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## Stress relaxation behavior in single crystal superalloys

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#### ABSTRACT

The objective of this study was to examine the stress relaxation response and corresponding changes in microstructure of single crystal superalloys. The effects of temperature and time on stress relaxation response were examined for single crystal superalloys CMSX-4 and EPM-102. Stress-strain rate data from relaxation tests was in good agreement with constant load tests; it was further refined by testing samples from the same casting, and by eliminating the effects of primary creep in the stress relaxation test. From these tests, it was determined that EPM-102 had higher resistance to stress relaxation than CMSX-4 at and above 982 °C. Additionally, time-dependent strain recovery ("viscoelasticity") was observed after unloading during these tests. The single crystal alloys displayed directional coarsening after stress relaxation testing at high temperatures.

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#### 1. Introduction

High temperature creep testing and stress relaxation testing are closely related experimental approaches that measure an alloy's time dependent deformation behavior. The stress-strain-time path is different for stress relaxation tests, in which a strain rather than creep stress is applied and then held constant. Stress relaxation tests have been explored as an efficient means for gathering rate data over several decades in strain rate [1–3]. It has been observed in these studies that strain rate-stress data from stress relaxation tests can be compared to that from creep tests. Lack of exact agreement has been attributed to at least two factors. First, the secondary creep rate in a creep test is typically measured after several tens to thousands of hours duration, (depending on the applied stress), whereas the decades of strain rate data from a stress relaxation test are typically obtained in 24 h or less. This difference implies that the early portions (at least) of the stress relaxation test include effects of primary creep, whereas the creep test is measuring a secondary creep rate. Second, the different test durations allow for different microstructural evolution, which can also affect the measured strain rates.

Stress relaxation testing is also considered an essential calibration test for advanced deformation models that couple creep and plasticity [4,5]. However, the prime motivation for the tests in the current study was the need to increase the temperature

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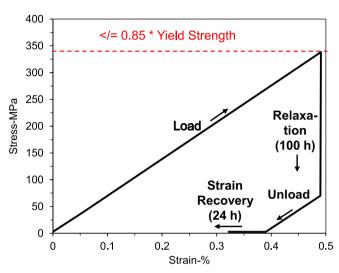
capability of high temperature seals [6,7]. Here, stress relaxation testing closely mimics the intended engineering application, where a spring is used to maintain a constant displacement between two sealing surfaces.

Compared to creep test data, stress relaxation data for superalloys is sparse [1,2,8-10] and especially rare for single crystal alloys [2,9,10]. Test durations at constant strain beyond 24 h are essential for a higher fidelity design of the high temperature seals, but such data are absent. Observations of microstructural changes during stress relaxation testing are similarly uncommon. Microstructural evolution is especially important for these high temperature applications, where  $\gamma'$  rafting is well characterized in creep testing [11–15] above the final aging temperature of these alloys, but has yet to be reported in any stress relaxation testing at these temperatures. Thus, the objectives of this study were to explore the stress relaxation responses and corresponding changes in microstructure of single crystal superalloys tested at high temperatures. The effects of temperature and time on stress relaxation were examined for two advanced alloys. Several generations of single crystal superalloys have been developed over the years, having increase levels of Re and corresponding creep resistance [16]. The 2nd generation alloy CMSX-4 and the 4th generation alloy EPM-102 were chosen because of the interest in determining the temperature capability of high temperature seals based on these superalloy springs. A few additional tests were performed on a first generation alloy NASAIR 100, to generate an exact comparison of the two test types (creep and relaxation) from specimens taken from a single casting.

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Table 1
Compositions of CMSX-4, EPM-102, and NASAIR 100 single crystal superalloys.

| Alloy (wt%)                     | Al                   | В               | С            | Со                 | Cr                   | Hf                | Mo                   | Ni                   | Re                | Ta                   | Ti                | W                     | Y                   | Other  |
|---------------------------------|----------------------|-----------------|--------------|--------------------|----------------------|-------------------|----------------------|----------------------|-------------------|----------------------|-------------------|-----------------------|---------------------|--------|
| CMSX-4<br>EPM-102<br>NASAIR 100 | 5.60<br>5.55<br>5.50 | -<br>0.004<br>- | 0.01<br>0.03 | 9.00<br>16.50<br>– | 6.50<br>2.00<br>9.50 | 0.10<br>0.15<br>— | 0.60<br>2.00<br>1.00 | Bal.<br>Bal.<br>Bal. | 3.00<br>5.95<br>– | 6.50<br>8.25<br>3.20 | 1.00<br>-<br>1.20 | 6.00<br>6.00<br>10.00 | 0.005<br>0.010<br>- | 3.0 Ru |



**Fig. 1.** Stress-strain curve showing test designed for determination of stress relaxation and strain recovery in tests of CMSX-4 and EPM-102.

#### 2. Materials and test procedures

The compositions in weight percent of the two tested single crystal blade superalloys are listed in Table 1. CMSX-4 is a 2nd generation single crystal superalloy [17] which has often been used for many turbine airfoil applications. It has a modest Re content of 3.0% and displays good creep resistance at a relatively low measured density of 8.69 g/cm<sup>3</sup>. EPM-102 is a 4th generation superalloy [16] having a higher Re content of 6% and higher creep resistance, albeit at a higher density of 9.16 g/cm<sup>3</sup>.

These superalloys were produced as rectangular slabs using standard single crystal casting practices. The slabs each had a

nominal width of 5 cm and length of 15 cm, and thicknesses of 0.6–1.3 cm. CMSX-4 slabs were homogenized and solution heat treated to about 1300 °C, then gas fan quenched. EPM-102 slabs were homogenized and solution heat treated to 1305 °C, then gas fan quenched. Both materials were then given a subsequent heat treatment of 1141 °C for 6 h plus 871 °C for 20 h. Slabs were then macro-etched and X-rayed, to insure they contained no high angle grain boundaries. Specimens were machined so as to be oriented within 10° of the [001] crystallographic direction.

Additionally, two confirmation tests were performed on companion specimens of a first generation single crystal superalloy NASAIR 100, of the same material composition and casting mold which had been extensively characterized for creep response and associated directional coarsening in references [12,18].

Tensile stress relaxation tests were performed at temperatures of 870-1093 °C on specimens having nominal gage diameters of 3.2 mm, with a gage length of 21 mm. They were performed in an electro-mechanical universal testing machine (Instru-Met Corp.), using a clamshell resistance heating furnace (Applied Testing Systems) and a contacting axial extensometer (MTS Systems Model 632.53E-14) measuring strain over a gage length of 12.7 mm. These tests were performed in general accordance with elevated temperature tensile testing specification ASTM E21-09. However, tests initiated with strain ramped at the specified strain rate of  $8.3 \times 10^{-5}$  1/s were interrupted at a strain corresponding to 0.85\* yield strength at 870 °C, 983 °C, and 1093 °C for each alloy, and held there with strain held constant for 100 h to measure relaxation of stress as a function of time. Supplementary tests of EPM-102 were also performed at lower percentages of yield strength and for different test times. A few tests were also performed to examine a lesser known behavior [4,5] of time-dependent strain recovery at nominally zero stress. In these tests, the specimen was given a standard stress relaxation period of 100 h, and then unloaded to a very small load sufficient to maintain a tight load train and the strain recovery was monitored while held

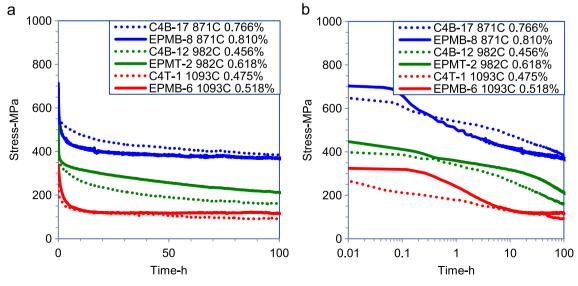


Fig. 2. Comparison of stress relaxation responses at strains corresponding to 0.85 \* yield strength for CMSX-4 and EPM-102: (a) linear time, and (b) log (time).

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