

High Temperature Stress-Strain Analysis of P91 Steel

Relevant for: Nuclear research, cutting tools manufacturers, aerospace industry

The evaluation of mechanical properties at elevated temperatures is crucial for materials used in applications such as cutting tools, power plants and aerospace industry. Instrumented high temperature nanoindentation is an excellent measurement method for obtaining various mechanical properties without demanding sample preparation. This application report presents results of P91 steel measured with the High Temperature Ultra Nanoindentation Tester (UNHT³ HTV) from room temperature up to 600 °C.



1 Introduction

Ferritic/martensitic (F/M) steels are generally selected, first, on the basis of their resistance to corrosion and, second, on the basis of their mechanical properties. However, elevated temperature properties become the primary concern as the temperature of the operating environment increases. P91 steel is a modern F/M alloy steel for applications in both conventional and nuclear power plants. It exhibits high strength up to temperatures in the range of 600 °C and has higher oxidation temperature limits. This allows engineering components, such as steam piping and heater coils, to operate with less thickness, and to have a longer thermal fatigue life.

The main alloying elements are chromium (9 wt%) and molybdenum (1 wt%). The former improves high temperature strength and increases oxidation resistance, the latter increases creep resistance. Minor quantities of Nickel and Manganese are present, increasing the hardenability of the steel. This alloy steel is formed by normalizing at 1050 °C and air

cooling down to 200 °C. It is then tempered by heating to 760 °C.

The knowledge of mechanical properties of this alloy at operating temperature is essential in order to ensure its long performance and to better understand its welding strategy, as preheating, maintaining inter-pass temperature, and post-weld heat treatment are very critical for the P91 grade. Significant progress in high temperature nanoindentation over the last years allows various material properties to be measured from one test at elevated temperature [1].

Nanoindentation measurements performed on P91 steel from room temperature up to 600 °C by means of the High Temperature Ultra Nanoindentation Tester (UNHT³ HTV) are reported. Both conventional quasi-static nanoindentation and stress-strain analysis are used to determine the hardness, elastic modulus and yield stress evolution as a function of temperature.

2 UNHT³ HTV Instrument

The Anton Paar High Temperature Ultra Nanoindentation tester (UNHT³ HTV) has been developed for testing different kinds of materials at temperatures up to 800 °C. Based on the proved and patented technology of the Ultra Nanoindentation Tester (UNHT³) [2,3] the UNHT3 HTV also uses active top referencing which minimizes thermal drift and gives unparalleled measurement stability. The instrument has a maximum load of 100 mN with a resolution of 6 nN, and a depth range of 100 µm with a resolution of 0.006 nm.

The indenter, reference and sample are heated separately by infrared heating elements, creating an infrared bath at the sample surface (Figure 1). An integrated cooling system combined with heat shielding allows stable control of thermal flow. Thermocouples placed within the indenters and at other strategic locations allow accurate monitoring of test temperature. The sample is held rigidly by means

of a retaining ring which clamps it in place, thus removing any need for use of glue or cement [4].

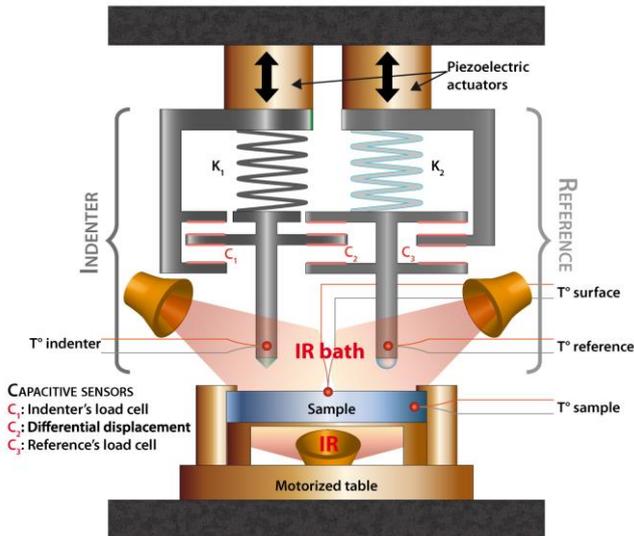


Figure 1: Schematic representation of the UNHT³ HTV system

The measuring head, sample stage and optical video microscope are mounted in a high vacuum chamber which can be pumped down to 10⁻⁷ mBar using a primary pump and a turbomolecular secondary pump. A vacuum buffer connected between the secondary pump and the backing valve allows operation over many hours without needing to use the primary pump. This can maintain a 10⁻⁶ vacuum for over 10 hours. Measurements are usually performed in the range 10⁻⁵ - 10⁻⁶ mbar.

Further details about the instrument, such as the heating system and its thermal stability, as well as the procedure for minimizing thermal drift prior to the measurement are described elsewhere [1].

3 Testing parameters and stress-strain analysis

The P91 steel sample was tested between room temperature and 600 °C in steps of 200 °C. A Tungsten Carbide (WC) spherical indenter of 20 μm radius was chosen due to the reactivity of diamond with steels [5].

Continuous multicycle (CMC) and continuous stiffness method (CSM) were applied for the measurements. For both loading modes the maximal applied force was set to 100 mN. Moreover, for CMC mode, 20 cycles with quadratic load increase, loading/unloading time of 10 s and 5 s dwell at maximal load for each cycle were used. For CSM mode, three constant strain rates of 0.05 s⁻¹, 0.01 s⁻¹ and 0.1 s⁻¹ were chosen with sinus frequency of 20 Hz.

The hardness evolution as a function of penetration depth is obtained from both measurement modes. The calculation of indentation stress-strain (σ - ϵ) is made by applying Tabor's formulas [6]:

$$\sigma = \frac{H_{IT}}{3}$$

$$\epsilon = 0.2 \frac{a}{R},$$

where H_{IT} is the indentation hardness, R is the radius of the indenter and a is the projected contact radius which can be determined from the measured contact depth h_c :

$$a = \sqrt{2Rh_c - h_c^2}.$$

The analyses presented here assume perfect spherical indenter geometry with 20 μm tip radius and a frame compliance measured with a Berkovich indenter inserted in a similar shaft.

4 Results

The calculated mean curves of hardness as a function of the penetration depth at each temperature and for the two different measurement modes are shown in Figure 2. As expected, the material is getting softer with increasing temperature.

It should be noted that the nanoindentation curves at intermediate temperature, 200 °C, especially for the strain rate of 0.01 s⁻¹, show more oscillations of the hardness curve compared to the other temperatures. A possible cause could be dynamic strain ageing of the steel [7], visible as characteristic serrated yielding. Further measurements would be needed to confirm this assumption.

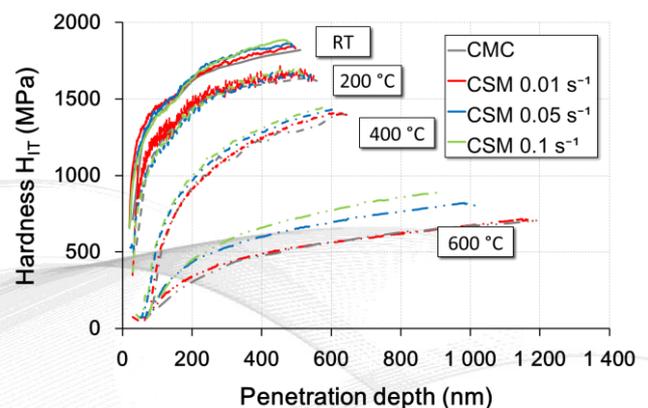


Figure 2: Mean curves of indentation hardness (H_{IT}) versus penetration depth for P91 steel obtained at room temperature, 200 °C, 400 °C and 600 °C, using CMC and CSM mode with different strain rates.

The average values of instrumented indentation hardness H_{IT} at different penetration depths for strain rate of 0.05 s^{-1} are shown in Figure 3. The average value of indentation hardness for a strain rate of 0.5 s^{-1} and penetration depth of 400 nm decreased approximately 3 times between room temperature and 600 °C.

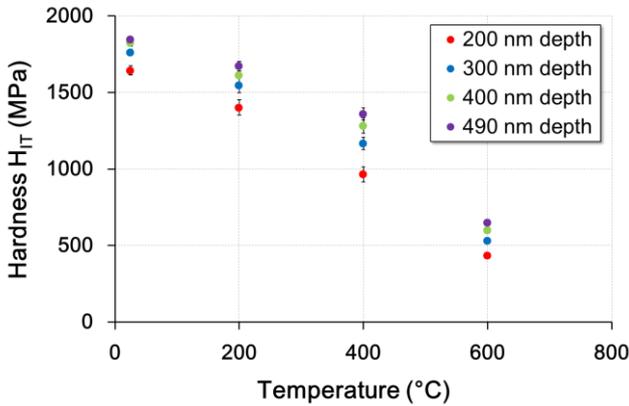


Figure 3: Average instrumented indentation hardness values (H_{IT}) as a function of temperature for P91 steel at different penetration depths for a strain rate 0.05 s^{-1}

Exposure to temperature seems to have an important influence on the observed microstructure as can be seen in the optical image of an indent taken after testing at 600 °C (Figure 4).

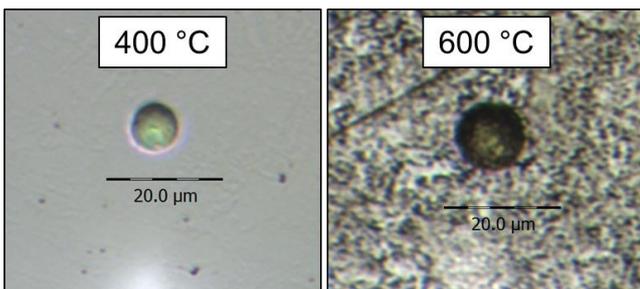


Figure 4: Optical images of indents and the sample microstructure after testing at 400 °C and 600 °C

The representative indentation stress-strain curves for different temperatures are displayed in Figure 5 and the calculated average indentation yield stress values as a function of temperature for different strain rates is plotted in Figure 6. Good agreement between the different testing modes is obtained when calculating the stress-strain curves at the range from room temperature up to 600 °C. The measured yield stress decreases approximately linearly with the increasing temperature for all strain rates when considering the standard deviation.

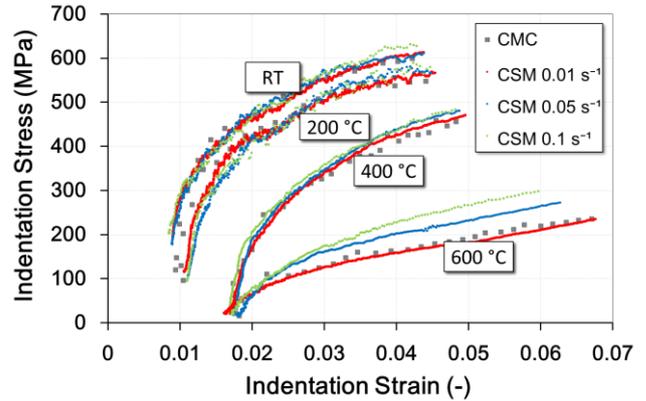


Figure 5: Representative indentation stress-strain curves for P91 steel obtained up to 600 °C for different strain rates

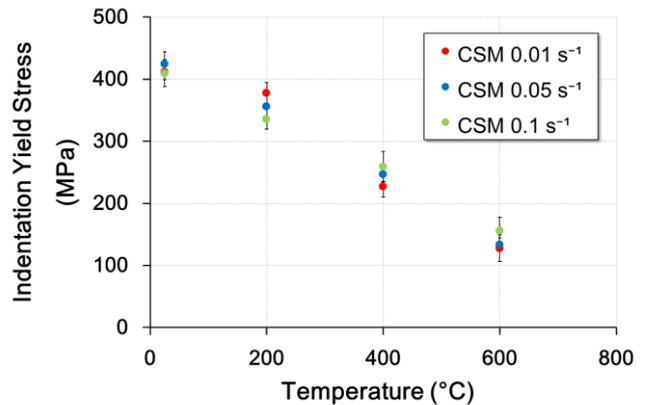


Figure 6: Average values of indentation yield stress as a function of temperature for P91 steel up to 600 °C for different strain rates

The yield strength measured by classical tensile testing at room temperature and 550 °C was 520 MPa and 388 MPa respectively. The results of indentation yield stress are often not in accordance with the results obtained by other methods such as uniaxial tensile or compression testing [8]. The different scale of these tests makes any direct comparison with nanoindentation very challenging. The size effect, in fact, plays an important role due to the complex stress distribution below the indenter and the surface interactions happening at such scale.

5 Summary

This application report demonstrates the capability of the Anton Paar UNHT³ HTV for characterization of mechanical properties at elevated temperature. The unique instrument design assures very low thermal drift during the measurements.

Using the sinus mode (continuous stiffness method), the evolution of hardness and elastic modulus can be recorded over the entire predefined penetration depth. The stress-strain analysis implemented in the Anton Paar software can be easily applied to obtain the comparative evolution of indentation yield stress with increasing temperature. However, further research is required to arrive at reliable predictions of the mechanical materials performance based on nanoindentation testing. Challenges persist with regard to, e.g. the understanding of the dependence of the dynamic response on the CSM parameters applied, the applicability of Tabor's formulae for the conversion of the indentation curves into stress-strain curves, the influence of the material's temperature and strain-rate sensitivity, and/or the robustness of calibration methods for spherical indenters especially for shallow depths.

6 Acknowledgements

Anton Paar Group kindly thanks Dr. Ana Ruiz-Moreno from the European Joint Research Center (JRC), Petten, NL for the collaboration that led to the presented results.

7 References

- [1] High Temperature Ultra Nanoindentation Tester (UNHT³ HTV) for measuring mechanical properties up to 800 °C, Application report G93IA049EN, www.anton-paar.com, 2016
- [2] J. Nohava, N. X. Randall, N. Conté, Novel ultra nanoindentation method with extremely low thermal drift, *J. Mater. Res.*, Vol. 24, No. 3 (2009) 873-882
- [3] US Patent 7,685,868 B2, EP 1828744, 2010
- [4] High Temperature Nanoindentation Adjustable sample support, Application Flash G93IA504EN, www.anton-paar.com, 2017
- [5] J. M. Wheeler, J. Michler, Indenter materials for high temperature nanoindentation, *Rev. Sci. Instr.*, 84 (2013) 101301
- [6] D. Tabor, *The hardness of metals*, Clarendon Press, Oxford, UK, 1951
- [7] P. Hähner, A. Ziegenbein, E. Rizzi, and H. Neuhäuser, Spatiotemporal analysis of Portevin–Le Châtelier deformation bands: Theory, simulation, and experiment, *Phys. Rev. B* 65 (2002) 134109
- [8] E.G. Herbert, On the measurement of yield strength by spherical Indentation, Thesis, University of Tennessee - Knoxville, 2006

Contact Anton Paar GmbH

Tel: +43 316 257-0

application@anton-paar.com | www.anton-paar.com