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Characterization of the creep properties of heat resistant 9–12% chromium steels by miniature specimen testing

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ABSTRACT

A miniature specimen geometry is presented that allows to extract small samples for tensile creep tests directly from critical components of steam generators in power plants like e.g. superheater tubes. In this way, material can be tested which has been subjected to the full processing chain of the component. The specimens then exhibit all microstructural modifications that are present in the component after the shaping process and heat treatment, and representative mechanical properties are determined. Similarly, specimens can be extracted from pre-corroded test pieces or used components after service to determine the impact of complex aging/loading/oxidation conditions during service on the mechanical properties of the material. The applicability of the miniature specimen test method is demonstrated in a comparative creep study involving standard and miniature specimens of P91 tempered martensite ferritic steel. Test results indicate satisfactory accuracy/repeatability of the method. Comparison with large scale specimen data reveals an influence of specimen size on the obtained creep behavior. These size effects need to be considered for a correct interpretation of results from miniature specimen creep tests.

1. Introduction

The knowledge of the mechanical and corrosive properties under service conditions constitutes a prerequisite for using any engineering material in high temperature applications. In the field of energy conversion in fossil-fuelled power plants, new challenges have arisen in materials characterization and selection over the last decade due to fundamental changes of the typical service conditions. Initiatives for climate protection by reducing the carbon dioxide emissions of energy conversion processes have stimulated research for high efficiency power plants with increased steam pressure and temperature parameters that exceed the typical working conditions [1], and new combustion concepts like co-firing of biomass [2] or the oxyfuel process [3] involve novel flue gas compositions which in turn modify the fireside corrosion conditions. In addition, conventional power plants are forced into flexible operation with increasing numbers of start-ups and shut-downs to balance the fluctuating feed-in of renewable energies [4].

The mechanical properties of candidate materials for power plant applications are typically determined in tensile tests, notched-bar impact tests and long-term creep rupture tests at different elevated temperatures, and respective minimum requirements are defined in national technical rules, e.g. [5]. The 100,000 h creep rupture strength is generally accepted as the key parameter for material selection and design of components. In the assessment of new material grades, corresponding values are typically established by extrapolating the results of 30,000–40,000 h creep tests to this lifetime [6].

The present work reports on a miniature specimen creep test approach that may be applied as a complementary creep characterization method. Compared to the traditional test set-up, it offers the following advantages:

(i) Specimens may be obtained from test pieces with small dimensions (down to 6 mm in thickness). This allows extracting specimens from thin-walled plant components like e.g. superheater tubes after manufacture, Fig. 1. In this case, a fully representative material condition is tested. It includes all microstructure modifications resulting from shaping and tempering processes and allows assessing their possible effects on the creep properties of the component.

(ii) Similarly, specimens may be obtained from thin-walled components after service to obtain information on the impact of the complex aging/loading/oxidation conditions during service on the mechanical properties and residual life of the material.

(iii) Miniature specimens may be obtained from near-surface areas of corrosion test coupons or from thick-walled components after service to clarify whether the material below the

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Fig. 1. Extraction of miniature creep specimens from a superheater tube for steam power plants. The tube (left) is cut into 60 mm long sections which allow extracting bars of 7 mm diameter out of the sidewall by electrical discharge machining. From these pieces, cylindrical creep specimens (right) are machined according to Fig. 2.

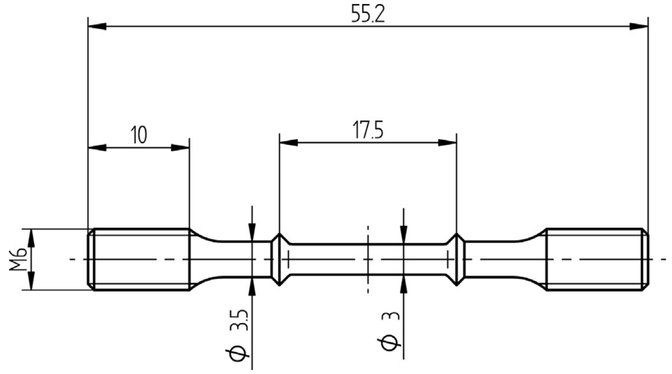

Fig. 2. Geometry of the miniature creep specimen used in this study (dimensions given in mm).

surface/corrosion layer exhibits degraded mechanical properties caused by depletion or enrichment of elements, like e.g. carburization.

This contribution first introduces the new test approach and reports on the details of the test rig. In a second part, preliminary results for P91 tempered martensite ferritic steel are presented which allow assessing the applicability of this method.

2. Design of creep specimen and test rig

The design of the miniature creep specimen used in this study is given in Fig. 2. It closely follows the requirements of the standards ISO 204 [7] and ASTM E139 [8]. A cylindrical test piece of $D=3$ mm nominal diameter with collars for extensometry application at the ends of the parallel length is used. This diameter represents the smallest size allowed by ISO 204 [7], where a lower limit of 7 mm² is defined for the original cross-sectional area. The specimen is connected to the load train of the creep test rig by 6 mm threaded grip ends. The parallel length L_c of the gauge section is 15 mm, resulting in an original reference length $L_{ro} \geq 5D$ as requested in [7]. For the calculation of strain values in cases where extensometers are attached to ridges or shoulders of the specimen, a corrected gauge length needs to be considered that accounts for the deformation contribution of the ridges. Following the procedure given in [8] for calculating an adjusted length of the reduced portion, a value of 15.15 mm results from our nominal specimen geometry. However, an accurate individual value was determined for each specimen of our study based on the measured specimen dimensions to avoid any machining influence. All strain values reported in this study were calculated using the adjusted lengths.

The elongation of the reduced section is determined using a custom-built rod and tube type extensometer with reduced weight and dimensions (rod and tube diameters of 6 mm and 3 mm, respectively) which transmits the relative movement of the specimen's ridges to two displacement sensors. The expected global deformation is low due to the reduced gauge length of the specimen. Therefore, digital inductive displacement probes with a resolution/step size of 0.076 μm (corresponding to a nominal strain increment of 5×10^{-6} in the gauge length) are used. The signals of the two applied sensors are averaged for higher accuracy.

The specimens are loaded in a conventional lever-arm test rig for constant-load creep testing with an original maximum load capacity of 25 kN. Typically, the creep characterization of ferritic–martensitic steels for power plant applications involves stresses in the 75–250 MPa range which in turn correspond to loads of less than 2 kN when using the miniature specimen. For testing in this low load range, the load train of the creep test rig was replaced by a lighter custom-built version with pull rods of 12 mm diameter, and the lever arm ratio was reduced to 3:1 to allow for accurate stress adjustment. In order to ensure correct and constant loading throughout the experiments, a load cell (5 kN load capacity) was integrated into the customized load train. The load and elongation signals are continuously recorded throughout the tests.

3. Example results for P91 tempered martensite ferritic steel

3.1. Material and experiments

For validation of the test method introduced above, a series of creep experiments was performed on a P91 tempered martensite ferritic steel. The ASME grade P/T91 material with a nominal composition of 9Cr–1Mo–0.2V–0.08Nb–0.05N (wt%) was originally developed in the 1970s; until today it is widely used for power plant components like superheater tubes, headers and steam pipes at maximum temperatures of 1100 °F/593 °C [1] and higher [9]. It represents the second generation of 9–12% chromium steels with optimized C, V and Nb contents [1,10], leading to a fine dispersion of V/Nb-carbonitrides (of type MX) in the microstructure that enhances the creep resistance of the material [11]. The tempered martensite microstructure of P91 and other 9–12% chromium steels as well as the microstructural evolution during high temperature loading of these steels and their long-term creep behavior have been analyzed comprehensively, e.g. [12–16].

For the present study, both miniature specimens according to Fig. 2 and specimens of a more common size with 6 mm diameter and a gauge length of 47 mm (denoted as standard specimens in the following) were extracted from a P91 steam pipe with a wall thickness of 18 mm. The investigated section of the pipe was heat treated in a two-step procedure (1100 °C/60 min followed by air cooling, then 750 °C/240 min and air cooling). Prior to mechanical testing, the microstructure of the material was checked by metallographic analysis. As shown in Fig. 3, the material exhibits a homogeneous tempered martensite microstructure with high amounts of internal interfaces and carbide precipitates that result in favorable creep properties [17,18]. The creep behavior of the material was assessed in constant load creep tests in air. Test temperatures (650–700 °C) and stresses