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Application of spherical indentation and the materials knowledge system framework to establishing microstructure-yield strength linkages from carbon steel scoops excised from high-temperature exposed components

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A B S T R A C T

Industrial power generation turbines operate at elevated temperatures for prolonged periods of time (around 100,000 h) which leads to significant microstructure evolution and mechanical property changes. Due to physical and structural constraints of operational turbines, only small scoop samples can be excised from heat-exposed steel components. Scoop samples at various service time intervals provide valuable information on microstructure changes, for example on graphite formation in carbon steels, with increasing service time. However, mechanical evaluation of such small material volumes poses significant challenges using conventional tests. A novel spherical microindentation technique is applied to evaluate a library of scoop samples ranging between 0 and 99,000 h of service. Furthermore, microstructure and yield strength data for the different exposure periods is used to construct a structure-property (S-P) linkage using the MKS homogenization approach that employs spatial correlations, principal component analysis, and regression techniques. The accuracy of the extracted S-P linkage was assessed on new samples that were not included in the calibration set.

1. Introduction

Industrial gas or steam turbine components exposed to prolonged high temperatures experience significant changes in their internal structure (referred as microstructure in this paper), which are generally accompanied with changes in the mechanical properties of the material [1]. Accurate assessment of the change in mechanical properties, such as yield strength, with service exposure is critical for life cycle management of such components. This entails quantifying and analyzing the trends in mechanical property degradation at various service intervals during the service lifetime of a component, which can exceed 100,000 h [2]. Arguably, simulating the extreme conditions experienced during service in a laboratory setting would produce the desired samples for mechanical and microstructure evaluation. However, the amount of time required to produce such samples presents a significant challenge to the industry. Although, considerable effort is spent in designing accelerated aging tests, it is not yet clear that they successfully reproduce the same microstructure evolution and the associated changes to properties as in the real components. This is because of the highly complex multiscale materials phenomena involved in the aging process, which exhibit high sensitivity to factors such as the environment, temperature fluctuations, and internal stresses. As a consequence, a practical way forward has been to excise small shallow scoops from the in-service components at selected time intervals in their service lifetime, and to extract useful and reliable information from them regarding both the changes in the material microstructure as well as the degradation of the mechanical properties.

One of the main challenges in assessing the mechanical

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properties of the small shallow scoops in this study is that the conventional mechanical tests (e.g., compression or tension tests) are not viable due to the small volumes of material and scoop shapes extracted from operating turbine components. Although small-scale testing (e.g., miniaturized samples) is possible, it usually incurs a high cost and highly specialized equipment and expertise. Moreover, since the in-service samples exhibit a gradient of degradation (in the direction normal to the exposed surface), it is not clear that the miniaturized samples would be able to evaluate reliably the degradation in the very top surface layer of the in-service components. On the other hand, instrumented indentation techniques offer distinct advantages in addressing this critical need. In particular, recent work [3,4] has demonstrated the feasibility of extracting indentation stress-strain (ISS) curves from instrumented spherical indentation tests. These novel protocols have been demonstrated on a wide range of materials [5–10], including samples subjected to surface modification or damage by ion-irradiation [11]. In this study, we explore for the first time, the feasibility of applying the novel microindentation stress-strain protocols on the small scoop samples excised from in-service components and correlating them to measurements from tensile tests.

Even with the ability to measure important mechanical properties, indentation tests on all components might not be possible due to physical and time constraints. This presents a significant challenge in determining the loss of mechanical properties in cases when physical samples are not available. However, portable microstructure imaging techniques [12–14] allow microstructure imaging in highly constrained areas even where shallow scoops are impossible to extract. Current industry approaches for component life estimation are primarily engineering-based correlations centered on nominal operation history (e.g., elapsed operation time, applied stress) or resort to destructive testing of service components, which can be prohibitively costly. Naturally, such methods do not account for important material microstructure changes that may occur throughout component service histories. Yet, it is well established that material mechanical properties are microstructure-sensitive [15–22], and therefore, it is imperative to use suitable measures of the microstructure in accurately predicting the mechanical performance of various engineering materials; this requires formulation of highly reliable and robust material structure-property (S-P) linkages [23–26]. Recent efforts to capture the complex physical material phenomena in predictive models using data science methods have resulted in the formulation of the MKS (materials knowledge systems) framework [23,24,27–31]. The application of the MKS framework on a broad range of multiscale materials phenomena has demonstrated its value in extracting robust and practically useful S-P linkages [4,23,29,32,33]. In this paper, we demonstrate the application of these concepts and toolsets in extracting high value S-P linkages for service-exposed gas turbine steel microstructures.

2. Background

2.1. Spherical indentation stress-strain protocols

Instrumented spherical indentation stress-strain protocols [6,11,34–36] have demonstrated a robust, high throughput ability to extract mechanical properties from small material volumes. Most of the prior effort was focused on very small length scales of the volumes probed in the indentation tests (controlled mainly by the indenter tip radii), which typically varied between ~50 nms and ~5 μm. This is because these prior studies were aimed at studying mechanical response of volumes within individual grains of a polycrystalline sample. These tests required low forces ($\ll$10 N) and benefitted from continuous stiffness measurement (CSM) [6,8,10,34,36] for reliably estimating the changes in the contact radius during the indentation tests. Only recently [3,4,37], these new protocols have been extended to studies where the sizes of the indentation zones are of the order of several hundreds of microns. The indentation stress-strain curves obtained using these relatively larger indenter tips aim to capture the overall response of a polycrystalline aggregate, and have been shown to be well-correlated to the stress-strain curves measured in conventional tension/compression tests [3,38]. Typically, indentations with the larger tip radii require larger forces ($\gg$10 N). Suitable instrumented testing machines allowing for these larger indents along with the CSM capability are not yet commercially available. In order to address this gap, suitable approaches have been developed [3,4,35,37] that employ multiple load-unload cycles during the test; these produce a more discrete indentation stress-strain curve compared to the ones produced using nanoindenters with a built-in CSM capability.

The framework used to extract stress-strain response of the indentation tests in this paper is largely based on Hertz's theory and follows the protocols developed recently [3,35] to convert the measured non-CSM load-displacement data to indentation stress-strain curves. It is emphasized that each load-unload cycle in these non-CSM test protocols produces a single point on the indentation stress-strain curve.

Hertz theory [39] for frictionless, elastic contact between two isotropic, homogeneous bodies with quadratic surfaces can be expressed using the following relations:

$$P = \frac{4}{3}E_{eff}R_{eff}^{1/2}h_e^{3/2} \tag{1}$$

$$a = \sqrt{R_{eff}h_e} \tag{2}$$

$$\frac{1}{E_{eff}} = \frac{1-\nu_s^2}{E_s} + \frac{1-\nu_i^2}{E_i} \tag{3}$$

$$\frac{1}{R_{eff}} = \frac{1}{R_s} + \frac{1}{R_i} \tag{4}$$

where P and h_e denote indentation load and elastic displacement, respectively, R and E are the radius and Young's modulus, and a denotes the indentation contact radius. Subscripts s and i are associated with sample and indenter, respectively, while R_{eff} and E_{eff} are the effective radius and elastic modulus of the indenter-sample system.

The effective modulus, E_{eff}, is determined from the first load-unload cycle, where the contact is elastic. Since no permanent deformation is induced in this cycle, the sample surface remains flat, i.e., $R_{eff} = R_i$ in Eq. (4). E_{eff} can be obtained from Eq. (1) using regression techniques. E_s is then obtained from Eq. (3) by using Poisson ratio (ν_s) of 0.3 for the sample and Poisson ratio (ν_i) of 0.21 and Young's modulus of 640 GPa for the tungsten carbide indenter [40].

Prior work [34,35,41–45] has emphasized the importance of the "zero-point" correction for the measured load-displacement data in indentation experiments. This is mainly because the zero-point established by the machine is not sufficiently accurate to produce meaningful indentation stress-strain curves. In the protocols employed in this work, the zero-point correction identifying the effective initial contact between the indenter and the sample are determined separately for each test. Load and displacement zero-point corrections are performed using the non-CSM analyses protocols described in prior work [3,35], where the corrections