Nondestructive Evaluation of Temper Embrittlement in Cr-Mo-V Rotor Steel*

Yoshikuni KADOYA**, Toru GOTO**, Mao TAKEI***

Nirou HARUKI***, Takeshi IKUNO*** and Yusaku NISHIMURA****

Temper embrittlement in low-alloy steels, one type of typical material degradation, occurs during service, and it is important to detect this embrittlement by means of a nondestructive technique. In order to develop a nondestructive method of evaluating embrittlement for Cr-Mo-V rotors, a study was conducted using lab-charged P-doped and Sn-doped steels. P-doped steels showed evident embrittlement by segregation of P at grain boundaries. On the other hand, Sn-doped steels showed little embrittlement when compared with P-doped steels. Various testing methods were investigated and compared in terms of their sensitivity and factors were detected, using these steels. The chemical etching test was found to be a very good measure of embrittlement. That is, it was found that there is good correlation between the width W of the etched grain boundary measured by the penetration of replicas and $FATT$. The correlation between W and $FATT$ was also recognized for samples taken from long-term-serviced rotors. The relation had the same slope as that found for lab-charged steels. From this, multiple regression analysis was conducted in order to express $FATT$ using W and variables which are known or are nondestructively measurable. As a result, a regression equation which gives an estimate of the actual $FATT$ with the scatter of $\pm 20^\circ\text{C}$ was obtained.

Key Words: Temper Embrittlement, Life Prediction, Nondestructive Evaluation, Steam Turbine Rotor, Chemical Etching Test, Replication Technique, Regression Analysis

1. Introduction

Many steam turbine plants have a record of their operation time over 100,000 hours, and the number of such plants will increase rapidly in the future. Under these circumstances, recently, problems of life evaluation have attracted wide attention in relation to the aged members of those plants which have been used for a long period, thus stimulating many researchers to tackle such problems in various ways^{(1),(2)}.

The problem of material degradations in Cr-Mo-V high-temperature rotors includes toughness degra-

dation due to their long duration of use (hereinafter referred to as temper embrittlement) as well as creep and fatigue⁽³⁾. Temper embrittlement in low-alloy steels, itself, is not a new problem, and a number of studies have been made from various aspects. Generally, it is said that temper embrittlement is caused by the segregation of impurity elements such as P and Sn at grain boundaries which lowers the bonding strength of grain boundaries⁽⁴⁾. Moreover, the investigation of retired rotors^{(3),(5)} confirmed a similar phenomenon of temper embrittlement in actual high-temperature rotors.

On the other hand, for nondestructive detection of temper embrittlement in rotor materials, the necessity of developing techniques is activating recent research. However, as shown in Table 1, the possible techniques are limited to only the chemical etching method⁽⁶⁾ and the EPR/ECP methods^{(7),(8)}, though they have limitations of accuracy and practical application under the present conditions. As one of the reasons for such limitations, it seems that these methods all focus their attention on the detection of P segregated at grain

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** Takasago Research and Development Center, Mitsubishi Heavy Industries, Ltd. 2-1-1 Shinhamma, Arai-cho, Takasago, Hyogo 676, Japan

*** Takasago Machinery Works, Mitsubishi Heavy Industries, Ltd. 2-1-1 Shinhamma, Arai-cho, Takasago, Hyogo 676, Japan

**** Technical Research Center, The Kansai Electric Power Co. Inc., 3-11-20 Wakaoji, Amagasaki, Hyogo 660, Japan

boundaries, but for the evaluation of temper embrittlement, it is also necessary to consider the effects of initial material properties that appear upon manufacturing. Therefore, these methods still seem to be more or less restricted. In any case, on the grounds mentioned above, for more precise corroboration of temper embrittlement in rotors after long use, it is an urgent problem to establish its nondestructive evaluation technique.

Therefore, in the present study, aiming at the establishment of a nondestructive evaluation technique to detect temper embrittlement in Cr-Mo-V high-temperature rotors, lab-charged steel materials of P-dope and Sn-dope were systematically prepared, and various nondestructive techniques were applied to those materials to test their sensitivity and the factors detected. Moreover, based on the test results, the correlation between the fracture appearance transition temperature (*FATT*) and detection parameters of the optimum nondestructive technique was investigated using test specimens sampled from actual rotors in addition to the lab-charged steels. Also taking the existing views into consideration, the methods of nondestructively estimating *FATT* after temper embrittlement were examined.

2. Samples and Testing Methods

2.1 Samples

The lab-charged steels were made, as shown in Table 2, as embrittlement simulated materials of which the chemical compositions were intentionally changed with various P and Sn contents. The sample

Table 1 Nondestructive methods of evaluating temper embrittlement

Approach	Testing method	Temper embrittlement	Ref.
Chemical	Chemical etching test	Poss.	(6)
Electro-chemical	EPR test	Poss.	(7)
	ECP test	Poss.	(8)
Electro-magnetic	Eddy-current test	Imposs.	(9)
	Barkhausen noise analysis	Prob. imposs.	(10)
Mechanical	Small punch test*	Dem.	(11)

Dem.: demonstrated, Poss.: possible, Imposs.: impossible
 Prob.: probably
 *: destructive test

Table 2 Chemical composition and heat treatment of laboratory-charged steels

Lab charge	Chemical composition (wt.%)										Note	Heat treatment
	C	Si	Mn	P	S	Ni	Cr	Mo	V	Sn		
A	0.30	0.25	0.78	0.004	0.004	0.38	1.14	1.12	0.23	0.005	Standard	1) As-received: AR
B	0.29	0.34	0.81	0.019	0.004	0.34	1.16	1.12	0.23	0.006		quenchy 955°C x 5h
C	0.28	0.25	0.79	0.017	0.004	0.34	1.14	1.12	0.23	0.005	P-dope	temper 680°C x 20h
D	0.30	0.34	0.80	0.033	0.004	0.38	1.15	1.14	0.23	0.005		2) Step cooling: SC
E	0.30	0.34	0.82	0.117	0.004	0.34	1.17	1.13	0.23	0.007		ref. (12)
F	0.29	0.34	0.81	0.004	0.004	0.38	1.17	1.13	0.24	0.012		550°C x 1h
G	0.29	0.34	0.80	0.004	0.004	0.38	1.16	1.15	0.24	0.021	Sn-dope	530°C x 15h
H	0.29	0.23	0.77	0.003	0.004	0.34	1.20	1.15	0.24	0.051		520°C x 24h / furnace
I	0.30	0.34	0.77	0.048	0.004	0.34	1.20	1.17	0.24	0.053	P+Sn dope	480°C x 72h / cool

steels were all made in a vacuum melt of 50 kg in weight and were forged into plates of 65 mm in thickness. These plates were cut into strips 30 mm in width, 130 mm in length and were heat-treated to be made equivalent to the 1200 mm-diameter drum center of the existing high-temperature rotor in service. The as-received (AR) steels were heat-treated to these conditions and used as test samples. Further, after the completion of the heat treatment mentioned above, the steels were subjected to step cooling⁽¹²⁾ (SC) and long-term heating of two kinds (LA) to be prepared as embrittlement-processed materials, which were then used in the following tests. Table 2 shows the thermal refining conditions and long-term heating conditions. For the examination of *FATT* estimation after temper embrittlement, in addition to the doped steels mentioned above, the test specimens were also sampled from the respective positions of five actual rotors (one newly manufactured rotor and four retired rotors). Table 3 shows the chemical composition and service time of each rotor.

2.2 Charpy impact test and metallographic tests

From among the samples described in section 2.1, test specimens for the V-notch Charpy impact test were prepared and used to measure *FATT*. After that test, the test specimens were subjected to the measurement of hardness and grain size and to Auger electron spectroscopy (AES).

2.3 Examination of detection sensitivity and factors detected in nondestructive techniques

Using the samples shown in Table 2, the following tests were conducted to determine the most effective technique. Based on the results of these tests, the selected effective technique was tested using the samples and the test specimens of actual rotors listed in Table 3. The correlation between *FATT* and detection parameters was investigated.

2.3.1 Chemical etching method After the impact test, the test specimens were polished for microstructural observation and then used in the chemical etching test. In the chemical etching method, the pre-austenite grain boundaries are etched with an etching fluid of picric-acid solution⁽¹³⁾ and the replica was made to estimate *FATT* by the etched level of grain boundaries⁽⁶⁾. It has been reported that good

Table 3 Chemical composition and history of rotors investigated

Rotor	Chemical composition (wt.%)										History
	C	Si	Mn	P	S	Ni	Cr	Mo	V	Sn	
rotor A	0.28	0.20	0.79	0.027	0.013	0.39	1.05	1.15	0.24	0.018	150000
rotor B	0.31	0.25	0.80	0.013	0.016	0.14	1.09	1.26	0.23	0.010	140000
rotor C	0.30	0.25	0.75	0.008	0.008	0.35	1.23	1.20	0.25	0.017	100000
rotor D	0.33	0.28	0.85	0.005	0.007	0.34	1.12	1.13	0.24	0.002	0
rotor E	0.35	0.27	0.80	0.035	0.030	0.16	1.05	1.29	0.27	0.010	163000

correspondence was confirmed between the etched level of grain boundaries and the quantity of P segregated at grain boundaries^{(6),(13)}. For measurement, from the viewpoint of accuracy, it is essential to maintain the etching conditions as constant as possible. Thus, taking this problem into consideration, an etching apparatus was constructed experimentally, the outward appearance of which is shown in Fig. 1. The etching test was carried out using this apparatus under the conditions of etching fluid temperature: 50 to 60°C and etching time: 3 min. For the quantification of the grain boundary corrosion level after replication, as a result of detailed examination, the width of an etched groove at the grain boundary was adopted. The replicas were subjected to SEM observation and the width of the etched groove at the grain boundary was obtained by means of an image analyzer.

2.3.2 Electrochemical potentiokinetic reactivation (EPR) method The same test specimens as those of (a) were used for the EPR test⁽⁷⁾. This method uses electrochemical techniques to evaluate the anode polarization characteristics. For the electrolyte, 14% $\text{Ca}(\text{NO}_3)_2$ was used. The test was performed at room temperature.

2.3.3 Eddy current test (ECT) method Small test specimens, 30 mm in width and 130 mm in length, were used for the ECT test⁽⁹⁾. This method, as one of the electromagnetic methods, makes effective use of impedance that varies with eddy current.

2.3.4 Barkhausen noise analysis (BHN) method The same test specimens as those of (c) served for the BHN test. This method, similar to the method of (c), uses electromagnetic techniques to analyze Barkhausen noises.

3. Test Results

3.1 Charpy impact and metallographic tests

The *FATT* values of lab-charged steels obtained by the Charpy impact test were rearranged according to the P and Sn contents, and the result was plotted in Fig. 2. In this figure, the white mark shows as-

Fig. 1 Chemical etching test device

received material, the black mark shows step-cooled material and the half-black mark shows long-term-heated material, while the different marks such as the circles, squares and triangles are used for the different content of P or Sn. (The same notation was used in Fig. 6 and Fig. 7.) Figure 2 also shows the grain size, hardness and the results of AES analysis. The *FATT* of P-doped material increases almost in proportion to the increase in P content. Further, it was confirmed that the step-cooled material and the $450^\circ\text{C} \times 10^4 \text{ h}$ long-term-heated material had a *FATT* larger than that of as-received material. In contrast, the *FATT* of Sn-doped material changed little with the increase in Sn content. However, the material doped with P+Sn composite (Make: I) showed a larger *FATT* compared with the material of P or Sn doped singly, and the effect of embrittlement-aimed heat treatment appeared conspicuously in that composite material.

The relative peak strengths of P and Sn at the grain boundary surface obtained by AES analysis

Fig. 2 Comparison of embrittlement behavior of P and Sn contents and heat treatment

proved to have generally the same tendency as expressed in the chemical composition of bulks shown in Table 2. In particular, the *FATT* values were rearranged according to the relative peak strength of P element, and the result was plotted in Fig. 3. It is noted that the *FATT* increases in proportion to the increase in the relative peak strength of the P element.

Figure 4 is a typical distribution of P and Sn elements in as-received material and embrittlement-aimed heat-treated material, respectively, in the depth direction from the grain boundary surface to the inside. As embrittlement proceeds, the segregation layer of these elements tends to become deeper. Particularly, in the distribution of the P element, this tendency is intensified.

3.2 Detection sensitivity and factors detected in various nondestructive techniques

The surface conditions of the replicas after the chemical etching test, obtained by SEM observation,

Fig. 3 Correlations between *FATT* versus P on grain faces measured by AES

Fig. 4 Distribution of P and Sn in the vicinity of grain boundary measured by AES

are shown in Fig. 5. From this figure, it is clear that the grain boundaries were etched selectively. As a result AES analysis, it was confirmed that mainly the P element was segregated at grain boundaries. Therefore, it is clear that the selective etching of grain boundaries is due to segregation of P at the grain boundaries. Figure 6 shows the correlation between the width of the etched groove at the grain boundary and *FATT*, where good correspondence was observed between them, though there were some variations. (The typical scatter band in estimated *FATT* is about 50°C.) The data plotted outside the band are of P+Sn composite material.

Figure 7 shows the correlation between EPR values after the EPR test and *FATT*. This plot indicates many variations and no distinct correlation was observed between the two. The EPR values were rearranged in relation to P content and to Sn content, which confirmed that the EPR values tended to become larger than the measuring sensitivity as P

Fig. 5 Scanning electron micrograph showing intergranular attack morphology after chemical etching test

Fig. 6 Correlation between *FATT* and width of penetration from replica in chemical etching test for laboratory-charged steels

content increases, but hardly showed dependency on Sn content. The ECT test and the BHN test results were also arranged in the same manner. However, these test results showed too many variation in the values obtained and yielded no significant conclusions.

4. Nondestructive Estimation Method for Temper Embrittlement

4.1 Correlation between the width of etched groove at grain boundary (W) and $FATT$

In the previous section, it was indicated that the chemical etching method had the most potential as a nondestructive technique for detecting $FATT$. The correlation between the width of etched grooves at grain boundaries and $FATT$ was investigated using the test specimens of actual rotors shown in Table 3,

Fig. 7 Correlation between $FATT$ and EPR coulomb density in EPR test for laboratory-charged steels

Fig. 8 Correlation between $FATT$ and width of penetration from replica in chemical etching test for laboratory-charged steels and rotors

in addition to the lab-charged steels. Figure 8 shows the result of this investigation. This plot, like Fig. 6, confirmed good correspondence between the two, and may be rearranged into a band of almost the same slope in the case of either the lab-charged steels or the test specimens of actual rotors. However, this plot also indicates that the location between the lab-charged steel band and the rotor test specimen band are different along the horizontal axis ($FATT$). This is considered to be due partly to the difference in the initial characteristics (chemical composition, hardness and grain size) at the time of manufacture, in addition to the difference in the quantity of P segregated at grain boundaries. This point has often been taken into consideration in many reports which discuss nondestructive estimation methods for temper embrittlement in low-alloy steels^{(14)–(17)}. Then, from the same standpoint, an examination was carried out using the multiple regression analysis in the following section.

4.2 Proposal of $FATT$ regression equation on multiple regression analysis

From the correlation between W and $FATT$ in Fig. 8, the regression equation shown below was supposed.

$$FATT = a \cdot W + \sum b_i \cdot X_i \quad (1)$$

Here, a is a coefficient which does not depend on the type of specimen, as recognized from Fig. 8; X_i denotes initial characteristic factors at the time of manufacture and b_i denotes their coefficients. That is, the correlation between W and $FATT$ may be expressed as a cluster of parallel, linear lines.

Then, X_i corresponded to the chemical compositions in Tables 2 and 3, and to hardness and grain size of which correspondence to $FATT$ were confirmed in many reports^{(14)–(17)}. Further, as a parameter to indicate the degree of embrittlement, in addition to the above, the J -factor⁽¹⁸⁾, $(P+Sn) \cdot (Si+Mn) \times 10^4$, a well-known factor, was also included. As for Sn content, for actual rotor materials, there is usually no manufacturing record of the analyzed value of S. (The Sn contents of actual rotors shown in Table 3 were the values obtained separately from sampling analysis.) However, as part of the investigation of temper embrittlement in long-term-serviced rotors, recently, core samples are often taken from actual rotors⁽¹⁹⁾. Thus, it is easy to determine the Sn content by analyzing those samples. In cases where these techniques are not applicable, the average value of the Sn content in the results of actual rotors so far manufactured⁽³⁾ is available. The information on hardness and grain size can be obtained simultaneously through the investigation of material degradations in high-temperature rotors⁽²⁰⁾.

As stated above, W , chemical composition (C, Si, etc.), hardness (Hv), grain size (G.S.) and J -factor (J) were applied as factors to evaluate Eq. (1). The analysis data for 75 samples were used. Then, using the parameters mentioned above (14 variables), the multiple regression analysis was carried out. These 75 samples are the sum of the samples in Table 2 which experienced different heat-treatment conditions and those in Table 3 taken from the respective positions of actual rotors. For the multiple regression analysis, it should be presupposed that the variables are all in normal distribution and are independent of each other. Accordingly, first, the basic statistics of each variable and correlation coefficient γ between variables were obtained. As a result, it was clear that chemical composition variables C, S and V did not show a normal distribution but showed a large peak (biquadratic moment) in basic statistics. Also, the correlation coefficient γ calculated was always $\gamma \leq 0.6$ between the narrowed-down variables mentioned later, so that it was considered that the variables were independent of each other. Table 4 shows the result of rearrangement made relative to correlation coefficient

Table 4 Correlation coefficients between $FATT$ and individual variables

Correlation coefficient	Factor												
	W	Hv	G.S.	C	Si	Mn	P	S	Ni	Cr	Mo	V	J
γ_{FATT}	0.62	0.52	0.27	0.05	0.23	0.02	0.36	0.03	0.06	0.35	0.22	0.11	0.35

Table 5 The results of regression analysis using standard method, enter variable method and remove variable method

Method	Factor													Multiple correlation coefficient (R)	Coefficient of determination (R^2)	Standard error
	W	Hv	G.S.	C	Si	Mn	P	S	Ni	Cr	Mo	V	J			
Standard	○	○	○	○	○	○	○	○	○	○	○	○	○	0.902	0.813	22.2
Enter variable	○	○	○	○	○	○	○	○	○	○	○	○	○	0.892	0.795	21.7
Remove variable	○	○	○	○	○	○	○	○	○	○	○	○	○	0.940	0.883	16.1

Fig. 9 Comparison between measured values of $FATT$ and predicted values of $FATT$ using the present method

γ to view the correlation of each variable with $FATT$. Within the range of the present analyzing data, W , Hv , P and J -factor have a comparatively high correlativity. These results also correspond well to the views on temper embrittlement so far obtained.

On the other hand, as shown in Fig. 8, analysis data were available for 14 samples consisting of lab-charged steels A to I and actual rotor specimens A to E. Therefore, when Eq. (1) is subjected to the multiple regression analysis, up to 14 variables can be used for X_i . In this case, the variables listed in Table 4 can all be adopted. Then X_i factors were selected and a and b_i were determined in the following procedure. First, all the variables in Table 4 underwent the multiple regression analysis as independent variable. Next, the multiple regression analysis was conducted by the enter variable method and the remove variable method as a sequential selection-type multiple regression analysis (a method of selection in the order of largeness of the F -level). The result is shown in Table 5. Therefore, the result of analysis by the remove variable method was adopted since it showed the highest multiple correlation coefficient and the smallest standard error. That is, the factors obtained for X_i are Hv , Si , Mn , P , Cr , Sn , and J -factor. Thereby, Eq. (2) was obtained as a $FATT$ estimation equation:

$$FATT = a \cdot W + b_1 \cdot Hv + b_2 \cdot Si + b_3 \cdot Mn + b_4 \cdot P + b_5 \cdot Cr + b_6 \cdot Sn + b_7 \cdot J\text{-factor} + b_8 \quad (2)$$

where $a=99.12$, $b_1=1.609$, $b_2=816.4$, $b_3=-652.5$, $b_4=3320$, $b_5=-310.4$, $b_6=3404$, $b_7=-0.282$ and $b_8=325.6$.

Figure 9 shows the comparison of the predicted values of $FATT$ obtained through Eq. (2) and the actually measured values of $FATT$. Here it can be seen that the result of prediction remains within $\pm 20^\circ\text{C}$ of the actually measured values, in either case of lab-charged steels or actual rotor specimens. In the prediction of $FATT$ using the actual rotor specimens, it was confirmed that there was little change in the plot of Fig. 9, even if for Sn content the average of the values in the manufacturing results of actual rotors is used.

For $FATT$ estimation based on the multiple regression analysis, various methods have been proposed for Cr-Mo-V steel^{(14),(16),(17)} and Ni-Cr-Mo-V steel^{(15),(17)}. However, among these methods, some have the following disadvantages: the range of $FATT$ is quite limited;⁽¹⁴⁾ there is the necessity of AES analysis;⁽¹⁵⁾ and a special technique must be used in the multiple regression analysis because of the limited number of data⁽¹⁶⁾. Taking these points into consideration, the method proposed in the present study is considered no less useful than the other methods.

Therefore, in Fig. 10, the method proposed here

Fig. 10 Estimation procedure of predicted FATT using the present method (This method is referred to as *MACH-E*: Material Characteristic Embrittlement.)

was summarized as a flow chart. First, the results of the analytical calculation and of the investigation of material degradations are used as reference to determine the measuring position for evaluation. Then, the measuring position is subjected to the chemical etching test. After that, replication is executed to determine the width of the etched groove at the grain boundary W . Further, by additionally entering the manufacturing record data (chemical composition) and the investigation data on material degradations (hardness, etc.) into the $FATT$ regression equation, the $FATT$ estimation is finally performed. The proposal in this study enables the nondestructive estimation of temper embrittlement for Cr-Mo-V high-temperature rotors.

5. Conclusions

In order to develop the nondestructive evaluation method of temper embrittlement for Cr-Mo-V high-temperature rotors, P-doped and Sn-doped lab-charged steels were made systematically, and used in various nondestructive tests to examine detection sensitivity and the factors detected. Moreover, based on the results from these tests, the correlation between $FATT$ and the detection parameters in the optimum nondestructive technique was investigated, and the method of estimating $FATT$ after temper embrittlement was examined. The results are summarized as follows.

(1) In the chemical etching method, good correspondence was obtained between the width of the etched groove at the grain boundary and $FATT$. Furthermore, it was considered that the selective corrosion of the grain boundaries appearing in this method indicated the segregation of P at grain boundaries.

(2) No definite correlation was observed between the EPR values and $FATT$ in the EPR method. No

significant results were obtained from the ECT method or the BHN method.

(3) Based on the results of (1) and (2) above, the chemical etching method was adopted. The correlation between $FATT$ and the width of etched grooves at grain boundaries was investigated. It was clear that the plotted data might be rearranged into a band of almost the same slope in the case of either lab-charged steels or actual rotor specimens.

(4) Moreover, taking the initial material properties at the time of manufacture into consideration, along with the width of the etched groove at the grain boundary, the multiple regression analysis was conducted. As a result, the $FATT$ regression equation which can estimate the actual $FATT$ within the scatter of $\pm 20^\circ\text{C}$ of the measured values was obtained.

(5) For Cr-Mo-V high-temperature rotors, the method wherein $FATT$ after temper embrittlement is estimated by a nondestructive technique which uses the chemical etching method and the manufacturing record data was proposed.

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