JOURNAL OF MATERIALS SCIENCE **37** (2002) 3549–3553

Evaluation of fracture toughness of degraded Cr-Mo-V steel using electrical resistivity

SEUNG HOON NAHM, YONG IL KIM, KWANG MIN YU
Korea Research Institute of Standards and Science, P.O. Box 102, Yusong, Taejon, 305-600, Korea

AMKEE KIM
Department of Mechanical Engineering, Kongju National University, Kongju, Chungnam, 314-701, Korea

More effective nondestructive technology for the estimation on material properties has been sought. In this research, a new electrical resistivity method was attempted for the estimation of the fracture toughness of a degraded turbine rotor steel. 1Cr-1Mo-0.25V turbine rotor steel specimens with seven different periods of aging were prepared by an isothermal heat treatment at 630°C and the electrical resistivity was determined by a direct current four points potential method. The electrical resistivity at room temperature monotonously decreased with the extent of degradation of the material. It was also observed that the fracture toughness was correlated with the electrical resistivity. The microstructural changes of material during aging was examined by X-ray diffractometer and electron probe micro analyzer (EPMA). A larger amount $Mn_{23}C_6$ and $(Cr_{2.5}Fe_{4.3}Mo_{0.1})C_3$ appeared in grain boundaries of the more heavily aged material.

1. Introduction

When plant equipment is in service for a long period of time, the material suffers aging, and thus its properties change gradually. One of main consequences of aging is material degradation. The material degradation takes place due to long exposure to elevated temperature and corrosive environment combined with loading [1, 2]. In particular heat resistant materials, such as Cr-Mo-V turbine rotor steel, tends to be embrittled by such thermal activation [1]. Once the embrittlement occurs, the fracture toughness, as measured by the ductile brittle transition temperature (DBTT) or critical stress intensity factor (K_{IC}), deteriorates rapidly due to the fracture mode transition from transgranular to intergranular. Thus the continuous monitoring of the fracture toughness is essential for the evaluation of safety or residual life of the turbine rotor. However, the usage of conventional destructive methods such as Charpy impact test is limited since sampling the material for specimens without damage to the equipment is very difficult [3–5].

Monitoring electrical resistivity could be an alternative for the evaluation of the fracture toughness because it is nondestructive and the electrical resistance varies with the change of microstructure [6]. Although the electrical resistivity variation of turbine rotor steel due to the change of microstructure such as the precipitation of carbide, the dissolution of solid element within matrix and the distribution change of macroscopic defects like void or crack is less than a few micro ohm-cm [7], it can be detected by the modern high-resolution measuring devices.

In this research, the effect of isothermal heat treatments, which simulate the microstructural changes observed in turbine rotor steel at the turbine steam temperature, on the electrical resistivity was investigated. The correlation between the measured electrical resistivity and the ductile brittle transition temperature (DBTT) obtained by Charpy impact test was studied. The change in microstructure with the aging was also examined by X-ray diffraction and electron probe micro analysis (EPMA). In addition, the applicability of electrical resistivity measurements to the evaluation of turbine rotor steel fracture toughness was discussed.

2. Experimental

Test material is 1Cr-1Mo-0.25V steel which is widely used as a steam turbine rotor material [8]. The chemical compositions and the mechanical properties are given in Tables I and II, respectively. Virgin (as received) material was artificially aged by the isothermal heat treatment at 630°C for 0, 455, 910, 1365, 1820, 3640, 5460 hours, which is an accelerating aging process for simulating the microstructures of materials in service at the turbine steam temperature of 538°C for about 0, 3, 5, 8, 11, 22, 33 years respectively [9]. Periods of heat treatment for the simulation were selected based on Arrhenius self-diffusion theory with respects to the iron element.

The electrical resistivity of specimens with the seven different heat treatment periods was measured by a direct current four-point potential method at 24 ± 0.5°C.

TABLE I Chemical composition (wt%)

C	Si	Mn	P	S	Ni	Cr	Mo	V	As	Sn	Sb
0.31	0.23	0.76	0.006	0.001	0.36	1.11	1.32	0.27	0.006	0.005	0.001

TABLE II Mechanical properties

Temperature (°C)	Yield strength (MPa)	Tensile strength (MPa)	Elongation (%)	Reduction of area (%)	Hardness (Hv)
24	665.2	823.1	18.8	59.4	266
538	533.5	580.6	22.7	55.5	–

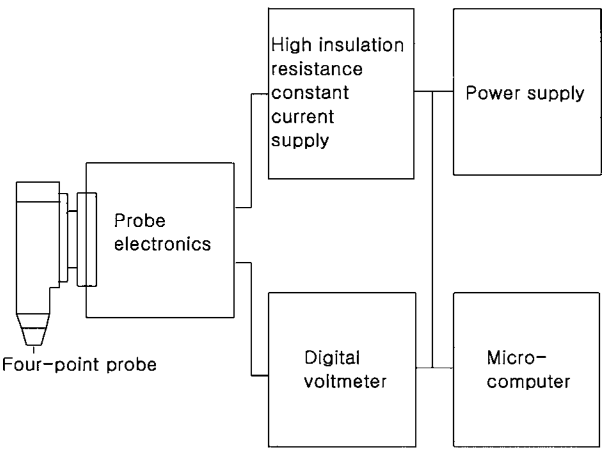

Figure 1 Block diagram for measuring system.

Plane specimens of 55 × 5 × 1 mm (length × width × thickness) were prepared. Four point probes of the measuring system (two for the input and the others for the output) were equally spaced by 1.5 mm and aligned along a line and pressed on a surface of specimen for the measurement. A constant current supplier and the voltage measurement system accommodated by the probe station of Napson RT-8A-8 were utilized for the measurement. Fig. 1 depicts the block diagram of the experimental setup.

The distance between two output probes was carefully measured using a traveling microscope since the measurement error of only 0.05 mm would cause an error of 0.13 $\mu\Omega$ cm. The polarity of input current supplied to the system was altered during the measurement, i.e., forward and backward, in order to reduce the measurement error due to the thermoelectromotive force. An average of output voltages obtained from the forward and backward currents was adopted as a datum of resistivity. A common electrical ground was shared to avoid the common mode noise signal. The electrical resistivities were calculated by following Equation 1 [10].

$$\rho \text{ (electrical resistivity)} = R_s \text{ (area resistance)} \times t \text{ (specimen thickness)} \tag{1}$$

Here $R_s = K_a R_a$. K_a and R_a represent the geometrical correction factor and the electrical resistance between two output probes respectively. Therefore if the correction factor is known (it is 2.86 in this research) [11], the electrical resistivity is calculated by the measured resistance and the thickness of specimen. ASTM standard Charpy impact test was performed in this research as well [12].

3. Results and discussion

3.1. Variation of electrical resistivity and DBTT with aging

Fig. 2 represents the variation of the electrical resistivity normalized by the electrical resistivity (ρ_0) of virgin material. As the period of aging increases, the value of electrical resistivity decreases rapidly down to 92% of that of virgin material and then becomes constant with further aging. In fact any further variation in resistivity is hardly discernable after 100000 hours taking the measurement error of the system into account.

Fig. 3 represents that there is an early rise of DBTT but then it also becomes constant after 50000 hours,

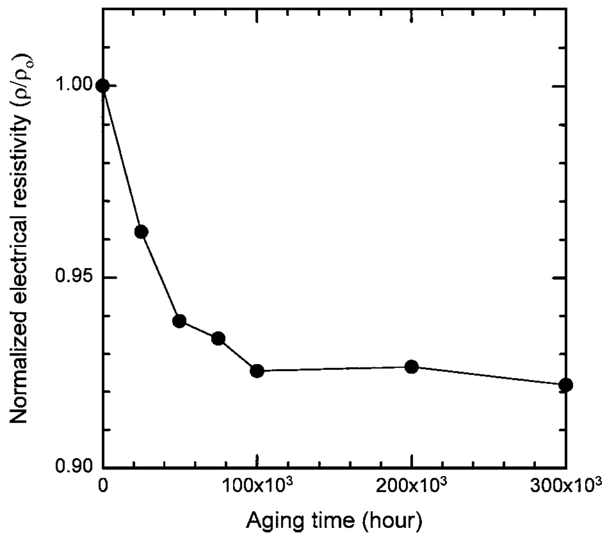

Figure 2 Dependency of electrical resistivity on aging time.

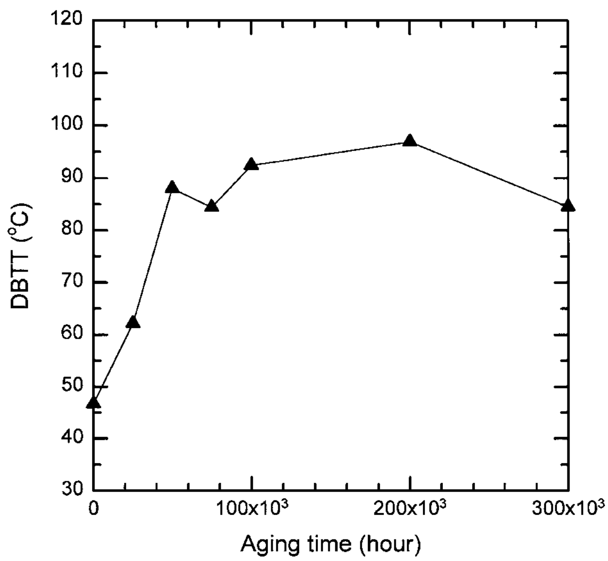

Figure 3 Dependency of DBTT on aging time.

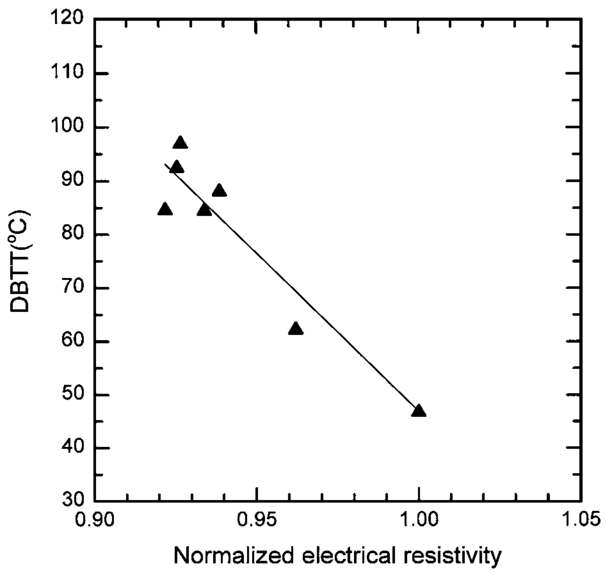

Figure 4 Correlation between electrical resistivity and DBTT.

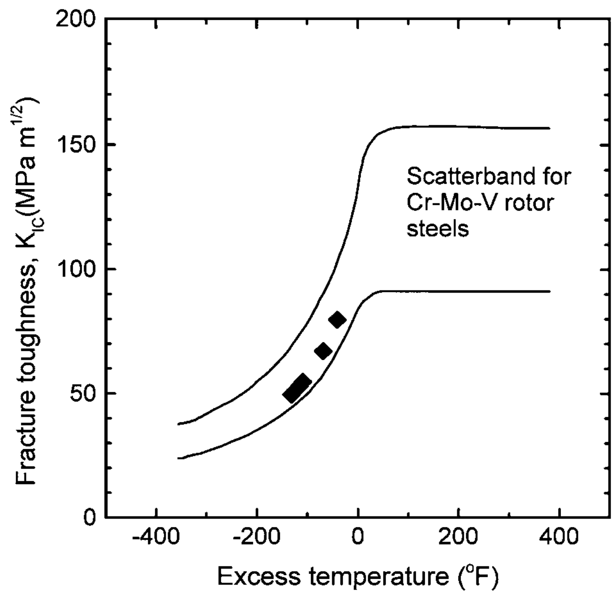

Figure 5 Excess temperature vs. fracture toughness.

which is in agreement with previous results [1]. Thus the electrical resistivity method would be effective for the evaluation of the extent of turbine rotor steel toughness degradation, especially at the early stage of the aging (up to 50000 hours).

3.2. Correlation between electrical resistivity and fracture toughness

Fig. 4 represents the correlation between electrical resistivity and DBTT of degraded turbine rotor steel. The electrical resistivity is linearly correlated with the DBTT. The obtained relationship between electrical resistivity and DBTT is fitted as follows:

$$\text{DBTT} = A(\rho/\rho_0) + B \tag{2}$$

where the fitting parameters are $A = -541°\text{C}$ and $B = 589°\text{C}$. Using the relationship allows one to estimate the DBTT of aged turbine rotor indirectly through the electrical resistivity. However the Equation 2 is only applicable up to 50000 hours because no variation of the DBTT is observed after that time as previously mentioned.

Based on the correlations of other research [13–15] between DBTT and fracture toughness, the fracture toughness, K_{IC}, of the turbine rotor steel can be obtained from the DBTT. The following correlation Equation 3 proposed by Jones was utilized in this research [15].

$$K_{IC} = 10800/[108 - (T - \text{DBTT})] \tag{3}$$

Here, the unit of K_{IC} is ksi in$^{1/2}$ and the unit of test temperature, T, and DBTT is °F.

Fig. 5 represents the fracture toughness, K_{IC}, versus the excess temperature, ($T - \text{DBTT}$). All data points of the turbine rotor steel estimated by using Equations 2 and 3 are inside the scatter band presented by the previous results [1]. It may imply that the electrical resistivity method for the evaluation of fracture toughness is valid at the early stage of the aging up to 50000 hours.

3.3. Microstructural changes of turbine rotor steel with aging

In order to investigate causes of degradation of fracture toughness of turbine rotor steel, scanning electron micrography (SEM) and energy dispersive spectrometry (EDS) were used. More precipitations appear at the grain boundary of the aged material whereas grain boundary precipitates in the virgin material are infrequently observed. In particular, many large precipitates at the grain boundaries of 200000 hours aged material are observed as shown in Fig. 6b. Thus the degradation of fracture toughness may be caused by the change of microstructure due to the precipitation of carbide and the dissolution of solid element within matrix. Table III indicates that Fe content of a precipitate at the grain boundary of aged material measured by EDS is less than that of a grain boundary in the virgin material. Moreover the considerable amounts of Cr, Mn and Mo is observed in the grain boundary precipitate in the aged material, which implies that the carbides were formed at the grain boundary by the carbon which was moved from matrix during the aging. Consequently physical properties like electrical resistivity and fracture toughness might change with the aging.

Fig. 7 represents the result of EPMA analysis on the grain boundary. As the aging increases, the amounts

TABLE III Chemical compositions of the grain boundary of virgin material and the precipitation at the grain boundary of aged material (wt%)

	Grain boundary of virgin material	Precipitation at grain boundary of aged material
Si	0.53	0.57
P	0.22	0.50
Mo	0.84	3.16
V	0.42	1.10
Cr	0.99	9.90
Mn	0.88	5.25
Fe	95.15	78.78
Ni	0.96	0.75

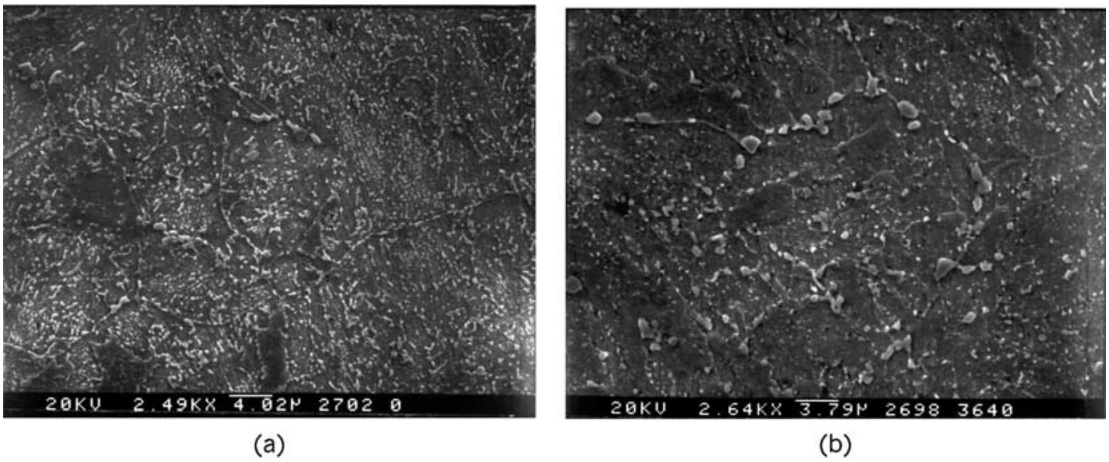

Figure 6 Microstructures of 1 Cr-1Mo-0.25V steel: (a) virgin material, (b) 200000 hours aged material.

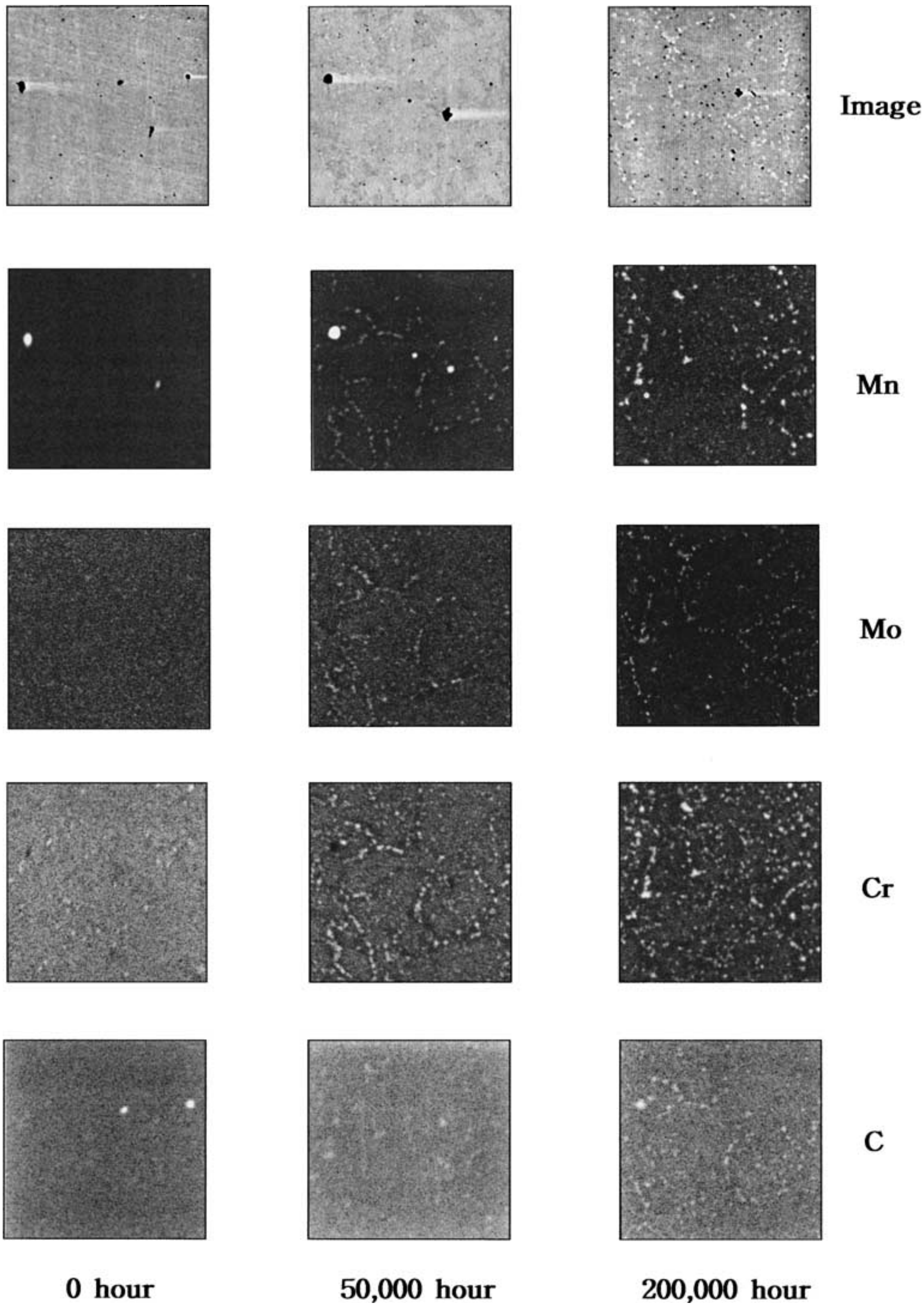

Figure 7 Area mapping for Mn, Mo, Cr and C in 1Cr-1Mo-0.25V steel by EPMA.

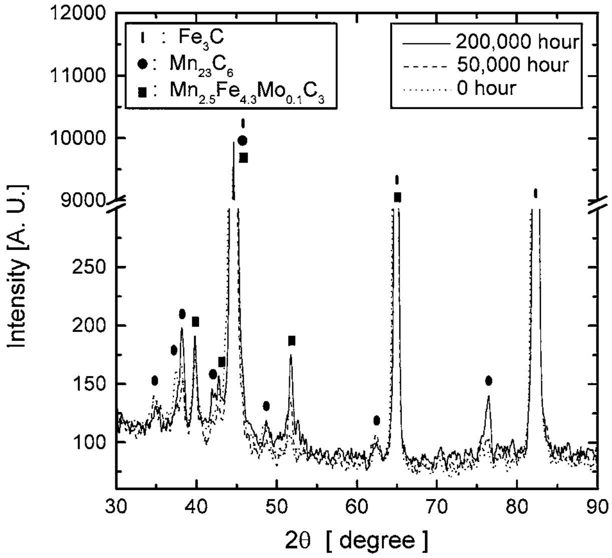

Figure 8 X-ray diffraction pattern of 1Cr-1Mo-0.25V steel.

of Cr, Mn, Mo and C at the grain boundary increase while those of other elements remain unchanged. Fig. 8 demonstrates that the X-ray diffraction (XRD) pattern also depends on the aging. It also indicates the existence of Fe_3C, $Mn_{23}C_6$ and $(Cr_{2.5}Fe_{4.3}Mo_{0.1})C_3$ at the grain boundary. Indeed, the diffraction peaks corresponding to $Mn_{23}C_6$ and $(Cr_{2.5}Fe_{4.3}Mo_{0.1})C_3$ are much more pronounce after 50000 hours. Thus as the amount of the carbide with the covalent bonding increases, the strength of the turbine rotor steel might deteriorate because of the discontinuity in bonding at the interface between carbide and matrix.

4. Conclusions

A nondestructive method for evaluating the fracture toughness of turbine rotor steel was developed using electrical resistivity. The obtained results are summarized as follows:

1. The electrical resistivity decreases with the increment of the aging at the early stage, which comes to be constant after 100000 hours aging time. DBTT increases with the increase of aging time at the early stage. However it also becomes constant after 50000 hours aging time.
2. A linear correlation between the electrical resistivity and the DBTT was established, which allows one to estimate the fracture toughness of turbine rotor steel aged within 50000 hours.
3. As the aging proceeded, more amount of $Mn_{23}C_6$ and $(Cr_{2.5}Fe_{4.3}Mo_{0.1})C_3$ was observed at the grain boundary of the turbine rotor steel.

References

1. R. VISWANATHAN and S. GEHL, *Journal of Engineering Material and Technology* **113** (1991) 263.
2. S. KIRIHARA, M. SHIGA, M. SUKEKAWA, T. YISHIOKA and C. ASANO, *J. Mater. Sci. Jpn.* **33** (1984) 137.
3. S. M. BRUMMER, R. VISWANATHAN and R. H. RICHMAN, in International Conference Life Assessment and Extension (June 1988) p. 92.
4. Y. WATANABE and T. SHOJI, *Metallurgical Transaction* **22A** (1991) 2097.
5. K. M. YU, S. H. NAHM and Y. I. KIM, *J. Mater. Sci. Let.* **18** (1999) 1175.
6. M. SUKEKAWA, *JHPI* **26** (1988) 361.
7. S. H. NAHM, A. KIM and K. M. YU, *Transaction of the Korean Society of Mechanical Engineers* **22** (1998) 814.
8. M. GOJI, L. KOSEC and P. MATKOVI, *J. Mater. Sci.* **33** (1998) 395.
9. A. M. ABDEL-LATIF, J. M. CORBETT, D. SIDEY and D. M. R. TAPLIN, in Proc. of 5th Int. Conf. on Fracture (ICF5) (Cannes, France, 1981) Vol. 4, p. 1613.
10. "Standard Test Method for Measuring Resistivity of Silicon Wafers with an in-Line Four-Point Probe," ASTM F 84-93, 1993.
11. F. M. SMITS, *The Bell System Technical Journal* (1958) 711.
12. "Standard Test Methods for Notched Bar Impact Testing of Metallic Materials," ASTM E 23-96, 1996.
13. R. C. SCHWANT and D. P. TIMO, "Life Assessment and Improvement of Turbogenerator Rotors for Fossil Plants" (Pergamon Press, New York, 1985) p. 325.
14. S. T. ROLFE and S. R. NOVAK, "Slow-Bend K_{IC} Testing of Medium-Strength High-Toughness Steels," ASTM STP 463 (1970) p. 124.
15. G. T. JONES, in Proc. of the Inst. of Mech. Eng. (1972) Vol. 186, p. 32.

Received 7 July 2000
and accepted 27 March 2002