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Nondestructive evaluation of flow properties in thermally aged Cr–Mo–V steel using instrumented indentation tests

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Abstract Instrumented indentation technique has been utilized to assess the flow properties in X20CrMoV12.1 steel, widely used in power generation facilities, at various heat treatment stages simulating thermal aging during the service. The steel samples were heat treated at 600 and 650 °C for 1–2,000 h and flow properties were evaluated at various heating temperature and times by instrumented indentation tests using spherical indenter. Microstructure evolution after various heat treatments has been investigated to correlate the degradation of flow properties with the microstructure evolution due to thermal aging. We show that the degradation in flow properties in the steel sample can be described by Larson–Miller parameter analysis.

Introduction

It is well known that the material properties of structural components in industrial facilities can degrade gradually when the components are operated under high-temperature condition for a longtime. Therefore, for safe and economic operation of high-temperature facilities, it is of importance to periodically monitor the degradation in the material properties of the facilities. The creep rupture tests have been generally used to evaluate the degradation of high-temperature materials [1–4], in which the Larson–Miller parameter (LMP) is used as a parameter to determine the degree of degradation as a function of operating temperature and time [5–7]. However, in addition to the destructive characteristics of the method, there are difficulties in applying it to the in-field components since it needs a large number of longtime experiments under various temperature and stress conditions. On the other hand, nondestructive hardness tests have been performed to evaluate the time-dependent degradation of the materials used in industrial fields [8, 9]. Nevertheless, the conventional hardness test has a fundamental limitation that hardness value is not a fundamental property useful for the mechanical design of an industrial structure.

In order to overcome the limitations of the current practice, instrumented indentation technique (IIT) [10–15] which can measure the flow properties of a material [16, 17] was adopted in this study to evaluate the lifetime of high-temperature materials. Samples with various degradation states were prepared by accelerating degradation at high temperatures (600 and 650 °C) for 1–2,000 h for X20CrMoV12.1 steel. Flow properties, degraded with heat treatments, were measured by IIT and were correlated with the LMP in consideration of thermal aging temperature and time. Microstructures of the materials were observed by optical microscope (OM) and transmission electron microscope (TEM). Flow properties of the same material used for 62,000 and 180,000 h as a tube in a power generation facilities were also measured for comparison purpose.

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Experiments

X20CrMoV12.1 steel tested in this study which standardized for use in steam pipes under the DIN designation (German grade F12) has been widely used for steam pipes as well as for other section components in power plants for decades. Table 1 shows the chemical composition of the X20CrMoV12.1 steel, and its manufacturing processes and properties are described in Ref. [18]. To control the states of degradation, tube samples with outer diameter of 39 mm and the thickness of 7 mm were treated by thermal aging at high temperatures (600 and 650 °C) for 1–2,000 h. Temperature condition was determined by considering operating temperature of in-filed facilities (540–570 °C) together with austenite phase transformation temperature (~660 °C). LMP was used to describe the degree of degradation of the material properties by

$$\mathrm{LMP} = (T + 273)(\log t + C), \tag{1}$$

where T is the heat treatment temperature in Celsius, t the heat treatment time, and C is the material constant. C is known to be 20–25 for Cr–Mo steels, 20 is used in this study [19]. A tube that was used in the thermal power plant for 62,000 h (~7 years) which was made of the same material was prepared.

Flow properties were estimated by IIT with a spherical indenter based on representative stress–strain approach using the measured indentation force–depth curves. Detailed procedure for the estimation of flow properties is introduced elsewhere [16, 17]. IIT was performed using AIS 3000 (Frontics Inc., Seoul, Korea) with a depth resolution of 0.1 µm and a force resolution of 0.055 N. Spherical indenter with a radius of 250 µm was used, and 15 partial unloadings were conducted. Maximum indentation depth was 150 µm, unloading ratio of each unloading was 50%, and rate of indentation loading/unloading was 30 µm/min. Since destructive sample preparation was available, uni-axial tensile tests were performed by Instron 5584 (Instron Inc., MA, USA) to confirm the flow properties measured by IIT. Uni-axial tensile testing was performed along the longitudinal direction of the tube with a rectangular shape specimen with a gauge length of 25 mm, with a loading rate of 1 mm/min. Microstructures of the materials were observed by OM and TEM. TEM observation was performed by JEOL-JEM 2000FX, and electrolytic polishing was carried out in a solution of methyl alcohol and perchloric acid at 40 V and −20 °C for the TEM sample preparation.

Table 1 Chemical composition of and safety criteria for X20CrMoV12.1 steel

Components						
C	Si	Mn	Cr	Mo	Ni	V
0.2	0.3	0.5	11.2	1.0	0.5	0.3

Desired conditions for safety			
Standard	Yield strength (MPa)	Ultimate tensile strength (MPa)	Elongation (%)
DIN: 17175-79	490	690	23

Results and discussion

Figure 1 shows the values of yield strength σ_y and ultimate tensile strength σ_{UTS} measured by IIT and uni-axial tensile testing. They are in very good agreement, which shows the possibility to measure flow properties of materials in-use only with IIT. Flow properties (yield strength, ultimate tensile strength, work-hardening exponent n) with LMP values in eq. 1 were presented in Fig. 2. For the material

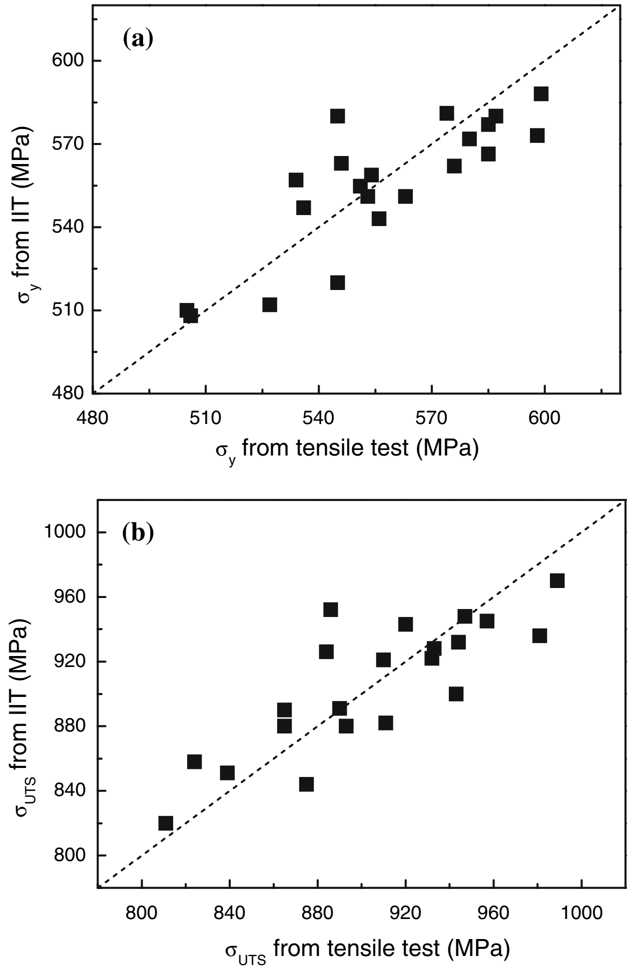

Fig. 1 Comparisons of **a** yield strength and **b** ultimate tensile strength measured by instrumented indentation tests and uniaxial tensile tests

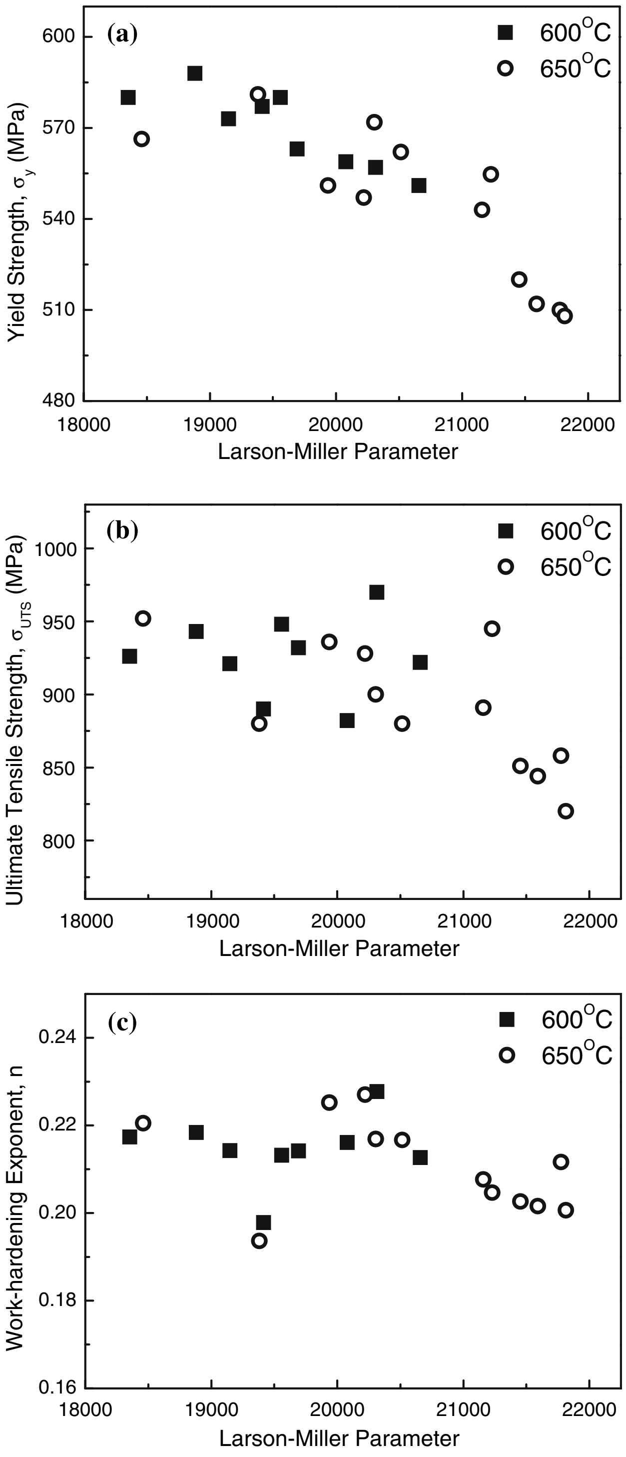

Fig. 2 **a** Yield strength, **b** ultimate tensile strength, and **c** work-hardening exponent versus the LMPs

examined in this study, yield strength and ultimate tensile strength values decrease gradually with increasing LMP, whereas work-hardening exponent values are less sensitive to LMP. It is known that work-hardening exponent can be used to describe the deformation and fracture behaviors of materials. However, for the material used here, it is not sufficiently sensitive to LMP and thus it cannot be used as an indicator for the degree of degradation. Trend in decrease in yield strength with increasing LMP is clearer than ultimate tensile strength. Note that a criterion based on yield strength is more conservative than one based on ultimate tensile strength, since only reversible elastic deformation is permitted within the yield strength criterion and severe plastic deformation of the materials in a facility can even induce a plastic collapse [20, 21]. Therefore, yield strength is determined as a degradation indicator in this study.

In order to analyze the mechanism of the degradation in flow properties, microstructures of the materials thermally aged at 650 °C for 1 and 1000 h were observed by OM, as shown in Fig. 3. Matrix phase is tempered martensite of lath type with dark and bright needle-shaped phases stacked in turns. The dark and bright phases are believed to be ferrite and cementite phases, respectively [18]. It is seen

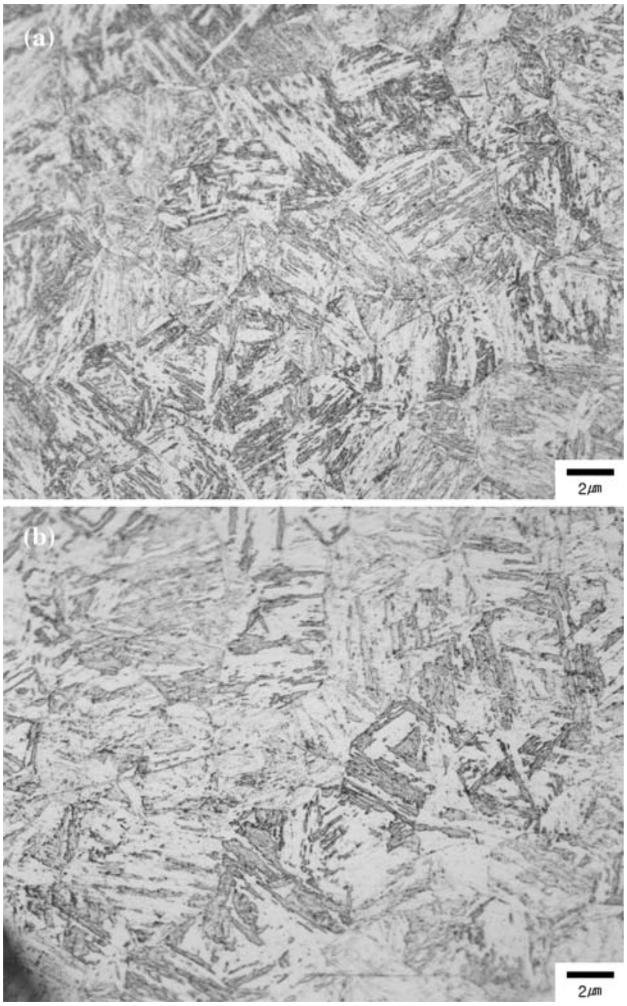

Fig. 3 Microstructures observed by OM for materials heat treated at 650 °C for **a** 1 and **b** 1000 h

that laths become larger and the gaps between them become wider during thermal aging. Figure 4 is the TEM images of the materials thermally aged at 650 °C for 1 and 1000 h. Microstructure in the TEM image consists of martensite laths and carbides which are separated or connected by thin boundary layers. From the analyses of shape and diffraction figures of the carbide, it was defined as M23C6. With increasing aging time, lath boundaries became clear and volume fraction of the M23C6 carbides was found to be increased due to their coalescence [22, 23]. In X20CrMoV12.1, the Cr and Mo are continuously discharged from matrix during longtime use at high temperature, which leads to coalescence of the carbides. This carbide coarsening might reduce the contribution of precipitation hardening, which is known to be associated with important cause of degradation of material properties [24]. Collectively, the decrease in the yield strength of the material with thermal aging might be due to growths of laths as well as coalescence of carbide in matrix. In addition, rearrangement of dislocation or dislocation recovery facilitating easy glide of other dislocations can be another cause of decrease in strength with degradation [9, 20, 25, 26].

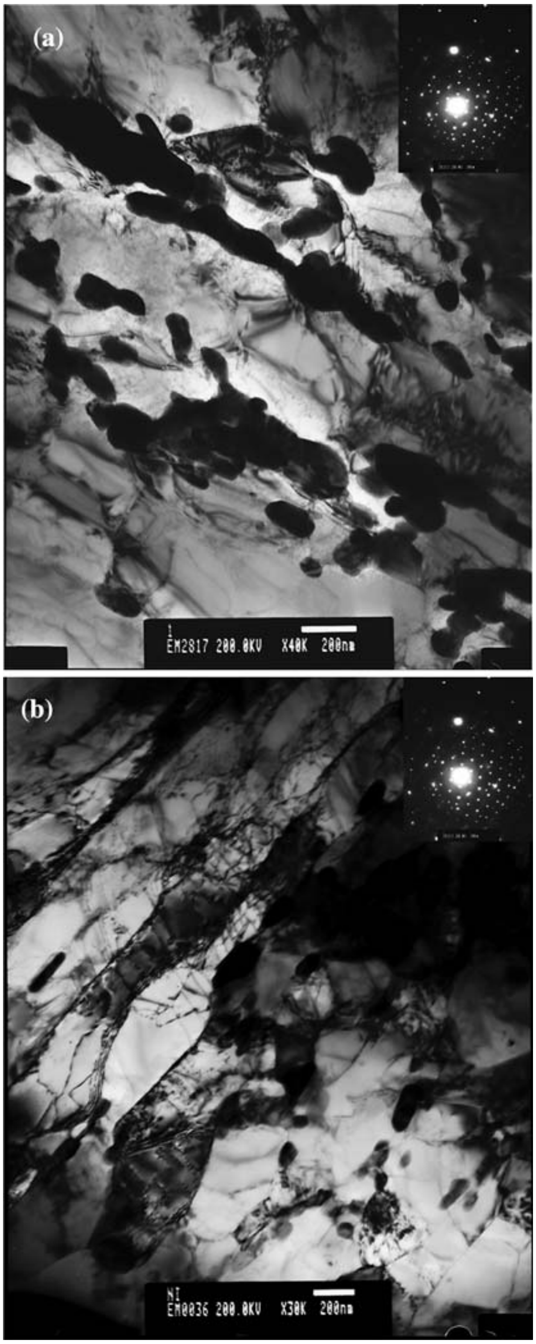

Fig. 4 TEM images of the materials heat-treated at 650 °C for **a** 1 and **b** 1000 h

Yield strength values with LMP were fitted to the second-order polynomial function and shown in Fig. 5. Master curve of material property degradation is generally fitted to experimental results with orthogonal polynomial approximation and linear regression method [27], which can be approximately described by polynomial function. To verify that this master curve can reasonably describe the degradation of yield strength, two experimental results were also presented in Fig. 5; one is measured from the same material used for 62,000 h (~7 years) in a thermal power plant, the other is the result from the material used for 180,000 h reported in previous research [24]. As shown in Fig. 5, the results from the materials are also well fitted into the master curve.

Master curve for degradation of yield strength makes it possible to evaluate lifetime of the material by determining the failure criterion for yield strength. We have made an attempt to evaluate the lifetime of the material even though correct failure criterion for yield strength of this material was not established. Failure criterion was determined by considering the desired conditions for safety in the standard. The desired strength values in standard (Table 1)

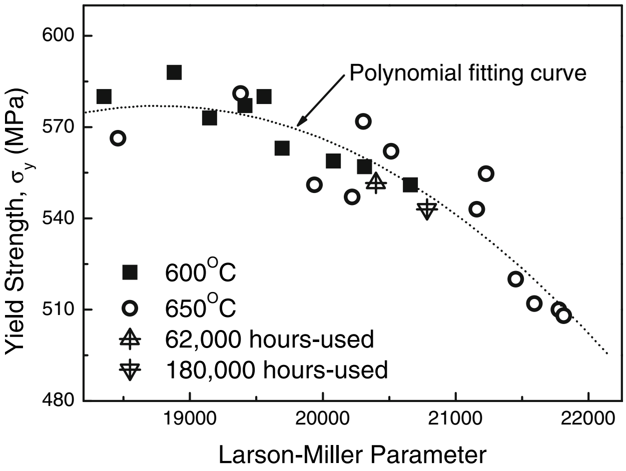

Fig. 5 Master curve for degradation in yield strength with LMPs

were determined to satisfy creep strength more than 100,000 h for as-manufactured material [18, 28]. Supposing 10% safety margin for yield strength is taken into account for conservative safety assessment, lifetime of this material was evaluated as 390,604 h. The high-temperature materials in generation facilities are usually under high pressure that is converted into applied stress. By considering maximum applied stress, geometry of facility, and safety margin, one can determine the failure criterion and lifetime of high-temperature materials based on degradation master curve for yield strength.

Conclusions

We have adopted nondestructive IIT for evaluating the degradation of flow properties of a high-temperature material. Various degradation states of X20CrMoV12.1 were made by acceleration aging. Among measured flow properties (yield strength, ultimate tensile strength, and work-hardening exponent), yield strength was chosen as an indicator of degradation of material properties since decrease in its values with increasing LMP is clear and it can provide more conservative criterion than one based on ultimate tensile strength. The decreasing tendency of yield strength was described as a polynomial master curve. The usefulness of the master curve was verified by putting the experimental results of the practically aged materials (used for 62,000 and 180,000-h, respectively, in a thermal power plant).

References

1. Marahleh G, Kheder ARI, Hamad HF (2006) Mater Sci Eng A 433:305
2. Masuyama F (2007) Int J Press Ves Pip 84:53
3. Hur SK, Hong KT, Doh JM (1994) Bull Korean Inst Met Mater 7:356
4. Mathew MD, Bhanu Sankara Rao K, Mannan SL, Paknikar K, Singh R (1997) Key Eng Mater 171–174:537
5. Larson FR, Miller J (1952) Trans ASME 74:765
6. Zhou Y, Devarajan B, Murty KL (2004) Nucl Eng Des 228:3
7. Chellapandi P, Srinivasan R, Chetal SC, Raj B (2006) Int J Press Ves Pip 83:556
8. Viswanathan R (1989) Damage mechanism and life assessment of high temperature components. ASM International, Metals Park, OH
9. Wheeler F et al (1992) Demonstration of life assessment techniques for boiler superheater and reheater parts. Electric Power Research Institute Report, Palo Alto, CA
10. Oliver WC, Pharr GM (1992) J Mater Res 7:1564
11. Oliver WC, Pharr GM (2004) J Mater Res 19:3
12. Kim JY, Lee BW, Read DT, Kwon D (2005) Scr Mater 52:353
13. Kim JY, Lee JS, Lee KW, Kim KH, Kwon D (2006) Key Eng Mater 326–328:487
14. Kim JY, Lee JJ, Lee YH, Jang JI, Kwon D (2006) J Mater Res 21:2975
15. Kim JY, Kang SK, Lee JJ, Jang JI, Lee YH, Kwon D (2007) Acta Mater 55:3555
16. Dowling NE (1993) Mechanical behavior of materials. Prentice Hall, Englewood Cliffs
17. Kim JY, Lee KW, Lee JS, Kwon D (2006) Surf Coat Technol 201:4278
18. DIN 17175-79 (1979) Seamless steel tubes for elevated temperatures
19. Ahn JH, Kwon D (2001) J Mater Res 16:3170
20. Dieter GE (1988) Mechanical metallurgy. McGraw-Hill, Singapore
21. Anderson TL (1995) Fracture mechanics. CRC Press, FL
22. Hu ZF, Yang ZG (2004) Mater Sci Eng A 383:224
23. Kostka A, Tak KG, Hellmig RJ, Estrin Y, Eggeler G (2007) Acta Mater 55:539
24. Seok CS, Koo JM (2005) Mater Sci Eng A 395:141
25. Jang JI, Choi Y, Lee YH, Kwon D (2005) Mater Sci Eng A 395:295
26. Shoji T (1994) Time-dependent degradation and life-time prediction. Realize Inc., Tokyo
27. Ray AK, Tiwari YN, Sinha RK, Chaudhuri S, Singh R (2000) Eng Fail Anal 7:359
28. ASME B&PV Part A (2004) Specification for seamless ferritic and austenitic alloy-steel boiler, superheater and heat-exchanger tubes