Diagnostic Technique for Deterioration Inspection of Metallic Products through the *In situ* Measurement Using a Thermophysical Handy Tester*

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Abstract

This paper proposes a non-destructive technique using a thermophysical handy tester to inspect the deterioration of metallic products. In the early stage of fatigue in a metallic body, many micro-cracks appear on the surface of the stressed body. As fatigue progresses, these micro-cracks multiply and grow, while the apparent thermal conductivity of the surface layer spontaneously decreases. This phenomenon introduces the possibility of determining the progress of deterioration through *in situ* measurement of thermophysical properties. To estimate the degree of deterioration of materials, a dimensionless deterioration factor $\alpha$ is introduced. In order to corroborate this technique, several fatigue tests using carbon steels were conducted herein. Throughout the tests, apparent thermal conductivities and deterioration factors up to the limit of fatigue were periodically measured using a thermophysical handy tester. Furthermore, microscopic observations to investigate the evolution of micro-cracks were performed for stage-assessment during the period of fatigue tests. The results clearly demonstrate that this technique is useful for the non-destructive diagnosis of such deterioration.

Key words: Deterioration Inspection, *In situ* Measurement, Non-Destructive Inspection, Thermophysical Handy Tester, Thermal Conductivity

1. Introduction

Accurate estimation of component life is an important factor in the safe management of facilities. For the purpose of maintenance, non-destructive inspection methods must be employed in the detection of flaws and cracks. Therefore, diagnostic technology which allows easy *in situ* measurement is required. There are many methods to evaluate the deterioration of engineering materials. Especially in the inspection of cracks at a microscopic level, there is X-ray microdiffraction, Mossbauer spectroscopic analysis, ultrasonic spectroscopy, and the electrical potential technique(1). However, the large equipment utilized in these methods is not suitable for *in situ* measurement.

In this study, a diagnostic method using *in situ* measurement of thermophysical properties is proposed. In fracture mechanics, it is known that many micro-cracks occur on the surface of materials due to fatigue and it is thought that these micro-cracks give rise to an increase in thermal resistance. To validate this concept, the influence of micro-cracks on
the thermophysical properties was theoretically evaluated. Fatigue tests were then conducted to validate the theoretical results. From periodic measurements of thermophysical properties throughout the fatigue tests, it was evident that the apparent thermal conductivity of the tested sample decreases and the deterioration factor increases with the degree of fatigue in the early stage.

Nomenclature

\[ a_p : \text{thermal diffusivity of probe, m}^2\text{s}^{-1} \]
\[ b : \text{thermal probe parameter, } b = 2r_0(\eta - 1)(\pi a_p)^{0.5}, s^{-0.5} \]
\[ C, C_1, C_4 : \text{thermal probe parameters} \]
\[ D : \text{parameter in Eq. (2)} \]
\[ N : \text{number of cycles to failure} \]
\[ N_f : \text{fatigue life} \]
\[ r_0 : \text{thermal probe contact radius, m} \]
\[ r_1 : \text{position of temperature sensing point from contact point, m} \]
\[ T_p^* : \text{non-dimensional temperature of thermal probe} \]
\[ t : \text{time, s} \]
\[ \alpha : \text{deterioration factor, } \alpha = (\delta/\lambda_e)(\lambda_e/r_0) \]
\[ \beta : \text{thermal conductivity ratio, } \beta = \lambda_s/\lambda_p \]
\[ \delta : \text{thickness of micro-cracked layer, m} \]
\[ \zeta : \text{thermal effusivity ratio, } \zeta = \xi_s/\xi_p \]
\[ \eta : \text{thermal probe parameter, } \eta = r_1/r_0 \]
\[ \lambda : \text{thermal conductivity, Wm}^{-1}\text{K}^{-1} \]
\[ \xi : \text{thermal effusivity, Jm}^{-2}\text{s}^{-0.5}\text{K}^{-1} \]
\[ \sigma_{\text{max}} : \text{maximum stress in fatigue test, MPa} \]

Subscripts

\[ e : \text{apparent value in micro-cracked layer} \]
\[ p : \text{value of thermal probe} \]
\[ s : \text{value of sample} \]
\[ 0 : \text{initial value} \]

2. Diagnosis through Measured Temperature Response

A thermophysical handy tester has already been developed for the in situ measurement of thermophysical properties\(^2\,3\). The tester was utilized for the quality inspection and non-destructive diagnosis of engineering materials\(^4\). Thermophysical properties are determined from the temperature response obtained by contacting the surface with the preheated probe-tip of the tester.

In this study, the tester was applied for the diagnosis of deterioration. The thermal conductivity measured by the tester is evaluated as a bulk property from the point-contact surface to a depth of several millimeters. Thus, the tester can be used in regions of local stress concentration, and it is expected that this technique will be useful for local deterioration diagnosis.

In fracture mechanics, when a material is subjected to cyclic stress, micro-cracks appear on the surface in the early stage of fatigue. The growth-direction of these micro-cracks is generally inclined by 45 degrees to the load-stress direction. This inclination corresponds to the angle of the maximum shear stress plane. As fatigue progresses, the micro-cracks propagate and also grow. Although most micro-crack growth ceases after the early stage of fatigue, only one crack grows large enough to penetrate grain boundaries of the material.
This crack stretches perpendicular to the direction of the normal stress until fracture occurs. The growth pattern of micro-cracks in crystalline metallic materials is illustrated in Fig. 1. The micro-cracks originate at the surface and extend to the interior of grains, in a process known as fatigue stage I. Further elongation of cracks to sizes larger than the grain size is referred to as stage II.

When the probe-tip makes contact with the test sample, one-dimensional heat flow will occur radially from the contact point to the sample. The micro-cracks which exist in the surface layer will then give rise to increased thermal resistance in that layer. The measured thermal conductivity is an apparent value influenced by the higher thermal resistance layer. It is predictable that the apparent thermal conductivity is lower than that of the surrounding body.

In order to relate the reduction of apparent thermal conductivity \( \lambda_e \) to the degree of deterioration, the thermal contact resistance for the thermophysical handy tester\(^5\) can be theoretically evaluated. Since the micro-cracks only exist in the near-surface layer, the thermal contact conductance \( \lambda_e/\delta \) can be evaluated. In this study, it is proposed to characterize the degree of deterioration by a factor \( \alpha \), which is given by Eq. (1).

\[
\alpha = \left( \frac{\delta}{\lambda_e} \right) \left( \frac{\lambda_p}{r_0} \right)
\]

where \( \lambda_p \) is the thermal conductivity of the probe, \( \lambda_e \) is that of the micro-cracked layer, \( \delta \) is the thickness of the micro-cracked layer, and \( r_0 \) is the effective radius of the contact area. A micro-cracked layer is illustrated in Fig. 2. The right-hand side of Eq. (1) involves the thermal conductance of the micro-cracked layer and a ratio of probe parameters. As fatigue progresses, micro-cracks grow and increasingly multiply. It is expected that if \( \delta \) increases as the fatigue progresses, \( \lambda_e \) will also decrease, resulting in a larger \( \alpha \). Since \( \lambda_e \) and/or \( \delta \) change with the progress of deterioration, the value of \( \alpha \) allows the degree of deterioration to be estimated.

In order to estimate the deterioration from the value of \( \alpha \), it is necessary beforehand to establish the probe parameters \( \eta (= r_1/r_0) \), \( b (= 2r_0(\eta-1)(\pi a_p)^{-0.5}) \), the ratio of thermal conductivity between the sample and the probe \( \beta (= \lambda_s/\lambda_p) \), and that of thermal effusivity \( \zeta (= \xi_s/\xi_p) \), using a sample without cracks. The authors have analyzed the effect of the existence of a micro-cracked layer on both the temperature response and the thermal conductivity. The theoretical temperature response for any \( \alpha \) can be obtained from the following equation\(^6\):

\[
\text{Fig. 1 Growth pattern of micro-cracks in metallic materials}
\]
Fig. 2  Micro-cracks layer occurred on the material surface

\[ T_p^* = \frac{C_2}{\eta} \left\{ \text{erfc} \left( \frac{C}{\sqrt{t}} \right) - \exp \left( \frac{C_1^2 - C_2^2}{t} \right) \text{erfc}(C_1) \right\} + \frac{C_4}{\eta} \left\{ \text{erfc} \left( \frac{C}{\sqrt{t}} \right) - \exp \left( \frac{C_3^2 - C_4^2}{t} \right) \text{erfc}(C_3) \right\} \]

(2)

where:

\[ C = \frac{\sqrt{\pi} b}{2} \]

\[ C_1 = \frac{b\sqrt{\pi} + (\eta - 1) \zeta(\alpha + 1 + \alpha \beta + 1 - D) \sqrt{t}}{2\sqrt{\pi} ba \zeta} \]

\[ C_2 = -\frac{\zeta(\alpha + 1) - \alpha \beta + 1 - D}{D[\zeta(\alpha + 1) + \alpha \beta + 1 - D]} \]

\[ C_3 = \frac{b\sqrt{\pi} + (\eta - 1) \zeta(\alpha + 1 + \alpha \beta + 1 + D) \sqrt{t}}{2\sqrt{\pi} ba \zeta} \]

\[ C_4 = \frac{\zeta(\alpha + 1) + \alpha \beta + 1 + D}{D[\zeta(\alpha + 1) + \alpha \beta + 1 + D]} \]

\[ D = \sqrt{\zeta(\alpha + 1 + \alpha \beta + 1)^2 - 4a \zeta(\beta(\alpha + 1) + 1)} \]

The value of \( \alpha \) is evaluated from the temperature-response curve of a fatigued sample by curve-fitting Eq. (2) with the measured temperature response curve.

In Fig. 3 the results of curve-fitting the theoretical temperature responses from Eq. (2) with the measured ones are shown. The dimensionless temperature \( T_p^* \) is defined as \((T_p - T_{p0})/(T_{s0} - T_{p0})\), where \( T_{p0} \) is the initial probe temperature and \( T_{s0} \) is the initial sample temperature. Additionally, the parameter \( N/N_f \) denotes the ratio of cycles for the fatigue test under the condition of \( \sigma_{\text{max}} = 430.0 \) MPa. It is ascertained from the results that with increasing \( N/N_f \), the temperature response \( T_p^* \) becomes smaller and the value of \( \alpha \) becomes larger. As a result, it can be applied to the diagnosis of fatigue.

3. Measurement of Thermal Conductivity in Fatigue Tests

Figure 4 shows an outline of the fatigue-test specimens. These are 5-mm-thick mirror-finished plates made from carbon steel for machine-structure use, JIS-S45C. These specimens were annealed at 1083 K for 2 hours in a vacuum furnace. The specimens have a centrally located constriction to localize a fracture. The location of the measuring position was selected as shown in Fig. 4, under the consideration of both the stress concentration at
the constriction and the penetration depth of the temperature change after contacting the specimen with the tip of the tester. A fatigue-test machine (E100kN, Shimadzu Corp.) was employed to apply cyclic loads to the specimens. A pulsating tension (stress ratio $R = 0$) was applied at a frequency of 5 Hz.

In order to view clear micro-cracks on the surface of specimens, some low-cycle fatigue tests were conducted. Figure 5 shows the results of the fatigue tests. Some initial fatigue tests were performed to obtain an S-N diagram (circles in Fig. 5), which was used to select the maximum stress levels employed in the tests. Four maximum stresses were selected in the range from 30000 to 130000 of fatigue life. The maximum stresses (triangles in Fig. 5) are determined as 470.6 MPa (level A), 449.0 MPa (level B), 430.0 MPa (level C) and 412.5 MPa (level D). The changes in thermal conductivity as fatigue progressed were determined under these conditions using the thermophysical handy tester.

Figure 6 illustrates the sequence of measuring thermophysical properties over the period of the fatigue test under the stress level B. The sample containing no cracks was measured immediately after mounting the specimen on a fatigue-test machine. After a certain amount of cycles applying stress, the machine was paused for more than 15 minutes to allow dissipation of the heat generated by the cyclic stresses, and the thermal properties of the sample were also measured using the tester. This procedure was repeated until failure occurred. The fatigue life obtained by the usual test was short compared to that acquired by the test with several pauses to measure thermophysical properties. It is supposed that micro-cracks may be healed if the fatigue-test machine is paused.
4. Results of Measurements at Several Stages of Fatigue

Figure 7 shows the change in thermal conductivity over the period of the fatigue test for the case of level A (shown in Fig. 5). The ordinate shows the ratio of thermal conductivity of a cracked sample $\lambda_s$ to that of a sample containing no cracks $\lambda_{s0}$, $\lambda_s/\lambda_{s0}$, while the abscissa shows the ratio of the number of cycles $N$ to the fatigue life $N_f$, $N/N_f$. Since the surface condition has a strong influence on the measured thermophysical properties, the values of thermal conductivity are plotted as averages over 5 measurements at each cycle number. The upper and lower values of the error bars indicate the maximum and minimum measured values, respectively. It is obvious that the value of $\lambda_s/\lambda_{s0}$ decreases with an increase in $N/N_f$ in the range from 0 to 0.4, and then increases with $N/N_f$ over the rest of the range. Figures 8, 9 and 10 show the changes of $\lambda_s/\lambda_{s0}$ with $N/N_f$ for stress levels B, C and D, respectively. From these results, it was ascertained that the results of measurements under different $\sigma_{\text{max}}$ values (e.g. 449.0 MPa, 430.0 MPa and 412.5 MPa) clearly exhibit the same trend. Additionally, the greater the value of $\sigma_{\text{max}}$, the larger the decrease in $\lambda_s/\lambda_{s0}$. Since the specimen was maintained vertically on the fatigue test machine, the sliding direction of the probe in the tester was horizontal. It is considered that the measurement errors of $\lambda_s$ were caused by the deflection of the probe when sliding, as there was not enough clearance.
Fig. 7  Change in $\lambda_s/\lambda_{s0}$ for the period of the fatigue test at the level A

Fig. 8  Change in $\lambda_s/\lambda_{s0}$ for the period of the fatigue test at the level B

Fig. 9  Change in $\lambda_s/\lambda_{s0}$ for the period of the fatigue test at the level C
Figure 10 shows the change in the deterioration factor $\alpha$ at the stress level A over the period of the fatigue test. The values of $\alpha$ are evaluated by curve fitting to the measured temperature responses. Figures 12, 13, and 14 show the change in $\alpha$ for the stress levels B, C and D, respectively. From these results, it was ascertained that the values of $\alpha$ increase with increasing $N/N_f$ reach a maximum at about $N/N_f = 0.4$, then begin to decrease for $N/N_f > 0.4$. It is also revealed that the maximum values of $\alpha$ increase with increasing $\sigma_{\text{max}}$. The reason why $\alpha$ begins to decrease from the middle stage of fatigue may be that most of the cracks are healed for a period of more than 0.4, while the micro-cracks grow as fatigue progresses until $N/N_f = 0.4$.

Consequently, it is possible that a residual-life prediction can be made from the determination of the value of $\alpha$. That is, the residual life of the material may be 60% when the value of $\lambda_s$ becomes a minimum or $\alpha$ becomes a maximum.

Although the values of $\lambda_s$ and $\alpha$ vary by contraries with the degree of fatigue, it is not necessarily the case that the ratio of cycles at which the value of $\alpha$ becomes maximum is identical with that at which the value of $\lambda_s$ minimum under the same stress level. The value of $\alpha$ including the thermal conductance of the micro-cracked layer is much better at diagnosis of deterioration by using the thermophysical handy tester than the value of $\lambda_s$.
5. Microscopic Observation of Specimens at Specific Stages of Fatigue Tests

In order to investigate the growth of micro- and macro-cracks, microscopic observations of the surface of a specimen were performed. Since it was difficult to observe the surfaces of specimens remaining on the fatigue-test machine, four specimens were prepared and tested under the same stress level, but using different number of cycles; $\sigma_{\text{max}} = 449.0$ MPa, $N/N_f$ are 0.10, 0.41, 0.59 and 0.79. The microscopic observations were made at
the positions shown in Fig. 15. A macro-crack, which is the cause of fracture, appeared at the constriction of the specimen. Micro-cracks and a single macro-crack were observed on the surface of the specimens after the fatigue tests.

The micrographs shown in Fig. 16 are of the surface at the measuring positions for $N/N_f = 0.10$ and 0.41. The traces which resemble wrinkles in Fig. 16 are micro-cracks, which are seen to overlap each other. It is observed in these photographs that the density of micro-cracks increases with increasing $N/N_f$ in the early stage of fatigue. Therefore, the growth of micro-cracks causes an increase in $\alpha$ in the early stage of fatigue.

Micrographs from the region of the constriction are shown in Fig. 17. From these images, no macro-cracks were observed for $N/N_f = 0.41$, but a macro-crack appeared at $N/N_f = 0.59$. The turning point where $\alpha$ begins to decrease corresponds to the appearance of the macro-crack in the latter stage of fatigue. Moreover, it is presumed that the healing of micro-cracks may begin taking place just after the formation of the macro-crack; the size of micro-cracks may shrink in the latter stage of fatigue due to stress-focusing(7). As a result, recovery of the apparent thermal conductivity in the micro-crack layer occurs and leads to a decrease in $\alpha$.

![Fig. 15 Observing positions of the fatigue specimen](image)

![Fig. 16 Microscopic views of the surface of the measuring position](image)
6. Conclusions

An innovative non-destructive diagnostic technique for inspecting the deterioration of metallic products due to the existence of a micro-cracked layer has been presented herein. This technique involves the use of a thermophysical tester. In order to evaluate the degree of deterioration, a deterioration factor $\alpha$ was proposed. Fatigue tests of metallic specimens and periodic measurements of the thermal conductivities thereof were performed. From the experimental results, a number of useful conclusions may be drawn as follows:

1. In the early stage of fatigue, the apparent thermal conductivity of the surface layer decreases and the value of $\alpha$ increases as fatigue progresses.
2. In the middle stage of fatigue, the value of apparent thermal conductivity reaches a minimum and the value of $\alpha$ reaches a maximum. They do not necessarily correspond with each other in the ratio of cycles.
3. The decrease of the thermal conductivity tends to speed up as the maximum stress $\sigma_{\text{max}}$ increases, and the increase of $\alpha$ also tends to speed up as $\sigma_{\text{max}}$ increases.
4. The value of $\alpha$ including the thermal conductance of the micro-cracked layer is much better at diagnosis of deterioration than the value of apparent thermal conductivity.
5. By detecting the turning point of change in the value of $\alpha$, it may be possible to predict the residual life of the testing material.

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References


