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The effect of testing environment on small punch creep

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Abstract: Small punch creep testing (SPCT) is currently experiencing a resurgence of interest as a small-scale testing technique (SSTT) for a wide range of uses, including testing in-service components and materials with limited availability. SPCT is particularly pertinent for the fusion community since it is difficult to irradiate large volumes of material due to the high damage levels required and relatively low number of available facilities. An important aspect of the SPCT development that still requires investigation is the effect of the testing environment, which has been shown to impact the creep properties of materials when using standard testing techniques. This paper investigates the effect of using an air or an argon environment on the SPCT behaviour of the leading European fusion reactor material Eurofer97 at 550°C. The test environment was found to impact on the small punch creep behaviour: testing in an argon environment significantly increased time to failure and deflection at failure by a factor of approximately 30 and 7%, respectively. The test environment also appeared to affect the behaviour of the test via oxidation of the punch head which should be an important consideration in future testing.

Keywords: Small punch testing; small punch creep testing; small scale test techniques; fusion materials; creep; stress-rupture; testing environments; RAFM steels; Eurofer97

1. Introduction

Nuclear reactors pose extremely demanding environments for structural materials. Therefore, comprehensive and rigorous testing is required of nuclear structural materials before they can be considered fit and safe for service. For the new generation of fission reactors and fusion reactors, a new generation of materials is being developed to withstand even harsher environments. They will require a large amount of testing; but new materials are often not mass-produced by industry and so are often in short supply. This places great pressure on the amount of destructive testing that can be carried out. Expensive and long irradiation times also restrict the volume of irradiated material that research groups can achieve. This is of even greater significance for the fusion community. Materials for fusion reactors will require experimental neutron irradiations with a 14.1 MeV peak to simulate the high energy neutrons that are produced by the deuterium-tritium (D-T) reactions which will be the dominant reaction in future commercial fusion reactors. There is currently no facility capable of producing such high energy neutrons in any significant volume.

One testing facility planned to be capable of producing high-energy, D-T fusion-like spectra of neutrons is the International Fusion Materials Irradiation Facility (IFMIF). The target is only planned to be able to irradiate a volume of ~0.5 litres, within the high flux region ($>20\text{dpa}_{(\text{fpy})/\text{year}}$), with irradiation campaigns planned to run for upwards of 1 year [1-4]. This represents an extreme, but very real, example of the amount of testing material available being a critical problem. Therefore, the nuclear community is particularly interested in developing novel testing techniques that are able to properly assess material properties while using a much smaller volume of material.

Small punch testing (SPT) is a small-scale testing technique receiving significant interest as a potentially powerful method for measuring the mechanical properties of nuclear grade materials, including irradiated material. Small punch testing may be invaluable as a testing technique in many other fields too, where saving material is sought after. Small punch testing may also be used to measure in-service components; the small volume of material removed should mean that the components can continue service. In such a case SPT is considered a quasi-non-destructive test.

In SPT, a small, hard ball or hemispherical tip (punch) is driven through a small, thin cylindrical disc which is clamped between two dies. The resultant force-displacement or time-displacement curves can be used to derive analogous values of corresponding mechanical properties derived from standard uniaxial mechanical testing. The data is predominantly used on a relative scale though significant work is being conducted to achieve reliably derivable mechanical quantities. However, this is proving difficult; largely due to the tri-axial stress state present during SPT [5].

In this work, the SPT will be operated as a uniaxial creep equivalent – with the load on the specimen held constant and the curve of interest being displacement-time. There is not yet an established nomenclature for this, but it will be referred to in this work as Small Punch Creep Testing (SPCT). However, with the failure times seen in this study the test may be more relatable to traditional stress-rupture. It is also possible to test as uniaxial tensile equivalent by running in displacement control, or, utilising a slightly different experimental set-up, Small Punch Shear Testing.

It has been demonstrated that the testing environment can have significant effect on the creep behaviour of materials in uniaxial creep testing [6-9]. These mechanisms may well also be active in SPCT. Currently, limited literature on the effect of testing environment on SPCT has been conducted [10-12]. Though limited, the evidence seems to suggest that the testing environment can cause a significant effect, with tests in air rupturing after shorter test times. Further investigations are still required to validate the results and also to cast greater light on the scale of the environment’s impact and the mechanisms involved; both of which are likely to be material dependent. These are important concerns for the technique if it is to become a fully standardized process and is of particular interest for the purposes of testing nuclear fusion structural materials as the components will be operating in an [ultra-high] vacuum vessel (VV).

This study will investigate the effect of testing environment on the Reduced Activation Ferritic-Martensitic (RAFM) steel Eurofer97 at a test temperature of 550°C. Eurofer97 is the prime candidate structural material and 550°C is the accepted operational temperature for present He cooled breeding blanket designs in the future DEMO fusion reactor.

2. Materials and Methods

2.1. Eurofer97

Eurofer97 is a 9%Cr Reduced Activation Ferritic-Martensitic (RAFM) steel. It contains very low concentrations of long-term activated elements, which significantly reduces any long-term activation of materials from future fusion reactors. The full elemental composition is given in Table 1. It was provided in the form of a fully martensitic, 6.5 mm thick, rolled plate and was tested in the as-received condition.

Table 1. Elemental composition of Eurofer97, in wt.% [13].

	Cr	W	Mn	V	Ta	C	N	Si	Al
Min	8.5	1	0.2	0.15	0.1	0.09	0.015	-	-
Max	9.5	1.2	0.6	0.25	0.14	0.12	0.045	0.05	0.01
	Mo	S	P	Ni	Cu	Co	Nb	B	Fe
Min	-	-	-	-	-	-	-	-	Bal.
Max	0.005	0.005	0.005	0.005	0.005	0.005	0.001	0.001	Bal.

2.2. Optical and electron microscopy

The as-received microstructure was characterised with a TESCAN Field Emission Gun Scanning Electron Microscope. An Electron Backscatter Diffraction (EBSD) map was created using an accelerating voltage of 20 keV with a step size of 0.11 µm. Data acquisition used Oxford Instruments AZtecHKL software and post-processing was carried out with Oxford Instruments HKL CHANNEL5 software. Optical macrographs of the tested specimens were taken on an Olympus SZX7 stereomicroscope.

2.3. Small punch testing

2.3.1. Small punch test rig and set-up

Testing was carried out on UKAEA’s ODIN high temperature-test rig manufactured by Phoenix Materials Testing Ltd, Dudley; with a 20 kN actuator and 17-litre capacity chamber, see Fig. 1(a). Heating of the specimen was performed using an infra-red (IR) heating system that surrounds the dies. The temperature was measured via type N thermocouples that are inserted into holes in the upper die, resting close to the specimen and held in place with high-temperature wire tied around the die. The dies are made from H39 WM steel alloy.

2.3.2. Small punch creep testing of Eurofer97

Cylindrical discs of Eurofer97 with 8 mm diameter were cut by Electrical Discharge Machining (EDM), initially to a thickness of approximately 0.75 mm, with the longitudinal axis of the disc parallel to the short-transverse direction of the plate. The discs were then mechanically thinned to 0.5 ± 0.05 mm with a finish of P1200 grit from SiC paper, as in accordance with the CEN Workshop Agreement CWA 15627 [14]. Specimens were comprised from only material within the central 3 mm region of the 6.5 mm thick Eurofer97 plate to maximise microstructural homogeneity between specimens.

The upper die was tightened to a torque of 10 Nm, also in accordance with CWA 15627. All testing was carried out at 550°C and using a load of 675 N, after initial ramp up from 0 N at 2.5 N/s. However, specimens were tested in an air or inert-argon gas environment. The punch used was Ni-based Nimonic 90 alloy (Special Metals, UK) [15], with a hemispherical tip of 2.5 mm diameter, Fig. 1(b). The dies were machined with additional holes to allow the flow of air and argon to and from the region within the die and around the specimen, see Fig. 1(c). A different punch head was used for each environment, though both were ostensibly identical Nimonic punches.

For testing in the argon environment, the sealed chamber was purged with argon gas at a rate of 5 litres per minute (lpm) for 20 minutes at room temperature. The rate was then held at 1 lpm while the system heated up to 550°C. Then reduced to a low and steady flow rate <0.5 lpm. The argon testing also included four getters, small high-purity discs of titanium, placed in the chamber to react with oxygen remaining in the chamber.

After the test temperature was reached, all testing, regardless of environment was held at temperature for an hour before testing started, to allow the components of the testing rig to thermally equilibrate. The measuring apparatus was zeroed after this period before the test started.

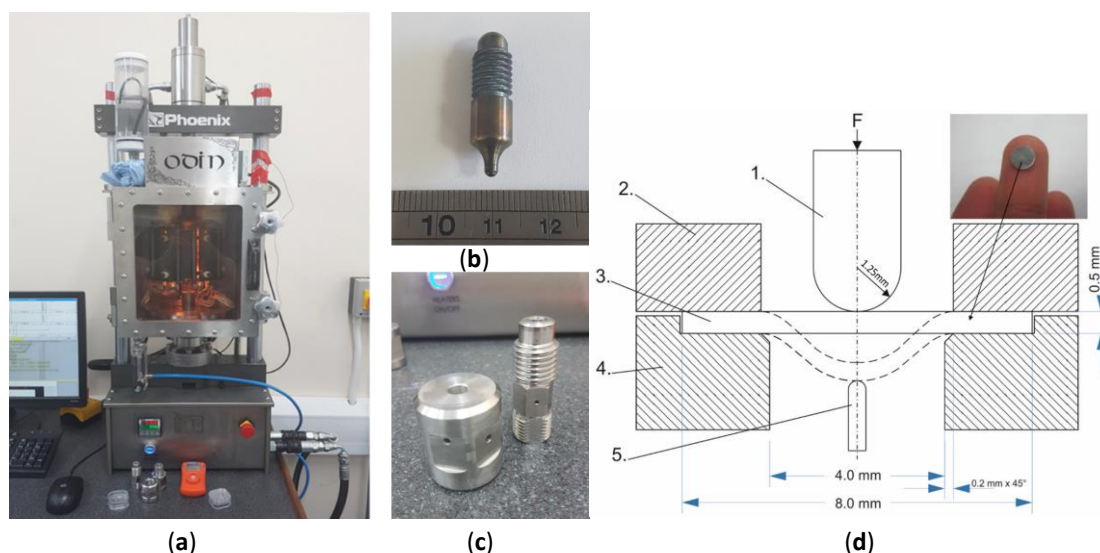


Figure 1. (a) ODIN small punch test rig; (b) Nimonic punch head used for testing. (c) H39 WM steel dies used for testing. The holes used for gas circulation and for housing the thermocouples can be seen. The upper die is on the left; (d) Schematic diagram of the test set-up. 1. Punch head, 2. Upper die, 3. Specimen, 4. Lower die, 5. Extensometer [14].

3. Results

3.1. Microstructural characterisation

A representative EBSD map of the as-received microstructure of Eurofer97, along the Rolling Direction–Long-Transverse (RD-LT) plane, is given in Fig. 2. This is the same face as is the circular face of the thin disk specimens. Thick black lines represent high-angle ($>10^\circ$) grain boundaries and thin lines represent low-angle ($>2^\circ$) grain boundaries. Noise reduction was applied in order to allow delineation of these boundaries. Zero solutions were replaced with the most common orientation found amongst their four nearest neighbours [16]. Eurofer97 contains a range of grain sizes typically 1-5 μm with no strong preference for orientation. The plane perpendicular to this map would be parallel to the 500 μm longitudinal axis of the specimen. Since this is perpendicular to the RD, we would

expect grains to be no larger than those in Fig. 2. Therefore, the through specimen thickness should contain at least 100 grains and constitute a representative testing volume.

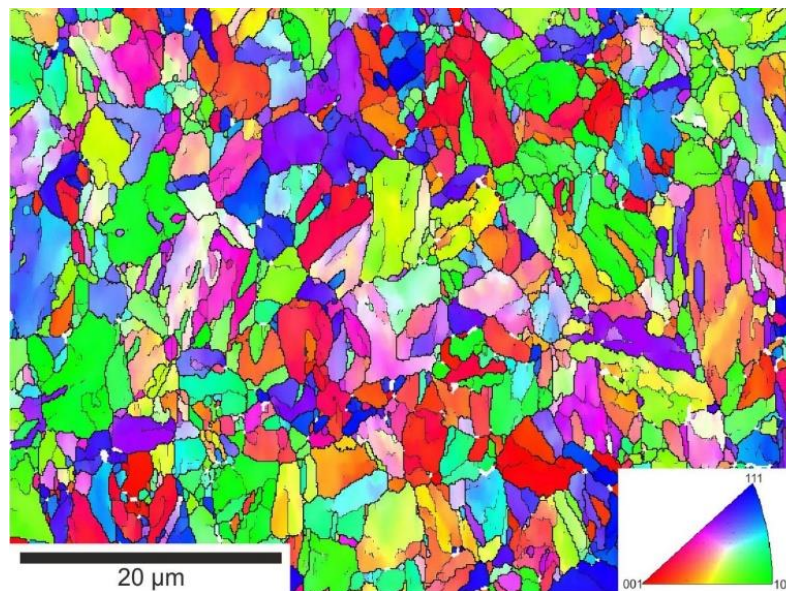


Figure 2. EBSD map of the Eurofer97 (RD-LT) and the associated Inverse Pole Figure (IPF). The map is of the RD-LT plane i.e. parallel to the top face of the plate. The reference axis (001) for the IPF is out of the plane of the page.

3.2. Small punch creep behaviour

The small punch creep behaviour was investigated by assessing three criteria: time to failure, deflection at failure and the minimum deflection rate. Deflection (δ) is defined as the change in position of the extensometer against the base of the sample as the specimen is deformed. Selecting start and end points of the secondary creep phase is somewhat arbitrary and therefore determining a reliable average creep rate is difficult. The minimum creep rate is a useful alternative; and was calculated using the following method. Utilising OriginPro software, the data range was set to exclude the majority of the primary and tertiary creep phases. A high order (9th) polynomial was then fitted to this data range. The differential ($d\delta/dt$) of this polynomial was calculated and plotted. The y-axis now represents the deflection rate and it follows that the minimum value is the minimum deflection rate. Fig. 3(d)-(f), illustrates the process used to derive the minimum deflection rates from the SPCT curves. Representative SPCT curves from both air and argon tests are also given, Fig. 3(a), along with the macrographs of the associated tested specimens, Fig. 3(b) and (c), respectively.

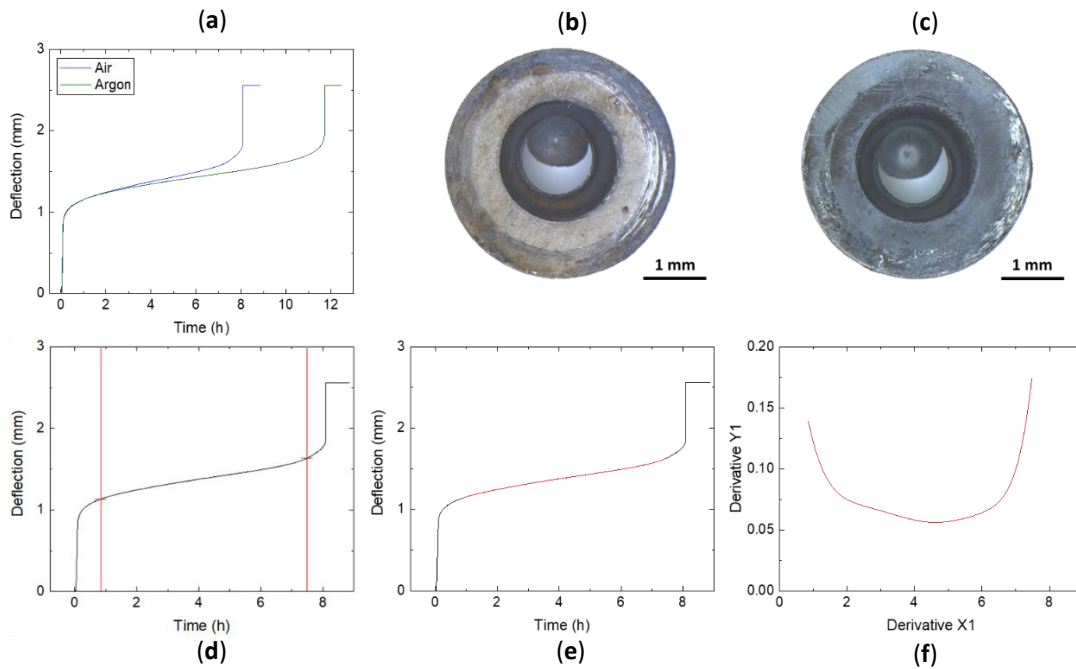


Figure 3. (a) Representative SPCT curves for the air and argon test environments; (b) Associated air tested specimen; (c) Associated argon tested specimen; (d) Representative example of the data range over which a polynomial was fitted in order to attain the minimum deflection rate; (e) The fitted polynomial, shown in red; (f) graph of the first order differential of the fitted polynomial. The minimum of this curve represents the minimum deflection rate; (d)-(f) are from the same air test data.

The results of the times to failure, minimum creep rate and deflection at failure are shown in Fig. 4 and Table 2. The results indicate that there is an effect of test environment on the small punch creep behaviour. The mean values for the time to failure and deflection at failure are larger for the argon test environment, while smaller for the minimum deflection rate. The error bars for the two environments do not overlap, implying that these differences in mean values are significant and therefore due to the environment rather than chance arising from scatter in the data. The error bars in the graphs represent the standard error of the mean.

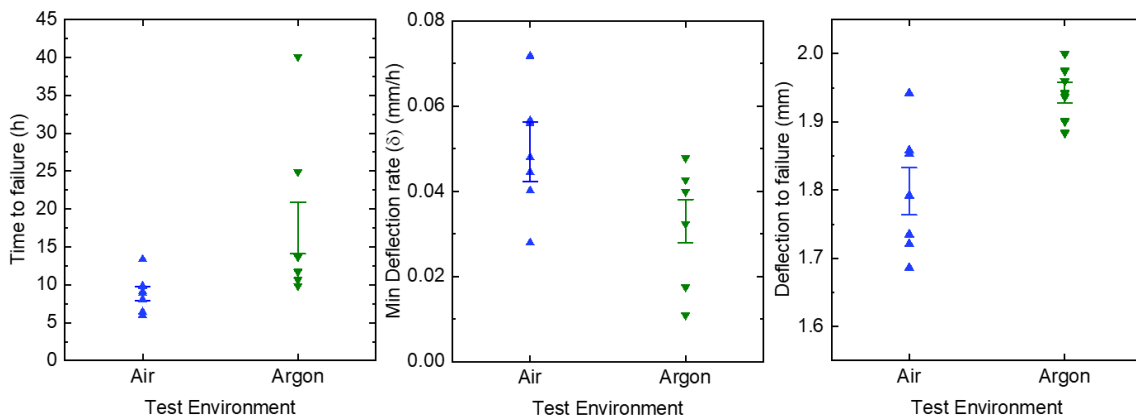


Figure 4. Graphs of results of the effect of test environment on (a) Time to failure; (b) Minimum deflection rate; (c) Deflection at failure. The error bar shown in each graph is the standard error of the mean.

Table 2. Summary of the SPCT results for air and argon test environments. The uncertainty is the standard error on the mean.

Environment	Time to failure (h)	Minimum deflection rate (mm/h)	Deflection to failure (mm)
Air	8.8±0.93	0.049±0.0070	1.80±0.034
Argon	18±3.4	0.033±0.0051	1.940±0.015

The results as a function of test number were plotted in order to help discern whether other factors may be influencing the results along with the different test environments; particularly since there appeared to be a relatively large range in the times to failure. See Fig. 5.

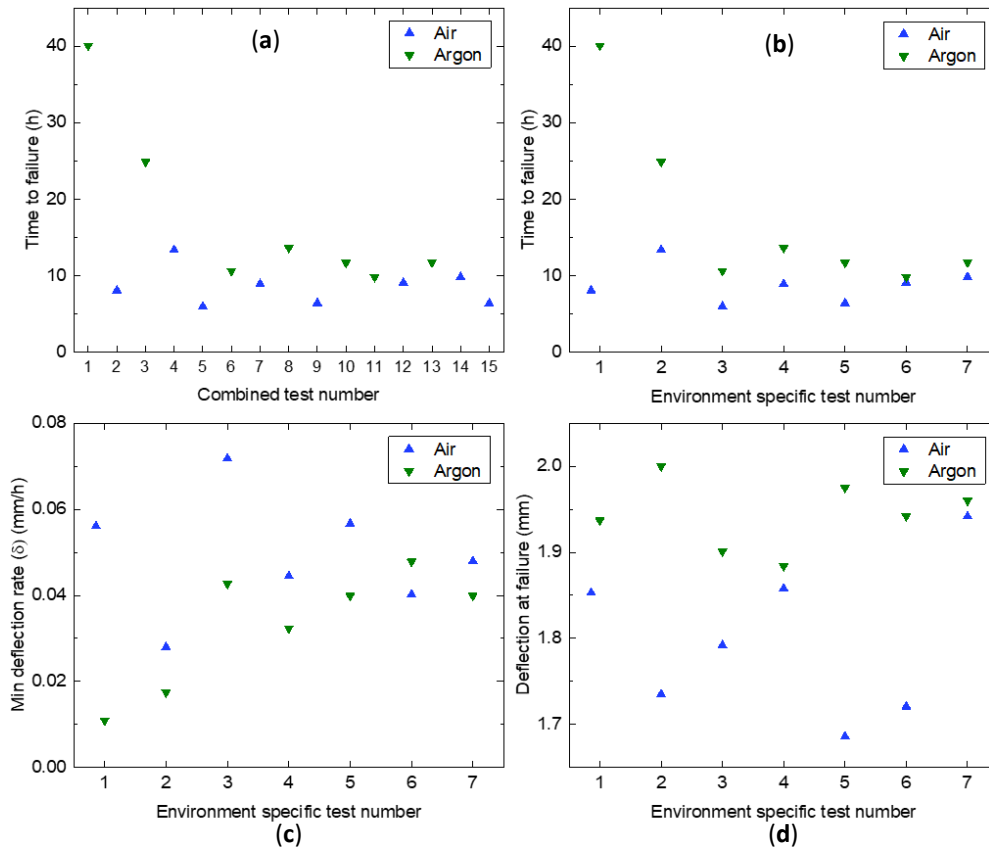


Figure 5. SPCT results as a function of their test number: **(a)** Time to failure as a function of combined test number, i.e. order the testing was carried out regardless of test environment; **(b)-(d)** as a function of the environment test number, i.e. counting only tests from the specific test environment; **(b)** Time to failure; **(c)** Minimum deflection rate; **(d)** Deflection at failure.

Fig. 5 does reveal a seemingly non-random evolution in the time to failure and minimum deflection with progressive test number for the argon environment. The first two argon tests have a time to failure that is much greater than the subsequent majority, with the first test being again significantly greater than the second. This effect is mirrored in the minimum deflection rate – with lower rather than higher values. The deflection at failure appears to be largely independent from this probable effect. The tests in air either do not share this time to failure evolution, or may do, but to a much lesser extent that one cannot discern from natural variation in the data. It is important to consider, however, that for the first two ‘anomalous’ argon tests, statistical variation cannot be ruled out and any systematic cause may simply be an illusion. Though due to the scale of this difference it seems unlikely.

The tests were deliberately run approximately alternately between argon and air. Therefore, any systematic change to the hydraulics, measuring equipment etc. of the rig seems unlikely. Likewise, the same dies were used in both environments and so any systematic change to the dies, either by oxidation, mechanical or dimensional changes seems implausible to be the cause – at least as the dominant cause.

Separate punch heads were used for each of the environments and therefore this is likely to have played a dominant role in the large decrease in time to failure observed in argon with successive testing. It is possible that a progressive change in oxidation could have caused a change in the observed mechanical behaviour, most likely due to a change in the coefficient of friction between the punch and the test specimen [12,17-19]. Nimonic 90 is an oxidation resistant alloy, so more specifically the bulk of the punch would not be oxidised but a chromate (Cr_2O_3) scale [20,21] would form on the surface. Therefore, the punch head should not experience significant softening, but the coefficient of friction would presumably change. Fig. 6 shows images of an unused punch (a) and the punch after a certain number of tests in argon (b)-(c). It is clear from the surface colour of the punch that it has oxidised despite the inert atmosphere. It should be noted that the chamber remained sealed and argon remained flowing until the chamber had cooled to [near] room temperature. Therefore, a post-test influx of air into the hot chamber with the still hot punch, is not the cause of the oxidation, but rather due to a small quantity of oxygen in the chamber despite the argon purge. It should also be noted that one preliminary test in argon was conducted using the same punch head, but the test used a different set of dies and therefore this test was not included in the data or in the test number counting.

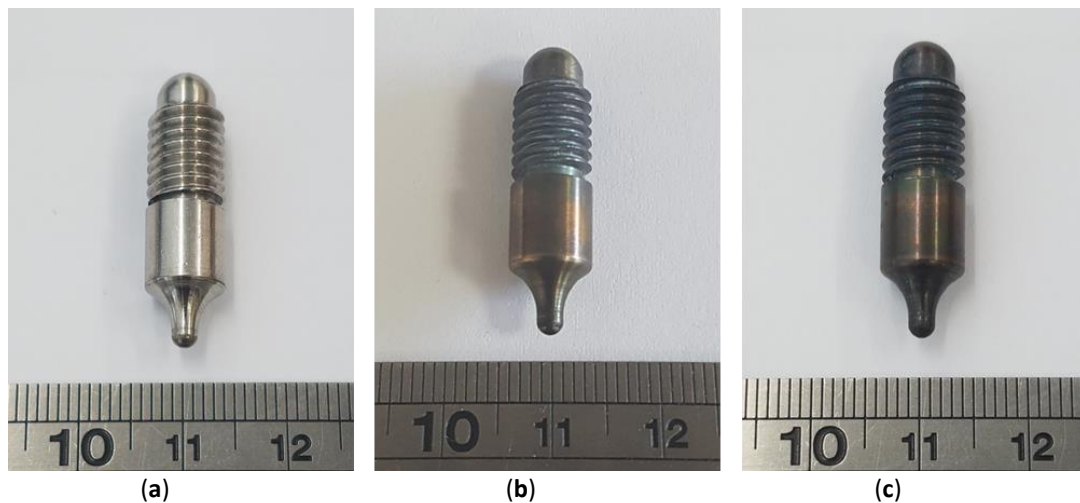


Figure 6. Nimonic 90 punch head: (a) Before testing; (b) After argon test no. 3; (c) After argon test no. 7.

Again, assuming that the sharp decrease in the argon time to failure is not down to natural variance in the data, a plausible explanation is that the relatively non-oxidised punch head in the first two argon tests, more notably for the first, has a different creep mechanism and significantly slower affiliated deflection rate. While testing in air, at high temperature, oxidation could happen quickly, and we would expect the punch to be near fully oxidised in the first test or perhaps before the test could begin during the equilibration stage. This would explain why the air test results were much more stable with successive testing. By test no. 3 for argon, the data suggests that the effect of changes to the punch head has stabilised. Therefore, the data has been re-represented with the first two argon tests omitted. This represents the results were the air and argon punch heads would be in a similar and pseudo-stable oxidation state, in which a comparison of the test environment's effect on tested specimens only can be investigated: see Fig. 7 and Table 3.

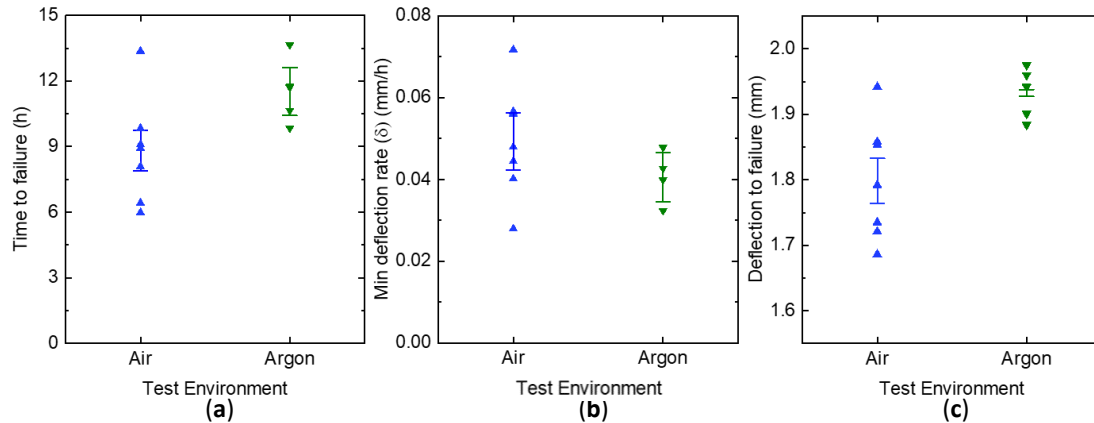


Figure 7. With the first data for the first two argon tests removed - graphs of results of the effect of test temperature on (a) Time to failure; (b) Minimum deflection rate; (c) Deflection at failure. The error bar shown in each graph is the standard error about the mean.

Table 3. Summary of the SPCT results for air and argon test environments after first two argon tests excluded. Again, the uncertainty is the standard error on the mean.

Environment	Time to failure (h)	Minimum deflection rate (mm/h)	Deflection at failure (mm)
Air	8.8±0.93	0.049±0.0070	1.80±0.034
Argon	12±1.1	0.041±0.0060	1.932±0.0053

The results now show that the test environment still has a significant effect on the small punch creep behaviour. The time to failure and the deflection at failure are significantly greater when Eurofer97 was tested in the argon atmosphere. The minimum deflection rate was slightly greater in air but not above the margin of error.

4. Discussion

Literature available to compare the results with is limited. Kobayashi et al. did not compare air with argon environments for SPCT, but rather air with vacuum and observed a significantly greater time to failure when the specimens were tested in vacuum [11]. This was attributed to the formation of an oxide scale that formed which for air i) introduced a significantly larger volume of the sample that was softened by oxidation and ii) lowered the coefficient of friction with the punch head. Modelling has shown that friction can play a significant role in determining behaviour of SPCT [17-19] with time to failure increasing with increasing coefficient of friction. It is possible that the lack of gas in the chamber may have also influenced the results due to the lubricating effect of the gas also affecting the coefficient of friction. Nishioka et. al observed that SPCT with a low argon flow experienced shorter times to failure than when a high flow rate was used [12]. It was also observed that different oxide films were formed under these conditions: an Fe-rich oxide for the low flow rate and a Cr-rich oxide film for the high flow rate. These reduced and increased the coefficient of friction of the specimen, respectively, which caused the difference in rupture times. Both aforementioned studies used 2.25Cr-Mo low alloy steel. While these are not too similar to the Eurofer97 of this study, similar mechanisms appear to be occurring. Auger et. al [22], working on 9% Cr T91 steel, also observed that a “low oxidised” condition produced a Cr₂O₃ surface oxide, while those that were “air-oxidised” produced Fe₂O₃ on the surface.

Optical macrographs of the tested specimens showed that the specimens tended to have a different appearance depending on which test environment was used. The optical macrographs are shown in Fig. 3(b)-(c) and Fig. 8. The air tested specimens tended to have a ‘flaky’ grey scale while the argon tested specimens tended to be more dominated by a better adhered blue coloured scale. The air tested specimen in Fig. 3(b) may be misleading in its relatively smooth appearance and probably represents a specimen where most of the scale has flaked off. Further macrographs are provided in Fig. 8. The grey flaky scale is presumably iron oxide while the bluer scale is chromium oxide scale. Future investigations will aim to confirm this and also investigate the through specimen thickness of

the oxidation layers created during testing. The oxidation resistant Nimonic 90 punch has high Cr% and low Fe% composition and so it is unlikely that a different oxide layer would form for the different test conditions on the punch [15,20,21]. The similar hue of the punches after exposure to each of the high temperature environments supports this assumption.

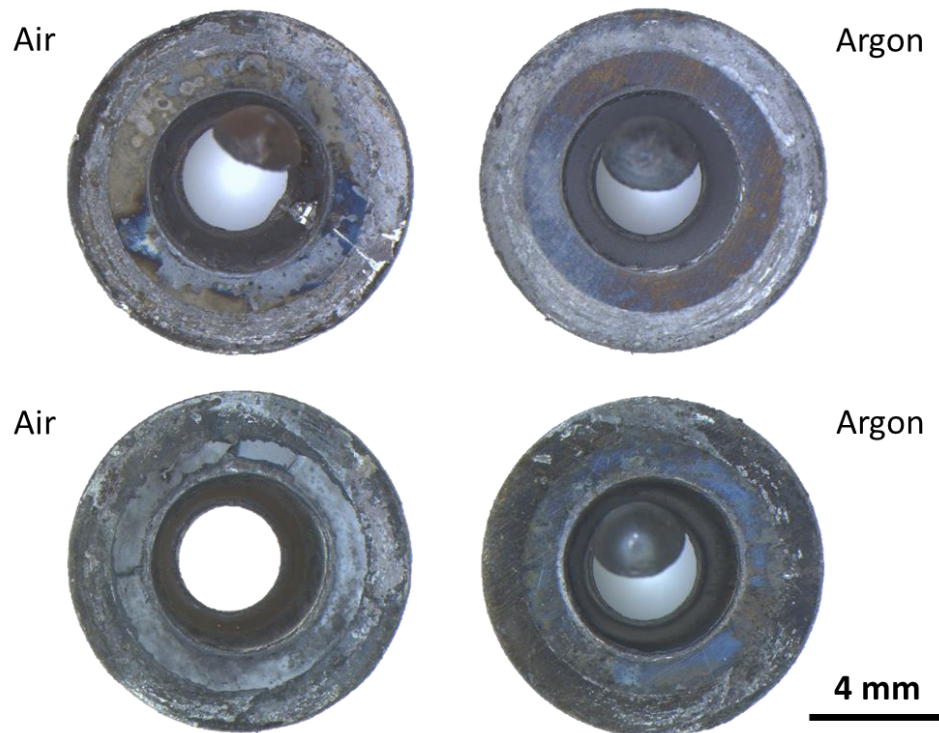


Figure 8. Optical macrographs of specimens tested in air (left) and argon (right).

It is therefore presumed that the difference observed in SPCT behaviour when using an air or argon environment can be largely attributed to differences in surface oxides formed on the specimens during high temperature testing, which alter the coefficient of friction between the punch head and the specimen. The influence of the oxidised material, with presumably degraded mechanical properties, has not been investigated yet in this study, but this might also play a significant role. Progressive change to the observed time to failure and minimum deflection rate is attributed to evolution of an oxidised surface on the punch head, which also affects the coefficient of friction between punch and sample. However, this effect appears to stabilise. The results of Fig. 7 and Table 3 are therefore considered much more representative of the true SPCT behaviour and of the scale of the difference between measured parameters from argon and air testing.

The failure mechanisms may also be influenced by the testing environment, which in turn could contribute to the differences in parameters measured in this study. Although relatively little work has investigated the failures involved in SPCT [23,24], particularly with regard to the influence of oxidation, it seems plausible that micro-cracks in the brittle oxidised material near the surface would be created and propagated much more easily than the more ductile un-oxidised steel during bending and the membrane-stretching of the material around the punch. The more brittle nature could in part contribute to the reduced time to failure and deflection to failure, and also the significantly greater scatter seen in the air-tested data. Oxidation of the fracture surfaces themselves, likely occurs too slowly to significantly influence the behaviour [24]. Further investigations, primarily scanning electron micrography, are required to make any conclusions on the differences and causes of the failure mechanisms. The scatter in both environments is significant but this is also a common feature of uniaxial creep and stress-rupture testing.

The results do have significant implications for high temperature SPCT. The test environment, and control of the test environment as Nishioka et. al demonstrated [12], must be considered for standardisation of the technique. SPCT data, especially at relatively high temperatures, will be difficult to compare when a different environment is used or when the same environment is used but at a range of different temperatures. A drop in mechanical performance with temperature may be partly an illusion due to oxidation. Testing in air, where the percentage of the

sample that becomes oxidised may become relatively high, may also confuse interpretation of the data particularly as different materials will oxidise differently and at different rates. In this regard, testing in argon may be superior to air at high temperature. The argon data also appears to produce a lower scatter in the data – allowing more reliable interpretation and less test material to be required.

The punch head selection and condition are also important considerations for standardisation and comparative analysis in SPCT. Regardless of test environment, different punch materials will exhibit different coefficients of friction which have been shown to impact time to failure in models. If changes to the punch with progressive testing can impact the results, as is suggested in this study, this must be taken into consideration as part of any study involving SPCT, and likely SPT [25,26]. Either by stabilising changes to the punch by forming a scale on the punch before testing by heating in air or selecting a different material for the punch that is hard, creep resistant, and oxidation resistant without the formation of a scale, such as diamond.

Along with investigations into the bulk and surface oxidation of the tested samples, future investigations will include fractography to analyse failure modes and further testing to ascertain whether the change in minimum deflection rate with test environment was significant. Much of the future testing will be conducted in vacuum. This will allow the further study of a different test environment, but also an opportunity to observe whether the progressive change in SPCT behaviour will be replicated in a different low oxygen environment.

5. Conclusions

The results show that the testing environment can have a significant effect on the small punch creep behaviour. Both time to failure and deflection at failure increased when testing in the argon environment compared to the air environment by a factor of approximately 30 and 7%, respectively. These values are calculated from the data with the initial two argon tests omitted. The minimum deflection rate also decreased in the argon environment compared to testing in air, but not to a significant degree. The investigations also strongly imply that the testing environment can affect the punch head which in turn can influence the outcomes of the testing.

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