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# Potential implications of step loading in impression creep testing

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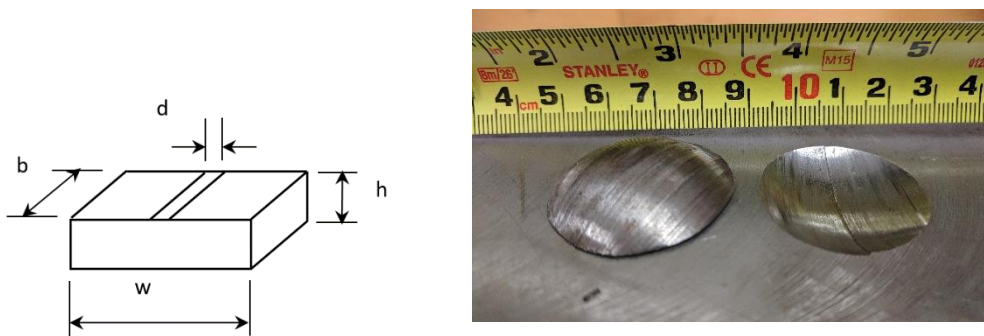
**Abstract:** The small sample impression creep test method has recently been of interest, as it can give a good indication of expected creep rates in uniaxial creep testing with minimal use of material. The compressively loaded test has also been shown to provide accurate results under multi-step loading conditions for a low alloy steel ( $\frac{1}{2}\text{Cr}\frac{1}{2}\text{Mo}\frac{1}{4}\text{V}$ ) to further extract value from a single test specimen. The Electric Power Research Institute (EPRI) has conducted step tests (step temperature and step loading changes) on another low alloy steel (Grade 22), as well as a tempered martensitic 9 Cr steel (Grade 91). Results have shown that there may be potential problematic areas when conducting step-up and step-down steps in these materials. Additional posttest evaluations have shown that material effects, such as strain hardening and strain softening, may add additional complexities when comparing strain rates of multi-stepped loaded strain rates. Hardness testing on posttest impression creep specimens have confirmed strain softening of tempered martensitic Grade 91 and no observed effect for an ex-service Grade 22 alloy. These findings have shown that careful considerations must be made before using creep rates obtained from multi-stepped loaded tests in situ of single loaded tests.

**Keywords:** Impression creep, strain softening, strain hardening, hardness, Grade 91, Grade 22

## 1. Introduction

In recent years, the impression creep test has gained interest in the testing community. The test method can be used as an alternative to standard uniaxial testing to determine steady state creep rates. Specifically, the test is used when an insufficient amount of material exists for uniaxial creep testing. Traditional uniaxial creep tests use a larger volume of material, about 100-200 times more than impression creep testing, which is considered a ‘destructive’ technique that generally requires weld repair or replacement of the extracted volume of material. Impression creep specimens can be machined from ‘scoop’ samples and is considered a ‘non-invasive’ testing technique. Scoop sampling has typically been used on main steam (MS) and hot reheat (HRH) steam piping components in fossil power plants [1]. Replications can be performed for microstructure evaluations, but the scoop can be machined into an impression creep specimen. The standard impression creep specimen geometry and an example of a scoop sample is shown in Figure 1.

Impression creep testing has shown to be useful for multi-step loading in service-aged  $\frac{1}{2}\text{Cr}\frac{1}{2}\text{Mo}\frac{1}{4}\text{V}$  low alloy steel [2]. Results showed that creep rates obtained under different loading histories were in good agreement with those of single loaded tests. It is also important to note that step up and step down testing was performed in this study. Recent testing by EPRI on two different materials has raised concern over the use of multi-step loading and multi-step temperature changes. Additional posttest assessments were also performed for further evaluation of the potential effects of step testing. This involved hardness testing at the cross-section perpendicular to the indentation formed during testing. Findings and potential implications are further expounded in the discussion section.

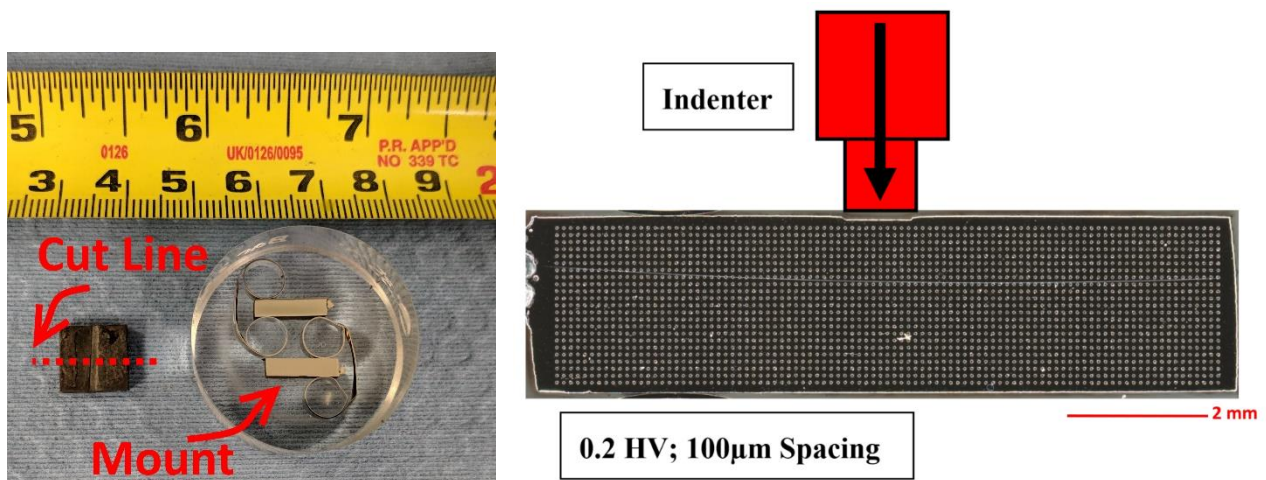


**Figure 1.** Standard Impression Creep Specimen (left) and Removed Scoop Sample (right).

## 2. Materials and Methods

After material has been removed (scoop sample, etc.) the impression creep specimen can be cut using electric discharge machining (EDM) to a thickness of ~2.55 to 2.60 mm. A final thickness of  $2.50 \pm 0.02$  mm is achieved using 600 grit sandpaper. The standard tested specimens have samples dimensions of 10mm x 10mm x  $2.50 \pm 0.02$  mm, as shown in Figure 1. Electric Power Research Institute (EPRI) is currently using a Tinius Olsen twin-screw servo-mechanical (H25KS) loading frame with a high temperature ceramic shell furnace using kanthal windings. A software program has been specifically designed for controlling a static load with a signal input for water cooled linear variable differential transformers (LVDTs) to measure displacement during the entire duration of the test. The independent three zone furnace uses type N thermocouples for control of temperature, but the sample temperature is measured with an external data acquisition (DAQ) system using three type K thermocouples. A high strength Waspaloy nickel based superalloy was chosen as the material for the indenter, as it has a much higher creep strength than that of the materials being tested. These specifications are outlined in the current code of practice [3].

Three different impression creep tests were carried out for a normalized and tempered martensitic Grade 91 steel. Test loads were varied (between 80-120 MPa) at a constant testing temperature of 625°C. After testing, the samples were cut in half perpendicular to the indentation that was formed. These were mounted using a hot mounting process and then polished using standard metallographic techniques. Hardness testing was performed at each of the three samples using a 0.2 kg load with 100  $\mu$ m spacing. Figure 2 shows an impression creep sample before cutting with the two halves mounted (left) and a macro image of the sample after hardness mapping (right).

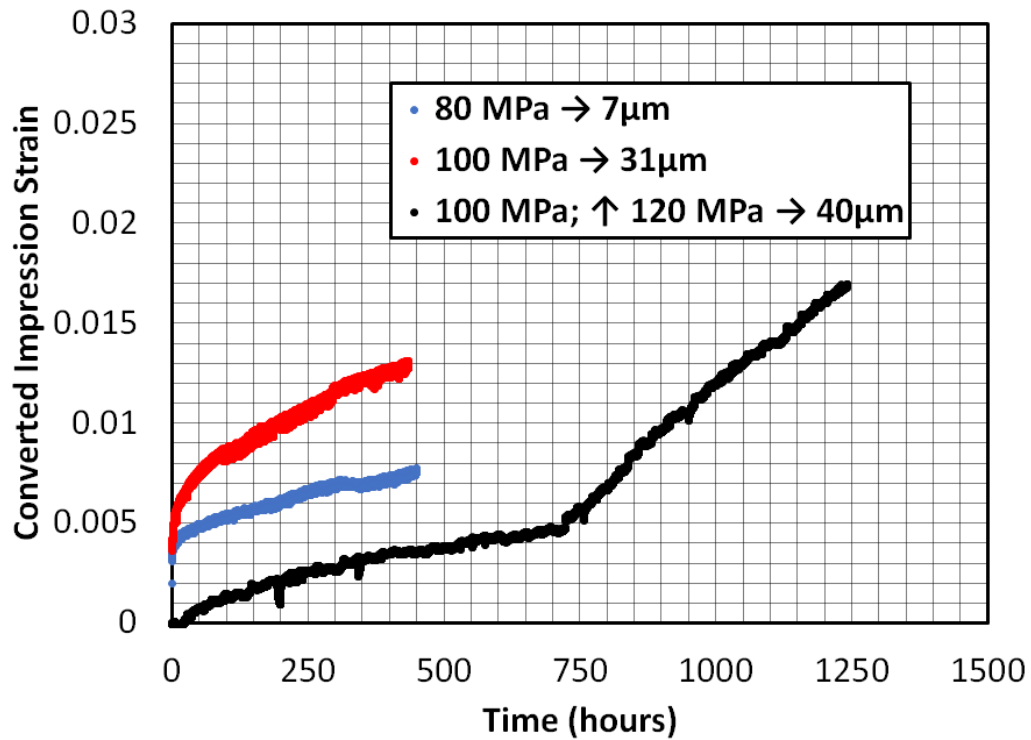


**Figure 2.** Impression Creep Metallographic Mounting (left) and Macro Image of Hardness Mapping Underneath Indentation Surface after Testing (right).

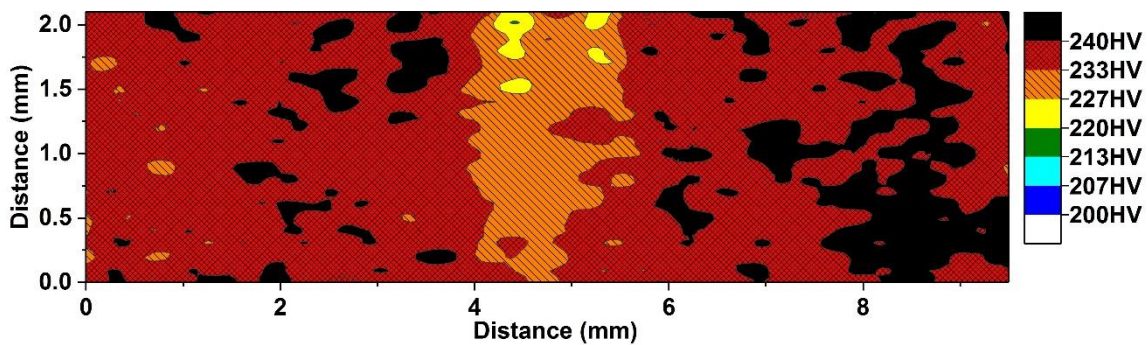
## 3. Results

### 3.1. Results for Normalized and Tempered Martensitic Grade 91

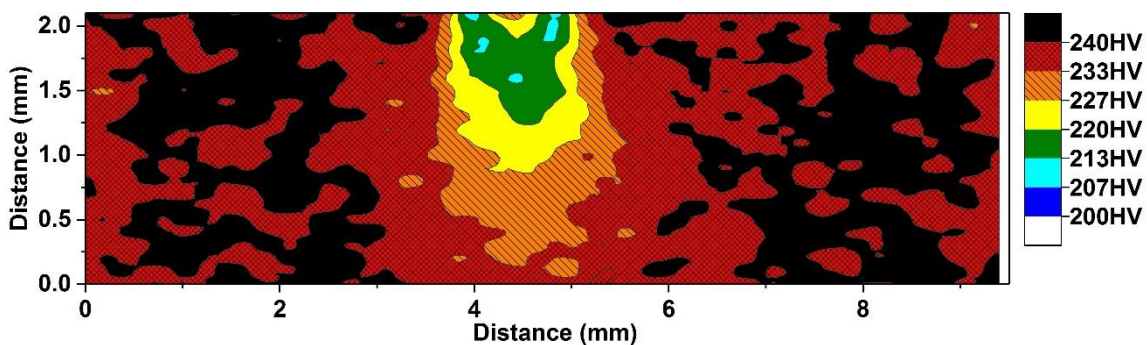
Three different impression creep tests were performed on the normalized and tempered martensitic Grade 91 material. Tests were conducted at 625°C and tested between a variation of stresses (80 to 120 MPa). The impression creep results are shown in Figure 3. The indentation depths for each test were measured using both the test results from the machine data files and confirmed with measurements from a confocal laser microscope (Keyence VK-X105). The measured depths were determined to be 7 $\mu$ m, 31 $\mu$ m and 40 $\mu$ m. It should also be noted that the 80 MPa test showed a decrease in strain around 300 hours because of a loss in furnace power. After testing, the samples were cut in half, mounted, polished and then tested using an automatic Vickers hardness tester (LECO AMH43). The hardness data is presented as color contour plots, shown in Figure 4 through 6.



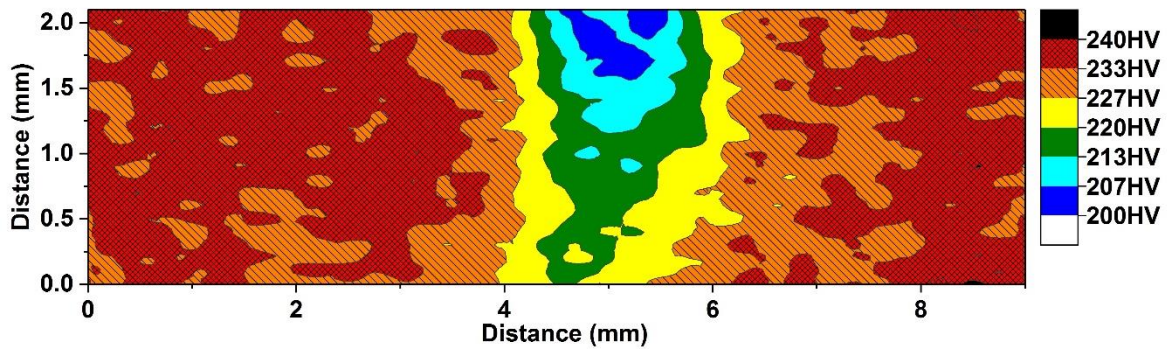
**Figure 3.** Comparison of Impression Creep Tests of a Normalized and Tempered Martensitic Grade 91 at 625°C and a Variety of Stresses (80 to 120 MPa)



**Figure 4.** Hardness Plot of a Cross Section through an Impression Creep Sample of Tempered Martensitic Grade 91 Tested at a Temperature of 625°C and Stress of 80MPa; Overall Indentation Depth is 7  $\mu$ m

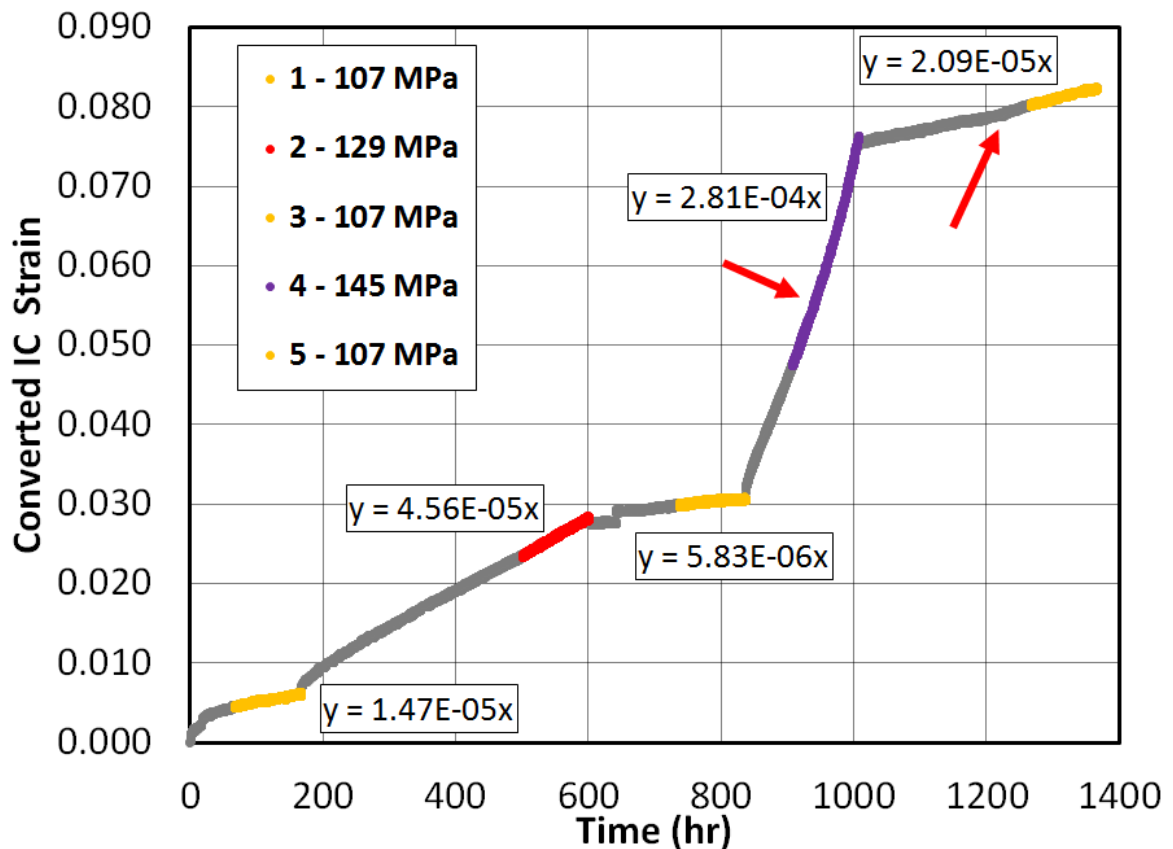


**Figure 5.** Hardness Plot of a Cross Section through an Impression Creep Sample of Tempered Martensitic Grade 91 Tested at a Temperature of 625°C and Stress of 100MPa; Overall Indentation Depth is 31  $\mu$ m

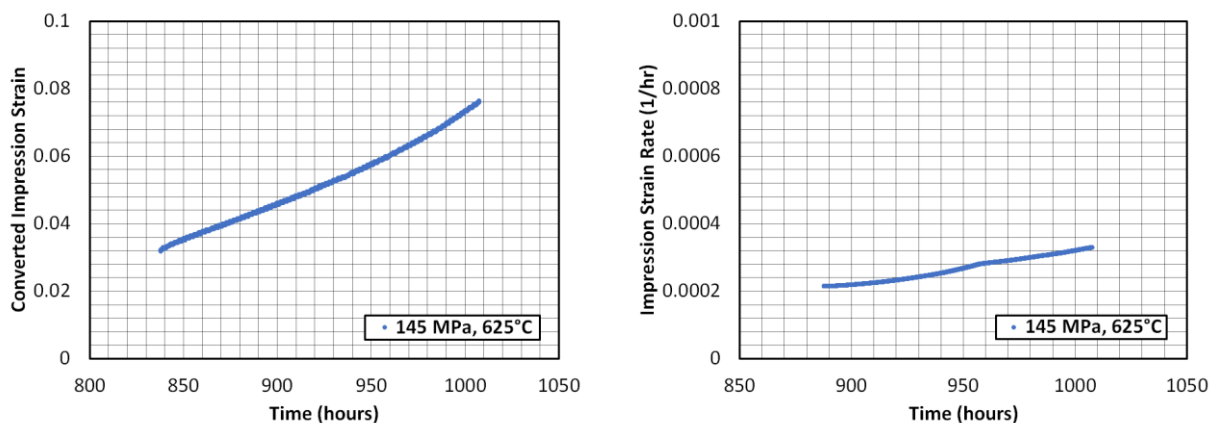


**Figure 6.** Hardness Plot of a Cross Section through an Impression Creep Sample of Tempered Martensitic Grade 91 after Stepped Loads from 100MPa to 120MPa at a Temperature of 625°C; Overall Indentation Depth is 40  $\mu$ m.

Another normalized and tempered martensitic Grade 91 specimen was tested at a variety of loading conditions. The test started at 107 MPa for about 200 hours, stepped to 129 MPa for about 400 hours, then stepped back to 107 MPa for another 250 hours before finally being stepped to 145 MPa for about 175 hours. This is shown in Figure 7. The impression creep strain accumulated before the step to 145 MPa was about 0.030. The next step, 145 MPa, is shown in Figure 8.



**Figure 7.** Impression Creep Curve for Stepped Load Test (625°C) of Normalized and Tempered Martensitic Grade 91 (107 to 145 MPa).

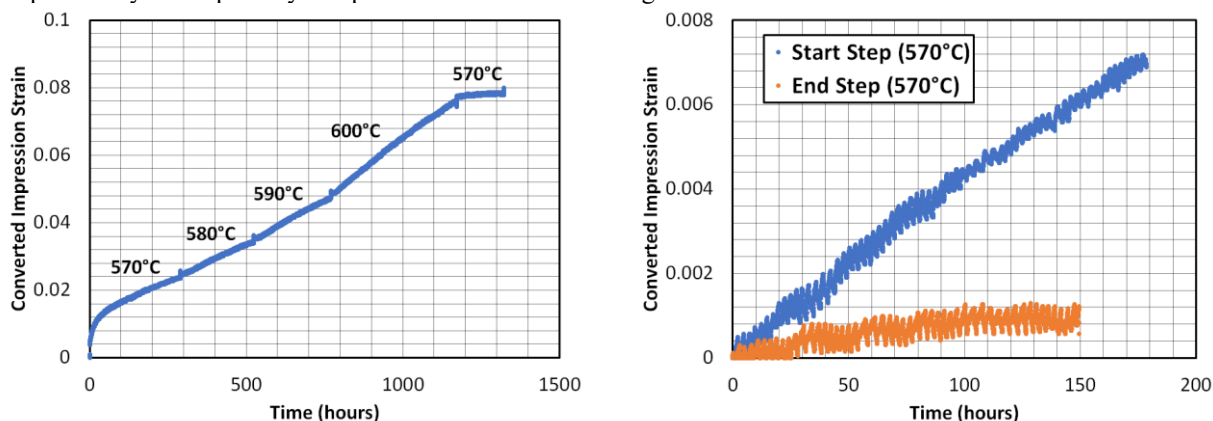


**Figure 8.** Impression Creep and Strain-Rate Curves for Stepped Load to 145 MPa (625°C) of Normalized and Tempered Martensitic Grade 91 after a Previous Strain History of 0.030.

The step load to a stress of 145 MPa clearly shows an initial steady-state creep rate that slowly increases with time. The reported strain rates were calculated using  $\pm 50$ -hour time increments. A  $\pm 50$ -hour increment that occurs 50 hours before the test terminates would be equivalent to the average rate in the last 100 hours of testing. It should also be noted that the testing stress of 145 MPa at 625°C is very close to reported yield stresses, which may be a possible reason for increasing strain rate behavior.

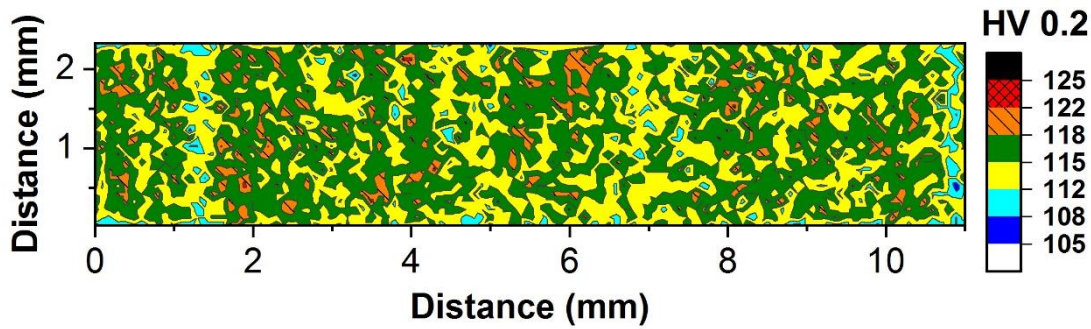
### 3.2. Results for Step Temperature Change in Service Exposed Grade 22

Service exposed Grade 22 base metal from a hot reheat piping system was tested at a constant stress of 80 MPa, but by stepping the temperature in 5 different increments. The test was initially started at 570°C for 300 hours, before being stepped up in 10-degree increments, up until 600°C. The final step was reduced to 570°C. The results are shown in Figure 9. The units for the comparison of the initial and final steps at 570°C are for comparison purposes only. The “primary creep zone” in the initial loading is also removed to better illustrate the difference.



**Figure 9.** Impression Creep Results for Service Exposed Grade 22 Base Metal Tested at a Constant Stress of 80 MPa (left); Temperatures Stepped in Increments of 10-degrees (570°C to 600°); Comparison of Initial and Final Step at 570°C (right).

Although hardness testing was not performed on the stepped temperature test above (Figure 9), a hardness test was conducted on another impression creep test of the same service exposed base metal. To date, observations of hardness tests have shown that Grade 22 does not exhibit strain softening or hardening behavior under impression creep loading. The example of this is shown in Figure 10 below.



**Figure 10.** Hardness Plot of a Cross Section through an Impression Creep Sample of Service Exposed Grade 22 Base Metal Tested at 80MPa and a Temperature of 570°C; Overall Indentation Depth is 86  $\mu\text{m}$ .

#### 4. Discussion

In martensitic steels, it has been observed that softening behavior is due to the disappearance of many of the microstructural subboundary characteristics and a decrease in the dislocation density in cyclic fatigue tests [4]. High temperature tensile tests at low strain rates, below  $2.5 \times 10^{-4} \text{ s}^{-1}$ , have also confirmed the existence of a softening stage. Microstructural observations have revealed that softening mechanisms are based on annihilation of subgrain boundaries and mobile dislocations [4-6]. Impression creep results in this paper have shown that softening also manifests in compressively loaded samples at high temperatures and low strain rates in some alloys, such as martensitic Grade 91. This is particularly important when step loading tests are considered, as the material may have a reduction in overall creep strength with each increasing step and this can lead to faster creep rates than would have been measured on virgin material (e.g. in the first step). The last two steps in the impression creep test shown in Figure 7 show increasing strain rates, which were tested at higher indentation depths from prior loading history. Results in Figure 8 show a steady-state creep rate that slowly increases over time at the stress of 145 MPa, which may be due to excessive softening at such high impression depths. This also indicates that there may be an applicable range of indentation depth, regardless of step-up/step-down tests. The maximum level of softening that occurs in comparison to uni-axial creep testing of the same material is currently unknown. Further interrogation of these samples will provide a better explanation of the accelerating strain rates that occur. Microscopy evaluations and hardness testing will be conducted on this sample to confirm softening behavior.

The stepped temperature tests shown in Figure 6 showed that a sudden reduction in temperature (step down) caused creep rates to be drastically lower than the initial rates in service exposed Grade 22. The sudden reduction in strain-rates may be due to previously increased dislocation pile-ups at the much higher strain rates. To overcome the increased resistance to dislocation movement, it may be that only step-up tests can be performed.

If the impression creep test is to be used in a quantitative manner, these questions must be addressed. There appears to be competing mechanisms that are occurring and step testing has further complicated the matter. The intent of this paper is to raise concern over potential problematic areas in impression creep with the goal of finding the true limitations of the testing technique.

#### 5. Conclusions

The findings in this paper have shown that there are problematic areas in stepped tests at certain testing conditions and changing material properties. It is important to highlight these findings, as stepped impression creep testing has been used for all materials. These findings are a first attempt to question the validity of stepped impression creep tests in all materials, as there appears to be limitations, specifically in martensitic Grade 91. Impression creep tests to various indentation depths have shown that strain softening behavior exists for normalized and tempered Grade 91. Softening mechanisms have also been shown in previous literature for a variety of martensitic steels. Additional hardness testing will be conducted for a variety of materials to better understand softening and hardening mechanisms in impression creep testing. The stepped temperature test for Grade 22 showed a significant difference in strain-rates between the initial and final step. It is thought that increased dislocation pile-ups are caused at previous high strain rates, thus lower strain rates are due to the difficulties of overcoming these dislocations. These findings have shown that careful considerations must be made before using creep rates obtained from multi-stepped loaded tests in situ of single loaded tests.

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## References

1. Sun, W, et al. "Application of Impression Creep Data in Life Assessment of Power Plant Materials at High Temperatures." *Proceedings of the Institution of Mechanical Engineers, Part L: Journal of Materials: Design and Applications*, vol. 222, no. 3, Dec. 2008, pp. 175–182.
2. Hyde, T.H.; Sun, W. (2001). Multi-step Load Impression Creep Tests for a 1/2Cr1/2Mo1/4V Steel at 565°C. *Strain*, 37(3), 99-103.
3. ECCC Guidelines for Impression Creep Testing; Discussion at 3th International ECCC Conference, 2014.
4. Eisentrager, J.; et. al, Analysis of Temperature and Strain Rate Dependencies of Softening Regime for Tempered Martensitic Steel, *SAGE Journals*, Volume 52, Issue 4, Pages 226-238.
5. Alsagabi, S.; Shrestha, T.; Charit, I. High temperature tensile deformation behavior of Grade 92 steel, *Journal of Nuclear Materials*, Volume 453, Issues 1–3, 2014, Pages 151-157.
6. Wang, L.; Li, M.; Almer, J. In situ characterization of Grade 92 steel during tensile deformation using concurrent high energy X-ray diffraction and small angle X-ray scattering, *Journal of Nuclear Materials*, Volume 440, Issues 1–3, 2013, Pages 81-90.