DC Potential Drop Method for Evaluating Material Degradation

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The remaining life estimation for the aged components in power plants as well as chemical plants are very important because mechanical properties of the components are degraded with in-service exposure time in high temperatures. Since it is difficult to take specimens from the operating components to evaluate mechanical properties of components, nondestructive techniques are needed to evaluate the degradation. In this study, test materials with several different degradation levels were prepared by isothermal aging heat treatment at 630℃. The DC potential drop method and destructive methods such as tensile and fracture toughness were used in order to evaluate the degradation of 1Cr-1Mo-0.25V steels. In this result, we can see that tensile strength and fracture toughness can be calculated from resistivity and it is possible to evaluate material degradation using DC potential drop method, non-destructive method.

Key Words : DC Potential Drop Method, Resistivity, Degradation, Four-Point Probe, Fracture Toughness, Tensile Strength

Nomenclature

V : Electrical potential
I : Current
ρ : Resistivity
r : Distance for electrode
t : Thickness of the specimen
t_1 : Aging time at 538℃
t_2 : Aging time at 630℃
T_1 : Temperature (538℃)
T_2 : Temperature (630℃)
Q : Activation energy
R : Gas constant
k : Correction factor of shape specimen
K_Q : Fracture toughness

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1. Introduction

Material degradation can be observed in deterioration of mechanical properties due to the change of the micro-structure of in-service facilities in high temperatures. Because it is a susceptible factor for the safety operation of facilities, it's required to estimate the extent of degradation (Viswanathan and Gehl, 1991 ; Viswanathan and Bruemmer, 1985 ; Nham and Kim, 1998; Jeong*, et al., 2002 ; Yun, et al., 2002).

One of non-destructive methods widely used is DC potential drop method which has a strong point in view of applicability to in-service facili-

ties. In particular, it is known that the resistivity is sensitive to not only the variation of macroscopic structure but also that of the micro-structure due to material degradation. The four-point probe has proven to be a convenient method for measuring resistivity and Valdes (1954) and Uhlir (1955) gave the functional relationship between resistivity and voltage for various geometries. All these treatments were concerned with three dimensional structures infinite in one direction. Later, Smits (1958) proposed the correction factors for resistivity of two-dimensional rectangular and circular samples. However such researches have most used it for measuring the resistivity of the semi-conductor of germanium and the crack growth, but only there are very little papers for applying it to measuring material degradation. Particularly, Seok, et al. (2000) attempted to measure material degradation by DC potential drop method, but it has a limit for an application because they uncertainly defined the relationships between resistivity and material degradation.

In this study, test materials with several different degradation levels were prepared by isothermal aging heat treatment at 630℃. The effects of aging on the mechanical properties of each specimen were investigated by tensile test and fracture toughness test. Then those were compared with the variation of resistivity. The purpose of this study is to evaluate the reliability of DC potential drop method from this result, and clearly define the relationships between resistivity and material degradation and provide a basis for determining the extent of material degradation by DC potential drop method.

2. DC Potential Drop Method

Material degradation can be made by a change of micro-structure due to precipitation of sigma phase and growth of grain size. Hence, it is possible to assess the degradation of material by comparing resistivity of aging material with that of virgin material. In this study, resistivity was gotten on the basis of "Standard Test Method for Measuring Resistivity of Silicon Wafers with an In-Line Four-Point Probe (ASTM B193-87, 1992)"

2.1 Resistivity

The potential is given by Eq. (1) at the distance r from the electrode, when the current I is flowing out through the electrode.

$$V=\frac{\rho I}{2\pi r} \tag{1}$$

where ρ is resistivity.

The basic model for four point probe measurement is shown in Fig. 1. Four probes are placed on a flat surface of the material to be measured. Current is passed through the two outer probes, and the potential is measured across the inner pair (Valdes, 1954; ASTM B193-87, 1992; ASTM F84-93, 1993). For $t \gg s$, namely, a thick sample, the resistivity is computed as follows.

$$\rho=2\pi s\left(\frac{V}{I}\right) \tag{2}$$

For a thin sheet, that is, thickness of the specimen t is smaller than the distance between the neighboring probes ($t \ll s$), the resistivity is as follows.

$$\rho=\frac{\pi t}{\ln 2}\left(\frac{V}{I}\right) \tag{3}$$

2.2 Deviation and correction factor

Equations (2) and (3) are derived from the presumptions for thick and thin specimen respectively, but the real specimen is different. An error for resistivity is actually occurred at measurement. When the distance between the nearest probe and the boundary is l, and if the thickness of specimen is more than l of $4s$ on the semi-infinite solid, Eq.

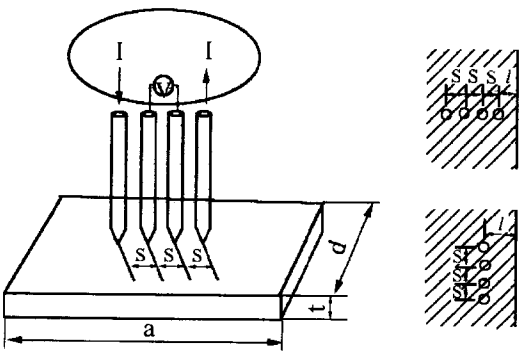

Fig. 1 Configuration of specimen boundary and 4 point probe

(2) can be applied to it. In this case, there is an error of less than 2 percent for measuring the resistivity (ASTM F84-93, 1993). In the same experimental condition, there is also an error of less than 2 percent if the thickness of the specimen is below 0.7s in the case of applying the Eq. (3) (Smits, 1958). So, it require correction factor to get the accuracy resistivity in the range of 0.7s~4s. Then it reduce the deviation of data (Logan, 1967).

Generally, resistivity can be obtained from the following equation.

$$\rho_s = k\left(\frac{V}{I}\right) \tag{4}$$

where ρ_s, k are sheet resistivity and correction factor of the shape of the specimen respectively.

The correction factor k consists with the correction factor F for corresponding thickness of specimen (t/s) and correction factor C for corresponding width of specimen $(d/s, a/d)$ to consider the shape of specimen and probe. F and C are found from referring to Smits' table (Smits, 1958). The probes, which take maintenance over the distance 4s from each edge of the boundaries of the specimen, must be placed at the center of the specimen surface (Shi and Sun, 1997 ; Yamashita, Nishii, and Kurihara, 1996).

3. Experiment

Low-alloy ferritic steel, 1Cr-1Mo-0.25V, is widely used for structural components in high temperature in power plants. The chemical composition of materials is given in Table 1.

3.1 Degradation materials

The present investigation has been carried out using the several classes of the thermally aged 1Cr-1Mo-0.25V steel specimen prepared by artificially accelerated aging method. Degradation time was determined from the self diffusion theory of Fe by Eq. (5) and Eq. (6). D_1 and D_2 of the equations are diffusion coefficients for 538℃ and 630℃, respectively and the degradation time (t_2) for 630℃ is followed as Eq. (7) (Abdel-Latif, et al., 1982 ; Abdel-Latif, et al., 1981).

$$D_1 = D_0 \exp\left[-\frac{Q}{RT_1}\right] = \frac{C}{t_1} \tag{5}$$

$$D_2 = D_0 \exp\left[-\frac{Q}{RT_2}\right] = \frac{C}{t_2} \tag{6}$$

$$t_2 = t_1 \exp\left[-\frac{Q}{R}\left(\frac{1}{T_2}-\frac{1}{T_1}\right)\right] \tag{7}$$

where R is the gas constant (8.314J/kmol/K) and Q is the activation energy (272kJ/mol) for the self diffusion of Fe and T_1, T_2 are degradation temperature and t_1, t_2 are degradation time. Aging was carried out for several different aging times at 630℃. The aging time at 630℃ for equivalent microstructure served at 538℃ are given in Table 2.

3.2 Specimens

Basically, four different class of materials procured by isothermal aging heat-treatment for 0, 453, 933, 1820 hours at 630℃, were prepared. The effect of aging on the mechanical properties of 1Cr-1Mo-0.25V steel has been investigated by using DC potential drop method and compared with the mechanical properties obtained from the tensile test and the fracture toughness test. Additional specimens for two different aging times, that is, 15,000 and 35,000 hours were prepared to consider the trend of resistivity. 3-type specimens were prepared for evaluating the material degradation with varying thickness and shape of specimen, such as type A (thin), type B (thick) and type C in Fig. 2. There are 6 classes of aging specimens for type A and type B and 4 classes of

Table 1 Chemical composition of 1Cr-1Mo-0.25V (Wt. %)

C	Si	Mn	S	P	Ni	Cr	Mo	V	Sn
0.29	0.01	0.74	0.004	0.007	0.060	1.29	1.24	0.25	0.0047

Table 2 Aging time at 630℃ for equivalent microstructure serviced at 538℃

Aging Time at 538℃ (hour)	0	15,000	25,000	35,000	50,000	100,000
Aging Time at 630℃ (hour)	0	273	453	634	933	1,820

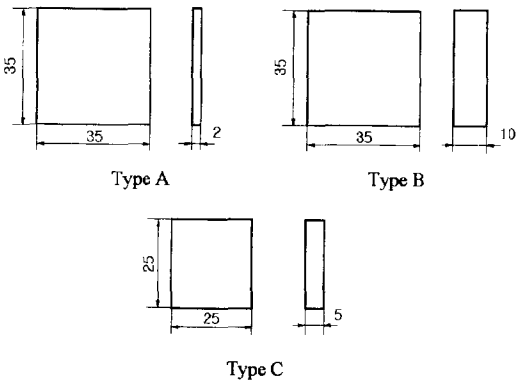

Fig. 2 Shape and dimensions of specimens for electrical resistivity test (unit : mm)

aging specimens for type C.

3.3 Experimental setup and measurement

The instrument for measuring resistivity consists of the four probes, the current meter, the voltage meter and Whiston bridge, which can control the direction of the current. The distance between the probes was 1.59mm.

When measuring for resistivity, it requires that the proper current be supplied because heating at the contact points causes a fluctuation of measurement. In this experiment, the current of 1A was supplied for the specimen and the experimental temperature was maintained at 13.5℃ in the insulation box to eliminate the effect of the external temperature. After getting a stable condition for measuring resistivity, and then the voltage meter and the current meter were fixed on the reference voltage and the reference current set to zero. The data were obtained with measuring for each specimen at 3~4 times and were averaged.

According to ASTM E 8, tensile tests were performed with standard tensile specimens at room temperature. The fracture toughness (K_{IC}) test was carried out using a 25 ton hydraulic dynamic tester according to ASTM E 399. CT typed specimens, *25.4mm* thick, were used. But because the tests didn't satisfy the size requirements for a valid K_{IC}, the results of toughness tests were transcribed as K_Q.

4. Results

4.1 Correction factor

Figure 3 shows the variation of resistivities on degraded time in view of size and thickness. The results for type A, type B and type C were obtained by using Eq. (4) with a correction factor and those for type A1, type B1 and type C1 by using Eqs. (2) and (3) in the raw. On the whole, the resistivities for each case were decreased with increase of degraded time. From Fig. 3, the resistivities without correction factor show dispersion for each case in the raw (type A1, type B1 and type C1). But the resistivities with correction factor (type A, type B and type C) almost are converged to the fixed values. So, it is reasonable to apply the correction factor for calculating the resistivities for each case. Table 3 shows the uncertainty of the resistivities with correction factor for degraded time and varied shape. The uncertainty is to obtain from a standard deviation for the repeatability error and repeatabity is the ability of the device to output the same value for the same displacement over a number of trials.

Figure 4 shows the variation of normalized resistivity on degraded time in view of size and

Table 3 Uncertainty of resistivity

Specimen	Aging time [hour]	Average resistivity [$\mu\Omega cm$]	Uncertainty [%]
Type A	0	21.80	0.113
	15,000	20.96	0.159
	25,000	20.76	0.186
	35,000	20.63	0.066
	50,000	20.57	0.121
	100,000	20.57	0.089
Type B	0	21.46	0.061
	15,000	20.52	0.052
	25,000	20.22	0.100
	35,000	19.91	0.042
	50,000	19.85	0.126
	100,000	19.89	0.211
Type C	0	21.95	0.084
	25,000	20.72	0.019
	50,000	20.45	0.109
	100,000	20.32	0.136

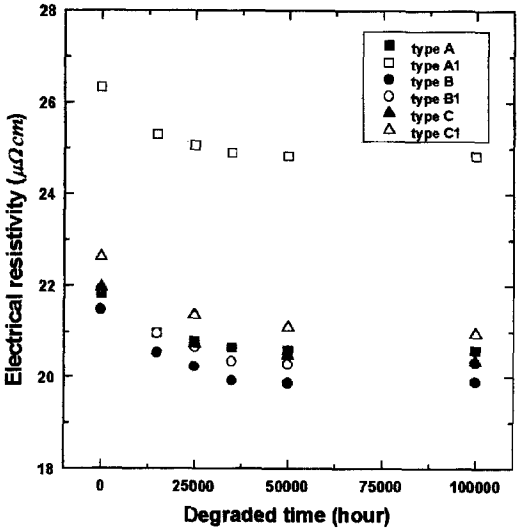

Fig. 3 Effect of degraded time on electrical resistivity

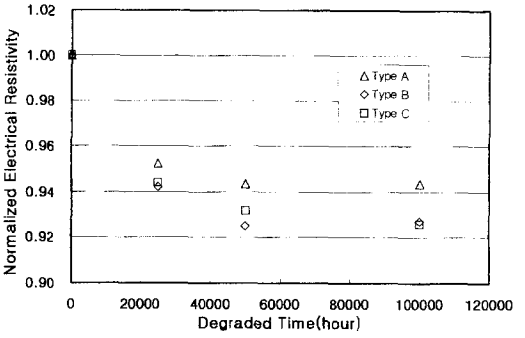

Fig. 4 Effect of normalized electrical resistivity on degraded time for different specimen type

thickness (type A, type B and type C) at in-service temperature, 538℃. Normalized factors divide the values of aged materials by those of virgin. These results show that one agrees well with others within the limits of 2% deviation. Hence, the effect of size and thickness on material degradation can be disregarded using Eq. (4) with a correction factor.

4.2 Tensile and fracture toughness behaviour

Figure 5 shows the effect of normalized factors, i. e., yielding strength, tensile strength of tensile tests, fracture thoughness and the resistivity of type C on degraded time at in-service temperature, 538℃. Normalized factors divide the values of aged materials by those of virgin. Tensile strength, yield strength were similarly decreased with respect to degraded time and resistivity of type C were decreased slightly. Tensile strength, yielding strength and resistivity are not linearly related, but fracture toughness is linearly related with degraded time. That is,

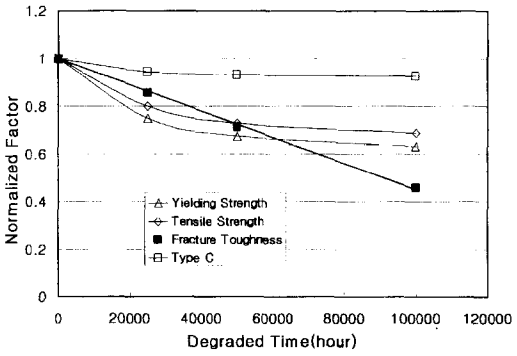

Fig. 5 Effect of normalized factors on degraded time

$$\frac{K_Q}{K_{Q0}} = B_d t + 1 \tag{8}$$

where t is the degraded time and K_{Q0} is fracture toughness of virgin material and $B_d = -5 \times 10^{-6}$.

The decrease of the fracture toughness can be interpreted as the weakness of material due to intergranular brittleness as degraded. Hence fracture toughness is a useful factor for evaluating the degree of material degradation.

4.3 Evaluation for material degradation using DC potential drop method

Figure 6 shows the relation of normalized tensile strength and normalized resistivity. There is the linear interrelation between them as follow.

$$\frac{\sigma_{TS}}{\sigma_{TS0}} = C_d \frac{\rho}{\rho_0} - D_d \tag{9}$$

where σ_{TS0} are tensile strength of virgin material and $C_d = 4.038$ and $D_d = 3.033$ and ρ/ρ_0 is normalized resistivity, the rate of aged resistivity (ρ) divided by the reference resistivity (ρ_0). By using Eq. (9), tensile strength of degraded material of 1Cr-1Mo-0.25V can be obtained from resistivity at in-service temperature, 538℃.

Figure 7 shows the relation of normalized frac-

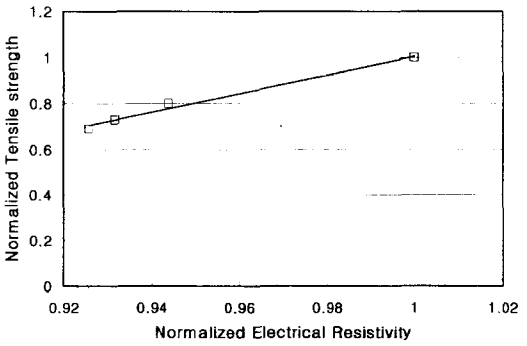

Fig. 6 Relation between normalized tensile strength and normalized electrical resistivity

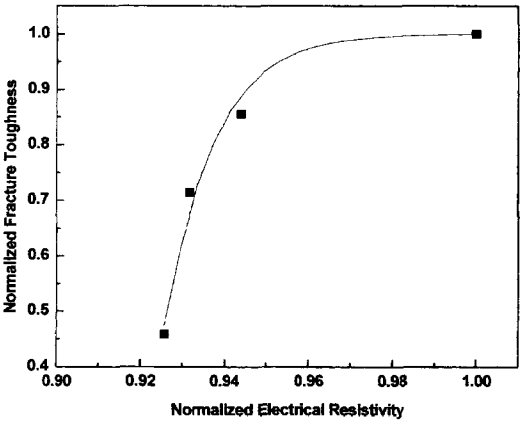

Fig. 7 Relation between normalized fracture toughness and normalized electrical resistivity

ture toughness and normalized resistivity. There is the interrelation between them as follow.

$$\frac{K_Q}{K_{Q0}}=\frac{A_1-A_2}{1+e^{(x-x_0)/dx}}+A_2 \qquad (10)$$

where $x=\rho/\rho_0$ and $x_0=0.903$ and $A_1=-3.748$ and $A_2=1$ and $dx=0.011$. Fracture toughness can be calculated from resistivity by using Eq. (10) in the same condition.

Hence, we can see that it is possible to evaluate material degradation using DC potential drop method.

5. Conclusions

In this study, we prepared for several different classes of simulated specimens of 1Cr-1Mo-0.25V steel by isothermal aging heat-treatment. As comparing various mechanical properties obtained from tensile and fracture test with resistivity, the following conclusions were obtained.

(1) The effect of size and thickness for resistivity of degraded material on degraded time can be disregarded using the equation with a correction factor.
(2) Because fracture toughness is linearly related with degraded time, fracture toughness is a useful factor for evaluating the degree of material degradation.
(3) In the case of 1Cr-1Mo-0.25V steel, tensile strength and fracture toughness can be calculated from resistivity and it is possible to evaluate material degradation using DC potential drop method, non-destructive method.

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