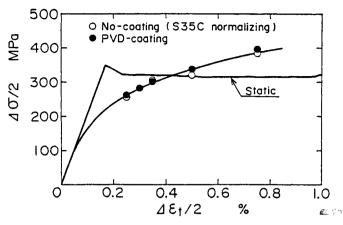
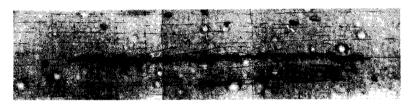
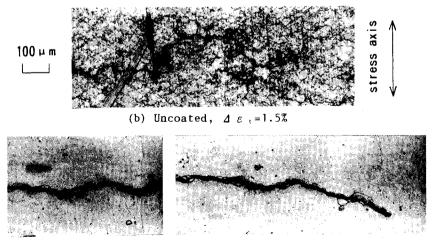


Figure 2 Experimental results of cyclic stress-strain curves compared with monotonic behavior.



(a) Coated by PVD,  $\Delta \varepsilon_{\pm} = 1.5\%$ 



Center part

Crack front

(c) Coated by PVD,  $\Delta \varepsilon_{\pm}=0.5\%$ 

Figure 3 Typical examples of surface cracks and flaws on TiN coating film obtained from optical microscope.

the plastic. The results of observation of flaws in coating films due to plastic deformation will be described later.

Figure 2 shows the cyclic stress-strain response compared with tensile stress-strain curve, for coated and uncoated specimen. It can be seen from this figure that cyclic and monotonic stress-strain relation of coated specimen is coincident with those of uncoated specimen. Therefore, it is suggested that increase or decrease of fatigue life in coated specimen will be controlled by performance of specimen surface.

#### Observation of Specimen Surface

Figure 3 shows the results obtained from the observation of replicas which were taken from the specimen surface after fatigue test. In testing condition of  $\Delta \varepsilon_{1} = 1.5\%$  where fatigue strength decreased, many flaws in coating film are perpendicular

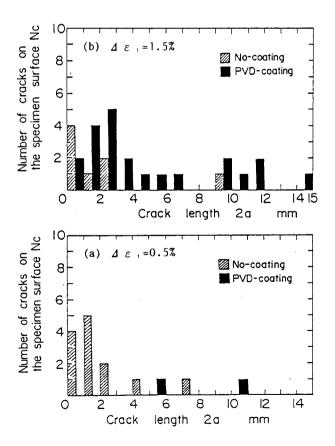


Figure 4 Experimental relationship between number of surface cracks on the specimen surface and crack length.

to the specimen axis, and are around the crack (Figure (a)). Crack propagates on the straight along a flaw in comparison with that of uncoated specimen as shown in Figure (b). On the other hand, any flaws can not be found on the specimen surface tested under  $\Delta \varepsilon_{\pm}=0.5\%$  where fatigue strength increased (Figure (c)).

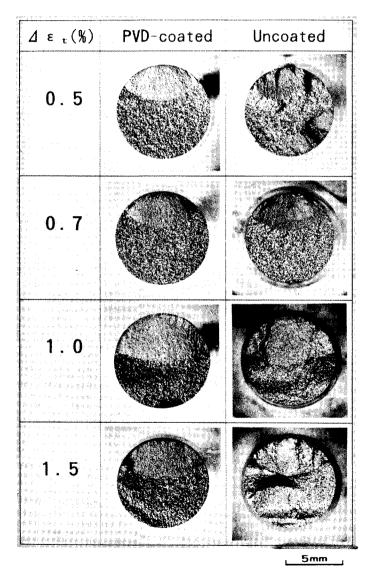


Figure 5 Macroscopic observation of the fracture surface of the coated and uncoated specimens.

Many small cracks were observed on the specimen surface In order to discuss the effect of coated after fatigue test. thin film on fatigue crack initiation, number of surface cracks on the specimen surface of 8mm gauge length were counted by optical microscope of the replicas which were taken from the Figure 4 shows the relationship between number of surface. Under the testing condition of high cracks and crack length. strain amplitude level,  $\Delta \varepsilon_{\pm} = 1.5\%$ , the number of small cracks on the specimen surface of coated material is larger than that of the uncoated one. On the other hand, the number of cracks is less than that of the uncoated specimen under the testing condition of  $\Delta \varepsilon_{\perp}=0.5\%$ . In this case, it is suggested that initiation of crack is constrained by hard coating film on the specimen surface, which can act as a barrier to the plastic deformation around the substrate surface. Also, it is considered that the number of surface cracks is correlated with the behavior of the rupture of coating film.

#### Fractography

Figure 5 shows macroscopic observations of the fracture surface of the tested specimen. It can be seen from this figure that fracture surface of coated specimens is to be smooth as compared with that of uncoated specimens. But from the observation in detail, the number of ratchet marks on the fracture surface of the coated material under the condition of decreased fatigue life is larger than that of the uncoated specimen, which are the result of multiple fatigue crack origins. On the other hand, this number was less than that of the uncoated specimen, under the condition of increased fatigue life. Figure 6 is the typical example of the observation by SEM around the fatigue crack origin of coated specimen tested under  $\Delta \varepsilon_1 = 1.0\%$ .

From the observation of fracture surface of the specimen, the mechanisms of fatigue crack initiation and propagation of coated specimen with flaws of thin film can be summarized as in Figure 7. Many cracks will initiate at the substrate along the



Figure 6 Example of the SEM observation around fatigue crack origin of coated specimen tested under  $\Delta \varepsilon_{\pm}$ =1.0%.

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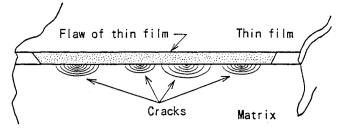


Figure 7 Schematic illustration of crack initiation at the substrate induced by rupture of coating film during fatigue process.

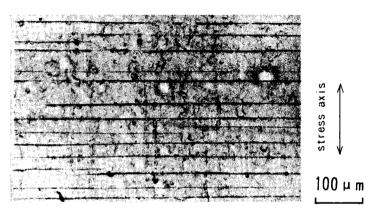


Figure 8 Typical example of flaws on TiN coating film during static tensile test ( $\varepsilon = 2.0\%$ ).

straight flaw of the coating film, which may act as a notch effect, and these cracks propagate independently and coalesce at an early stage of fatigue process.

Rupture of Coating Film under Static Tensile Test

From the experimental results of low-cycle fatigue test, it is suggested that fatigue life is affected by the flaw of coating film. In order to discuss the rupture behavior of coating film, static tensile tests of coated specimens were performed in air. Figure 8 shows the flaws on the coating film obtained from the observation by optical microscope of the replicas which were taken from the surface at various strains during the tensile test. Flaws of the coating film were straight and perpendicular to the stress axis, and its morphological aspect is same as the results of fatigue test.

Figure 9 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 coating film occurred at the total tensile strain of  $0.34 \sim 0.40\%$  and that they increased with the strain.

It is interest that the flaw initiation strain at tensile test ( $\varepsilon_{+}=0.34\sim0.40\%$ ) coincides with the strain amplitude  $\Delta_{-}\varepsilon_{+}/2=0.35\sim0.4\%$  where the reversal of fatigue life occurs. Under testing conditions where large deformation occurs, 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. 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 can act as a barrier to the egress of dislocations. The fatigue crack initiation mechanism of coated material was summarized as in Figure 10.

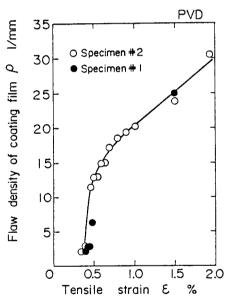


Figure 9 Experimental relationship between flaw density on TiN coating film and tensile strain.

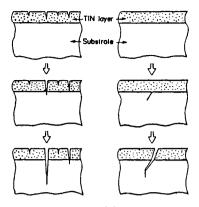
#### CONCLUSIONS

The following conclusions were obtained from low-cycle fatigue tests using specimens of carbon steel coated with TiN by PVD.

(1) The fatigue life of specimens coated was increased in the region of low strain amplitude below  $\Delta \varepsilon$ ,  $2 = 0.35 \sim 0.40\%$  as

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(a) High strain range ∠ ε,>0.7~0.8%

(b)Low strain range Δε.<0.7~0.8%

Figure 10 Schematic illustration of crack initiation of TiNcoated material during fatigue process.

compared with that of the uncoated one. On the other hand, for the region in high total strain range fatigue life decreased.

(2) From the static tensile tests, flaws on coating film occured at the tensile strain of  $0.34 \sim 0.40\%$  and flaw density increased with the strain.

(3) Decrease in fatigue life of coated specimens is due to the crack initiation in the substrate induced by rupture of coating film, which can act as a notch effect.

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