Commercialisation of impression creep testing

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Abstract: Impression creep testing is a technique in which the deformation resulting from load applied via a rectangular indenter can be converted relatively straightforwardly into a proxy for creep minimum strain rate. This offers a valuable route to assess the creep performance ranking of in-service high temperature plant materials for a number of reasons: the small specimen size makes extraction feasible without significantly affecting the structural integrity of plant; the possibility to test a single specimen at several stresses or temperatures enables multiple assessments; and, increasingly, the maturity of underlying technical understanding and quality of results increases confidence in the technique. However, the method is not without challenges, in particular the capital and running costs associated with servo-electric test rigs. Development of a bespoke deadweight loaded testing system at Wood (formerly Amec Foster Wheeler) has enabled commercially sustainable impression creep testing, which has been successfully applied to ex-plant Grade 91 steel.

Keywords: impression creep; creep strength; small specimen testing; strength ranking; lifetime assessment; Grade 91 steel; 316H stainless steel; deadweight loading; commercial testing.

1. Introduction

The proposition to assess material creep properties and thus remnant life from small scale specimens is clearly attractive to high temperature plant operators, since it is possible to extract and test material without significantly affecting the structural integrity of in-service plant. However, there are some concerns regarding the transfer of such techniques into the commercial sector. Reservations broadly fall into two categories: underlying technical understanding of test methods and interpretation of results; and equipment, costs and other commercial drivers. Research into various techniques has been ongoing for many years [1]; several are now in the infancy of their application. Wood has focused on consolidating the considerable foundational understanding of the impression creep test technique, with the aim of developing the method to a level of maturity that enables initial sustainable commercial application.

2. Background to impression creep testing and potential advantages

There have been significant efforts on the provision of a sound theoretical and practical understanding of impression creep testing; much of this work has been led by Nottingham University in collaboration with UK power utilities, especially RWE npower [1, 2, 3, 4].

Impression creep offers an alternative to conventional uni-axial creep testing to assess remnant life and determine material creep strength, using small-scale specimens. Typically, these specimens are 10x10x2.5mm, or 8x8x2mm, and so can be machined from samples extracted from plant components using scoop sampling or other on-site techniques. Importantly, when underpinned by appropriate engineering critical assessments (ECAs), specimen extraction need not significantly reduce the structural integrity of in-service plant.

A constant compressive load is applied to the centre of the specimen face, across the full width, via a 1mm or 0.8mm wide (depending on specimen geometry, above) rectangular indenter platen. It is important the indenter material has a significantly higher creep strength than the specimen. Thus for testing conventional power plant steels, the indenter has been produced from highly creep resistant nickel base superalloys (e.g. Nimonic 115) and no clear evidence for deformation of the indenter following testing has been observed. Generally, in laboratories around the world, this loading is carried out using servo-electric test frames [5]; Wood have also successfully designed, built and commissioned a bespoke deadweight-loaded impression creep rig, which is described below.

The indentation deformation in the specimen is measured throughout the test by high temperature extensometry; in Wood’s laboratories, this uses a side-loaded extensometer with twin-arm ceramic probes mounted immediately adjacent to the top and bottom of the specimen and indenter, as shown in Figure 1.
Figure 1. The impression creep testing arrangement used at Wood.

The specimen is heated to the test temperature; in Wood’s arrangement using a three zone furnace and the temperature monitored with two calibrated N-type thermocouples, mounted vertically adjacent to the specimen on the loading bars. Specimen temperature is thus monitored and controlled throughout the test. Following a soaking time to permit the temperature to stabilise, typically one hour, the full test load is then applied to the specimen and the deformation measured.

Following loading and full mating of the indenter and specimen, a constant rate of indentation is achieved, typically within ~400 hours. This indentation rate, by convention measured over the final ~100 hours, has been correlated to the equivalent uni-axial creep minimum strain rate using conversion factors related to the specimen geometry, derived from finite element analysis (FEA) [4] and hence permitting direct comparisons. Thus the equivalent uniaxial stress is given by:

$$\sigma_{IC} = \eta P,$$

(1)

where P is the pressure under the indenter (MPa) and \( \eta \) is a specimen geometry specific constant derived from FEA. Similarly the equivalent uniaxial strain rate is given by:

$$\dot{\varepsilon}_{IC} = \frac{\dot{\Delta}_{IC}}{\beta d},$$

(2)

where \( \dot{\Delta}_{IC} \) is the linear indentation deformation rate (mm/hr) measured in the impression creep test, \( d \) is the indenter width (mm), and \( \beta \) is a specimen geometry specific constant derived from FEA. The equivalence between impression creep and uni-axial creep is only between impression creep linear deformation rates and uni-axial creep minimum strain rates; no inference can be made more generally between impression creep data and uni-axial strain.

Typical values of the constants for the specimen geometries and indenter widths listed above are \( \eta = 0.43 \) and \( \beta = 2.18 \) [4]. It is worth noting that if the specimen geometry or material are changed significantly then further FEA is required to obtain the necessary constants for the correlations.

No formally recognized standards currently exist, but a code of practice [2] has been produced in an attempt to clarify the requirements of testing equipment and encourage a united approach to testing and subsequent analysis.

Once a stabilised indentation rate has been achieved and the associated equivalent uni-axial creep minimum strain rate derived, it is then possible to change either the test temperature or stress without interruption. Continued measurement of the indentation rate then provides additional analyses under alternative conditions; typically this can be repeated after 150 hours. Both changes in stress and temperature have been successfully applied to impression creep tests.

Recent tests at Wood have generally lasted 800 hours, comprised four ‘steps’, and provided four creep rate data points from each specimen, although some have produced up to eight. It is possible to continue testing until the total indentation deformation is approximately 10% of the specimen thickness, when it becomes clear the conversion to equivalent uni-axial creep data becomes invalid. An example of such an indentation vs time trace, taken from a recent test at 600°C and equivalent uni-axial stresses from 80 to 110MPa, on 100% ferritic Grade 91 material extracted from a combined cycle gas turbine plant component in the UK, is shown in Figure 2. It should be noted
that the four impression creep steps, each giving equivalent uni-axial creep minimum strain rates from which to assess material strength, were measured in approximately 800 hours, a time that could typically be required for an initial conventional creep rupture trial (or “sighter”) test.

Figure 2. Four steps of indentation deformation scaled by geometric conversion factors such that the gradients of the coloured linear regions are equal to the equivalent uni-axial creep minimum strain rates, for the various equivalent uni-axial stress applications.

An example of changing temperatures during a number of impression creep specimens of ex-service (from UK nuclear power generation plant) austenitic 316H stainless steel is shown in Figure 3. Here tests were commenced at 525°C but once a uniform indentation rate was achieved, then the temperature was reduced to 480°C. The equivalent uniaxial creep rates were determined for each step, as noted above. This work showed consistent reductions in creep rate associated with the temperature reductions, as might be anticipated. Where the temperature was subsequently returned to the initial 525°C, then the creep rate was found to return the original creep rate.

A further potential benefit for impression creep, is the ability to evaluate specific regions of metallic structures, for example welds and their associated heat affected zones (HAZs).

These examples demonstrate a significant commercial benefit of the technique over conventional creep testing: the ability to produce results relatively quickly and for multiple loads and temperatures from a single sample.

Figure 3. The equivalent uniaxial deformation behaviour for ex-service 316H stainless steel material, subjected to impression creep studies at 525°C, along with some temperature change trials to 480°C.
3. Commercial applications for impression creep testing

Assessment of in-service plant to determine remnant life is of obvious significant industrial interest, particularly to high temperature plant operators, where plant life extension or confirmation of existing plant life is needed. Impression creep testing offers an approach to such an assessment. An example of this type of work is the assessment of Grade 91 9Cr steel, one of the most widely used of a class of so-called Creep Strength Enhanced Ferritic (CSEF) steels within conventional power generation plant components in the UK and abroad. This material has been the focus of much study using impression creep testing [3, 6, 7]. One of the reasons for the interest in assessing this material has been the finding that Grade 91 steel in service in the UK was introduced in a number of microstructural conditions. In addition to the preferred martensitic structure, aberrant ferritic and mixed martensitic/ferritic microstructures have been observed [8]. The weaker ferritic material is worthy of particular scrutiny, since it severely limits creep performance. The widespread occurrence of this type of material has been established and poses a significant challenge to many plant operators [9].

For the assessment of remnant life the impression creep test is used to generate equivalent conventional uniaxial minimum creep rates. In turn these can be used with a Monkman-Grant type relationship to determine a rupture life, with the equation:

\[
\dot{\varepsilon}_{\text{min}} \cdot t_r = C,
\]

where \(\dot{\varepsilon}_{\text{min}}\) is the minimum creep rate (mm/mm/hr), \(t_r\) is the rupture life (hr), and \(\alpha\) and \(C\) are constants. The use of this approach has been validated by the undertaking of paired conventional creep rupture testing alongside impression creep tests [10]. A reasonably well established Monkman-Grant relationship for Grade 91 steel exists, based upon conventional creep testing, and this has been modified to give an Impression Monkman-Grant relationship, which has been shown to hold for impression creep testing of both martensitic and aberrant ferritic Grade 91 steel [11]:

\[
\dot{\varepsilon}_{IC} = 0.004575t_r^{-0.7391},
\]

where \(\dot{\varepsilon}_{IC}\) is an equivalent uni-axial creep minimum strain rate derived via Equation 2 from an impression creep test (mm/mm/hr), and \(t_r\) is the equivalent uni-axial creep failure time (hr). This then permits the prediction of a conventional creep rupture life but based upon the much shorter duration impression creep tests. At the same time the fit of the experimental data to the established Monkman-Grant relationship can be determined and if acceptable, can provide confidence in the use of this approach. Where the correlation between the experimental and literature equations is not strong, further work is required to determine a better fit to the test data.

Clearly equations (3) and (4) only give rupture lives for the test temperature but the relationship between stress and life is also required to enable the impression creep data to be utilised more widely to assess plant specific creep rupture life times. Again with Grade 91 this has been enabled by an existing relationship between the rupture life, mean stress and temperature [12] of the form:

\[
\log t_r = \left( 263.8 + 217.1 \log \sigma - 212.8(\log \sigma)^2 + 84.9(\log \sigma)^3 - 13.2(\log \sigma)^4 \right) (T - 438.7)^{-0.27} - 55.9
\]

where \(t_r\) is the rupture life (hr), \(\sigma\) is the mean stress (MPa) and \(T\) is the temperature (K). At the impression creep test temperature the result of equation (5) can be compared against the experimentally derived rupture data, as shown in Figure 4. Thence the stresses from equation (5), representing the mean performance of Grade 91 can be fitted to the experimental data to establish the level of difference in strengths between the experimental and predicted mean behaviour; in this case mean \(-41.6\%\). This then allows equation (5) to be used to plot stress vs rupture time at more relevant plant operating temperatures and compared with the maximum stresses in the components of interest, hence the remaining creep endurance can be established and the potential further plant operating time.
This type of assessment of creep strength relative to a known mean performance via impression creep test data can be powerful evidence for safe plant operation, and the ability to test a single specimen at multiple stresses or temperatures, maximises the value of extracted material. Where multiple specimens are available and can be tested over the same range of stresses, then such data should provide confirmation of a consistency in creep performance and confidence in the assessment of that material. To garner this type of understanding from conventional uni-axial creep testing, or other small-scale techniques, would require several tests on material from the same cast, which is not always commercially viable or indeed possible.

As with any small-scale test technique in relative infancy, concerns exist surrounding reliability of generating results and experimental scatter. However, impression creep testing is well placed to investigate and confront these concerns, since it is practical to examine creep properties of a single specimen at several conditions. Moreover, it is possible to reassess these properties by repeating steps from earlier in the test, for example by testing sequentially at 90MPa, 100MPa, 110MPa and then at 100MPa again. Previous work [13] has demonstrated that results from each step in a test are independent of previous steps, and so repeat steps are inherently expected to provide repeat results. This is a useful facet of the technique that simultaneously builds confidence in, and provides some measure of scatter of results.

There are other potential commercial issues that drive the use of small scale testing techniques, including the ability to evaluate the limited material available in the early stages of material development activities, however, some caution is required in this area, as illustrated below in section 4.2.3.

4. Challenges to impression creep testing

4.1. Commercial challenges

Given the potential technical benefits of the impression creep test technique, and the level of maturity of underlying theoretical understanding described above, there is potential for industrial (e.g. the power utilities) appetite in increased application of the method. It is anticipated that the scope of this work will be beyond the interest of most academic and dedicated research organisations, and require take up by industrially focussed organisations, with conventional financial constraints on investment. Thus it is worth considering the financial credibility of an investment in an impression creep test station. Currently most of the impression creep test facilities have been developed around either existing servo-electric load applications frames or new low load capacity servo-electric test frames. Table 1 shows illustrative costs of procuring a new servo-electrically driven impression creep testing system. A total investment of £42,500 – 51,500 could be required to provide such a facility.
Table 1. Illustrative break-down of costs for a new commercial servo-electric impression creep facility.

| Equipment                                      | Typical cost (£) |
|------------------------------------------------|------------------|
| Low load capacity servo-electric test frame    | 20,000           |
| Furnace and controller                         | 12,000           |
| Extensometer, sensor and conditioning unit     | 6,000 – 15,000   |
| Loading bars and indenter                      | 3,000            |
| Data recording system                          | 1,500            |
| **Total cost**                                 | **42,500 – 51,500** |

Set against these investment requirements, is potential income. Given the relationship to uni-axial creep tests, an initial assessment of potential income could be considered on the basis of current conventional creep strain measurement at similar temperatures (and excluding any specimen manufacturing and data reporting costs). This approach is likely reasonable given the tendency for conventional creep to set industrial expectations for impression creep testing. Recent commercial charges from a number of continental European laboratories for standard creep tests in the temperature regime of 500 – 650°C, including extensometry, indicated an hourly running rate of up to £0.70/hour. Additionally, there are costs associated with setting up creep tests; this could be up to ~£2000/year, assuming 4 – 5 tests are set up in a year. At these indicative rates, the earning capacity of a conventional creep test station would be ~£8,200/year. If it is assumed that a creep test achieves 10% profit, then a commercial organisation could expect to re-coup ~£820/year.

At a similar income level of current conventional creep testing then, it would take over 50 years to re-pay the capital investment in a new impression creep test facility. Even if higher profit levels were achieved (e.g. 20%), pay-back periods would still be significantly longer (e.g. 25+ years) than commercial organisations might require. With conventional financial constraints then, it would clearly not be feasible to make the investment required to produce a new test facility. The shorter duration of impression creep tests may allow for more test set-ups, which could supplement the testing income; this is still likely to be insufficient to achieve acceptable financial performance for most commercial organisations. A solution to this problem could be to increase the overall charges for impression creep testing.

Some consideration of aspects of the background to the current conventional creep test charges may be useful. Over the past two decades much commercial creep testing has been performed by laboratories that were originally set-up to support activities (e.g. material production, government research) other than commercial creep testing for third parties. However, as the requirement for the internal creep testing support within these organisations has diminished (e.g. as material production has declined), third party work has been attracted to supplement internal activities and help support the cost of the facility. In cases where the test facility already exists and is expected to be maintained, the charges levied for commercial creep testing for third parties did not need to reflect the level of original investment. Competition has driven prices down, to the point where one laboratory (now closed) was charging £0.14/hour for creep tests; which may not have even covered the cost of maintaining specimens at their test temperature. Such charging levels have inevitably started to set industrial expectations of costs but these suppressed prices are only sustainable whilst creep tests are required in a marketplace with excess capacity. There may be other factors that have also contributed to the relatively low current cost of creep testing but the above seems to be a strong contender to explain the testing charges now expected for creep. The unfortunate outcome now is the commercial difficulty of justifying investment in new creep equipment and modernisation of facilities. This background in conventional creep impacts small-scale creep test techniques, like impression creep, even if only by association and industrial expectation. There is thus a dual barrier to be overcome in the introduction of new creep assessments, with technical uncertainties but also financial difficulty in progressing the test technique from academia into widespread commercial operation.

To enable a more commercially practical return on investment than possible with servo-electric facilities, Wood has successfully designed, built and commissioned a bespoke deadweight loaded impression creep testing machine. This system, more akin to a conventional creep frame with an underslung lever and incrementally, manually loaded pan, incorporates a load cell to provide confidence that any frictional effects are overcome and the full test load is applied. Commissioning tests on a well-characterised cast of the 9Cr steel (Grade 91 - Bar 257) gave results consistent with those determined through servo-electric testing [5], as shown in Figure 5. Subsequent tests
on ex-service Grade 91 steel have confirmed this comparability with data previously generated using servo-electric loading frames.

Figure 5. Bar 257 impression creep test results using Wood’s deadweight loaded system and other servo-electric test frames.

4.2. Technical limitations

There are a number of potential limitations with impression creep testing, as with any small-scale technique, which must be recognised and which may inform the selection of the most appropriate test technique in different situations. Some of these issues are discussed below.

4.2.1 Compression testing

The impression creep test is only performed in compression and hence no fracture behaviour can be obtained. For materials with highly inhomogeneous deformation behaviour (especially brittle materials), alternative techniques to investigate tensile creep behaviour may be clearly advantageous.

The compressive nature of this test also leads to a limit on the depth of penetration of the indenter into a specimen where the deformation increases the effective load bearing area and hence reduces the applied stress significantly. It is currently thought that a maximum displacement of ~10% of the specimen thickness should be considered the practical limit for indentation penetration. There is not known to be an additional upper limit on test time, so long as the specimen material is unchanged due to exposure to high temperatures; tests at Wood have run for up to 2500 hours without any suggestion of test invalidity.

4.2.2 Correlation of indentation deformation to minimum creep rate only

There is a temptation to mis-interpret the indentation deformation vs time trace by associating the initial apparently reducing indentation rate (e.g. see Figure 3) with conventional uni-axial tensile primary creep deformation behaviour. However, FEA [14] has shown that the initial indentation deformation stages are more closely linked to small levels of mis-orientation between the indenter and sample surfaces, rather than primary creep. This is consistent with the observation that this initial stage of indenter deformation can be variable, even between nominally identical samples which produce similar equivalent uni-axial creep minimum strain rates. Impression creep does not provide a correlation to creep strain generally, only to the minimum creep rate of a material.

4.2.3 Material specific creep deformation behaviour

Wood has evaluated austenitic stainless steel 316H material using both uni-axial and impression creep testing. This material was tested in two conditions:
1. Virgin tube material evaluated after cold work and simulated post weld heat treatment, and
2. Ex-service tube material (from the UK nuclear power generation fleet).

Impression creep tests on the virgin tube material were undertaken at 525°C and a single equivalent uni-axial creep minimum strain rate was obtained from each specimen (i.e. no stepped stress tests). These creep rates are shown in Table 2 and produced no discernable trend. In some instances, increasing test stress resulted in apparently reduced deformation rates. Initially it was thought that this behaviour was associated with experimental scatter, although tests on other material, like Grade 91 steel, had shown that the general trends in deformation behaviour were well captured by impression creep testing.

Table 2. Applied equivalent uniaxial creep stress and creep rates for virgin 316H tube material, impression creep tested at 525°C.

| Equivalent uni-axial creep stress (MPa) | Equivalent uni-axial creep minimum strain rate (x10^-6/hr) |
|----------------------------------------|-----------------------------------------------------------|
| 240                                    | 0.20                                                     |
| 240                                    | 1.77                                                     |
| 240                                    | 2.64                                                     |
| 240                                    | 1.82                                                     |
| 280                                    | 0.90                                                     |
| 320                                    | 1.69                                                     |

The conventional creep deformation curves obtained from uni-axial creep tests on the same 316H tube material are shown in Figure 6. These reveal a double primary behaviour in a number of tests. Thus, the creep deformation behaviour in the initial stages of the tests was found to be highly variable, consistent with the unexpected and apparently misleading rates derived from the impression creep tests. The origin of the double primary behaviour in the virgin 316H material is not currently well understood, but may be attributed to microstructural instabilities in the as-manufactured material [15]. This hypothesis is to some extent substantiated by the uni-axial creep data obtained from the ex-service (i.e. thermally aged) material, which demonstrated a more conventional strain-time behaviour. Impression creep tests on this material are more readily understood, and the derived equivalent uni-axial creep minimum strain rates follow the ranking of applied stress, as demonstrated in Figure 3 for this material and consistent with the experience in Grade 91 steels.

Figure 6. Uni-axial creep deformation curves for virgin 316H material, some tests showing double-primary behaviour.

This experience emphasises the need to understand the conventional creep deformation behaviour of the test material before attempting to use relatively novel small-scale techniques. More generally, it may be that such techniques are best used to supplement, rather than replace, conventional creep testing.
4.2.4 Repeatability of data

There is currently limited direct experimental evidence of the level of repeatability, in terms of creep rates, that can be obtained from impression creep testing, thus this is an area for further development. Such information would be valuable in establishing the uncertainty of measurement for this technique.

5. Summary and Conclusions

There appear to be a number of motivating reasons to assess plant material using impression creep testing, in particular, the small specimen size and capability to test a single specimen at multiple conditions. Wood has attempted to resolve some commercial challenges associated with the method, and recognised some potential limitations. Development of a deadweight loaded testing arrangement has reduced costs sufficiently to enable commercially viable application of the technique. This has proved particularly successful in assessing ex-service Grade 91 steel in a variety of microstructural conditions from UK high temperature plant, and the underlying technical understanding of impression creep testing enables valuable creep strength assessment and remnant life determination.

Acknowledgments: The impression creep testing referred to in this paper was commissioned by the generating companies Scottish Power and EDF Energy, and the release of unpublished data is gratefully acknowledged.

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