Technical Note: Experimental techniques for Impression Creep and Small Punch testing

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Abstract: The test rig for Impression Creep testing was designed based on the application of resistance cable heating instead of a normal furnace. The thermal stability is crucial in Impression Creep testing because of small displacements, typically just some tens of micrometers. Good thermal stability was achieved by applying resistance heating cables with an integrated thermocouple inside. For frictionless displacement measurement, ceramic tube and rod are used, located inside the hollow loading bar, protected from direct heat radiation and thermal fluctuations, thus avoiding thermal strains. In an international Round Robin these innovative arrangements provided the best strain signal stability. For the Small Punch testing the aim was set to make the test rig gas tight in order to avoid ingress of oxygen. However, the combination of gas tightness and frictionless movement of the puncher are contradictory requirements. This was solved by using a metallic bellow. Only the spring load of the bellow needs to be compensated which is possible when the loading is provided by a servomechanical testing machine.

Keywords: Impression Creep, Small Punch, thermal stability, displacement measurement, frictionless

1. Introduction

Miniature test techniques are very useful for many material property characterisation applications, but the miniature test method will require some miniaturisation of the test rigs, which causes some technical challenges. In Impression Creep testing the biggest challenge is to achieve a good signal stability when so small displacements are measured, typically just some tens of micrometers. Many extensometer systems are capable of measuring these with sufficient accuracy as such, but the thermal fluctuation from a furnace will cause thermal expansion, which results in pseudo strain and scatter. In addition, friction in some extensometry systems will cause additional scatter.

In Small Punch creep testing the displacements are typically around two millimeters, so the accuracy in displacement measurement accuracy is not a problem. The fluctuation in temperature and frictional effects can cause some scatter in the creep rate of the test material. However, a serious problem in Small Punch creep testing is the oxidation of the small test specimen, which is just 0.5 mm thick. Argon flushing through the test rig with small tolerances will not protect the specimen from oxidation. Use of gaskets would give rise to friction, which is then impossible to compensate for.

2. Thermal stability

Using similar size furnaces for miniature testing as for traditional uniaxial creep testing is not an optimum solution because the test rigs are smaller and thus more sensitive to the inevitable thermal fluctuations caused by the furnace and a PID temperature controller. An on-off controller with a long idle time will certainly result in thermal fluctuations. Another problem especially in Impression Creep testing is that the contact surface between the specimens and the indenter is just 10 mm^2 and therefore it can easily occur that on one side (typically on the upper part of the rig) the tools are at higher temperature than on the other side because the thermal contact area is so small and as a result, the specimen temperature will be ambiguous. At VTT these problems were solved by using 250 W resistance heating cables wound around the test rig body with a 40 mm outer diameter. The heating arrangement is shown in Figure 1. Two coils are used which have their own PID temperature controllers. In this way the temperature of the upper and lower part of the test rig can be kept at the same temperature. The heating cables have a thermocouple integrated inside the metallic sheath, which was originally meant to work as overheating protection but has been used as the controlling thermocouple. In this way the temperature of the heating cable is constant and as a result the temperature measured from the test specimen is as constant as practically possible, most scatter coming from the inherent scatter of the measuring thermocouple itself. If the contact between the coil and the rig changes and the specimen temperature starts to drift away from the test temperature, small adjustments may be

needed in the temperature setting, especially when all parts are new. An example of the temperature stability is shown in Figure 2.

In Small Punch creep testing the thermal mass of the test specimen is very small compared with the much thicker surrounding parts of the test rig, so if there are large temperature gradients within the test rig, the temperature of the SP specimen can be ambiguous. Therefore, the same type of cable heating is used at VTT in the Small Punch test rig as for Impression Creep. During pilot testing phase the temperature can be measured from several different locations in the test rig and the location of the heating cable can be adjusted to reach the optimal temperature distribution.
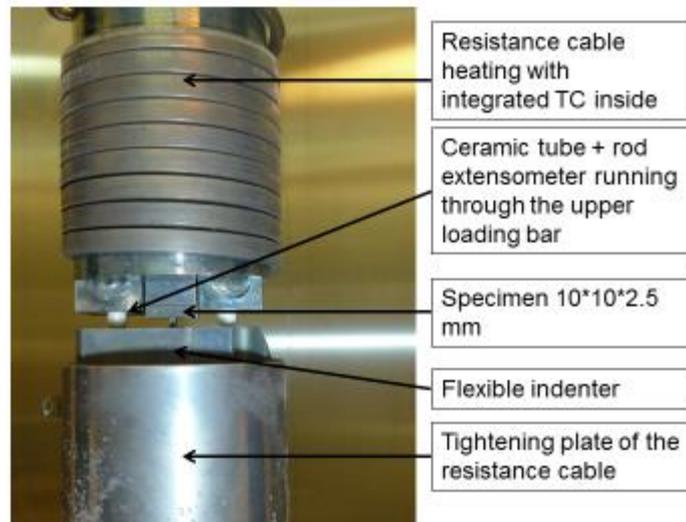

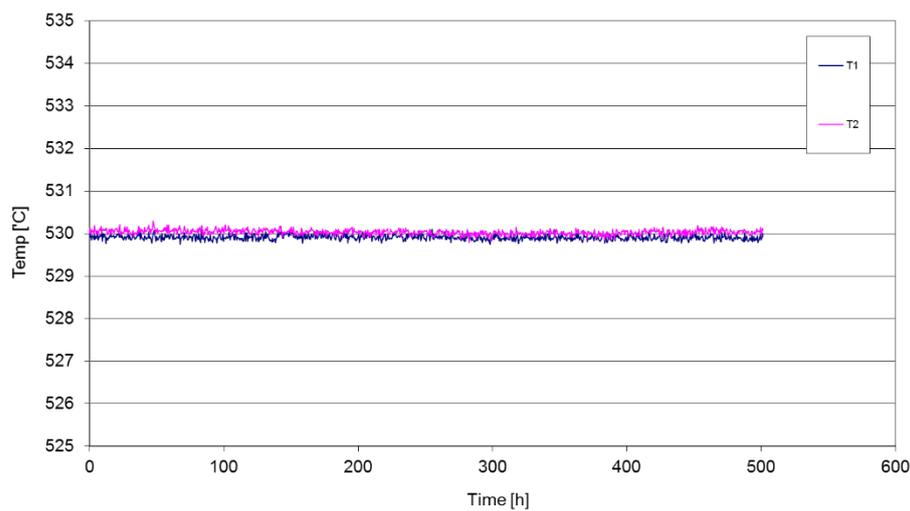**Figure 1.** Detail of the impression creep rig with the upper tightening plate removed.

Figure 2. An example of the specimen temperature in an Impression Creep test at 530°C.

3. Displacement measurement in Impression Creep

The basic idea in the displacement measurement system was to measure the indenter movement as close to the specimen as possible and to protect the extensometer from direct heat radiation and thermal fluctuations. This was achieved by using a tube and rod type of extensometer and placing it inside the hollow loading bar. Al_2O_3 tube with od 8 mm and id 5mm and a 4 mm rod are used as shown in Figure 2. Originally the tube and rod were run through the lower loading bar and the tube was held in place by pin loading which was not a very stable way of holding the tube in place. Later the whole system was reversed because then the tube is coming from above and is resting against a flat surface and the contact is better defined and stable. When the lower tip of the rod is centered so that it does not touch the tube and the upper tip is centered by the Heidenhain 12.5 mm digital displacement transducer with 10nm resolution, the extensometer is frictionless (disregarding the internal friction of the displacement transducer) which helps achieve very smooth displacement curves as shown in Figure 3. This in turn translates into steady strain rate curves as a function of time and a "constant" strain rate value calculated from the last 100h of the test data. To be precise, the strain rate does not become constant but is reducing continuously as a function of time.

During the setting up of a test the frictionless movement of the extensometer is verified at room temperature by doing a step-loading to 2000 N (or less if the test load is smaller) and recording the hysteresis loop of the load vs displacement curve. This could be a recommended practice to be introduced into a future standard for Impression Creep testing.
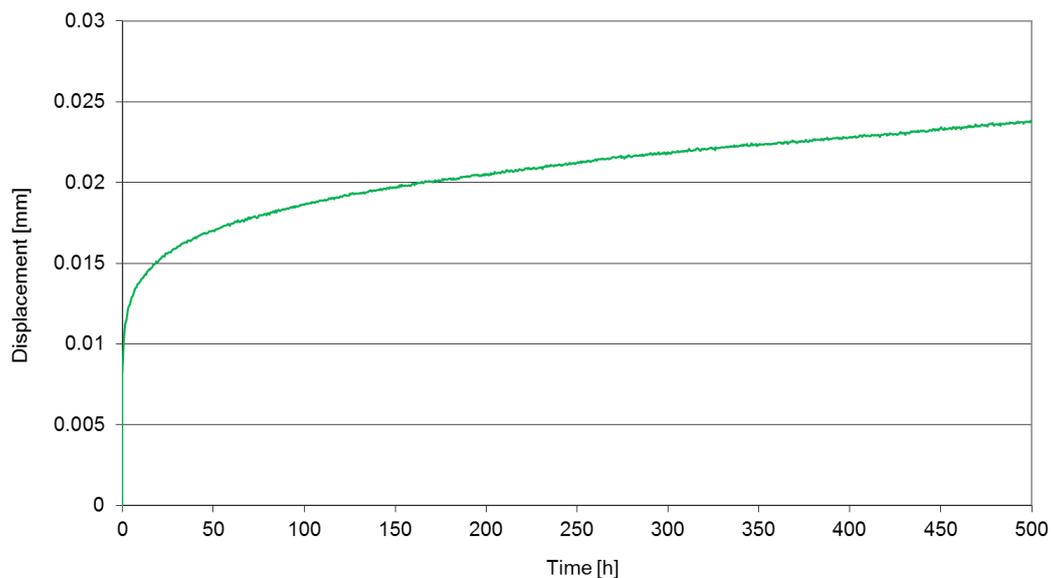

Figure 3. Impression Creep displacement curve for P91 at 89 MPa at 600°C.

4. Floating indenter

The loading bars illustrated in Figure 1 do not allow any flexibility to adjust the initial contact between the specimen and the indenter. Therefore, a flexible indenter was designed, which can tilt around the axis perpendicular to the indenter blade as shown in Figure 4. During setting up of the test the flexible indenter is moved sideways with a small load on (<10 N) until a good contact with the specimen is achieved. Flexibility around the second axis is of lesser importance as the indenter is just 1 mm wide and the initial plasticity will help achieve full contact of the indenter surface, as can be concluded from Figure 3 where the initial plasticity is several micrometers. The initial contact problem is valid only in the first test of step-loaded test series where the test load is increased after a "steady state" creep rate is achieved. A perfect contact is guaranteed in the subsequent tests.
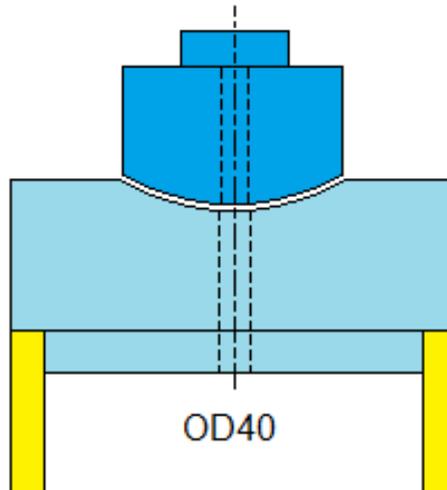
Figure 4. Schematic picture of the floating indenter with holes for the thermocouples

5. Gas tightness for Small Punch

Oxygen is a very aggressive element and will easily move against the gas flow and even against higher pressure. Therefore, oxygen ingress will be inevitable if the rig is not gas tight. For Small Punch testing at VTT a stainless steel tube is used instead of a ceramic tube because either this can be welded into the test rig body or as in our case a conical 45° part can be welded to one end of the tube and tightened against a counterpart in the test rig, allowing gas tightness and removal of the part. At the other end, a rubber bellow is used to prevent argon gas leak from between the tube and the 3 mm ceramic rod used for deflection measurement from below the SP specimen.

A much bigger challenge is to combine gas tightness with frictionless movement of the puncher. At VTT this has been achieved by using a metallic bellow which is welded into a stainless steel tube from both ends as shown in Figure 5. The puncher runs through the tube and is fitted by a flange connection. The wall thickness and the outer diameter of the bellow are 0.1 mm and 19 mm, respectively. Welding of such a thin wall proved to be painful when holes were burned and gas tightness lost. Finally, with orbital micro-TIG welding a sound joint was produced and gas tightness achieved. In hindsight, an easier option would have been to use factory made Swagelok corrugated tubes.

The metallic bellow will produce a spring load and this will reduce the load experienced by the specimen. The combined spring constant of the bellow and the displacement extensometers is 4.32N/mm and this has to be compensated for as a function of the puncher displacement. This is done either by increasing the load setting by hand or automatically by a computer controlled testing machine.

Figure 5. Metallic bellow welded into a stainless tube.

6. Combined thermocouple and extensometer rod for Small Punch

As the deflection of the SP specimen should be measured from below and the temperature should be measured preferably directly from the specimen a solution was introduced by combining these two functions by using a 3 mm ceramic rod with two holes inside for thermocouple wires. 0.5 mm S-type thermocouple wires were inserted through the holes and micro-TIG welded into a 0.5 mm thick Nimonic plate with 0.5 mm holes as shown in Figure 6. At the outer end, the thermocouple wire holes were sealed by epoxy glue. This arrangement then allows a direct measurement of the specimen temperature and the deflection measurement from below the specimen.
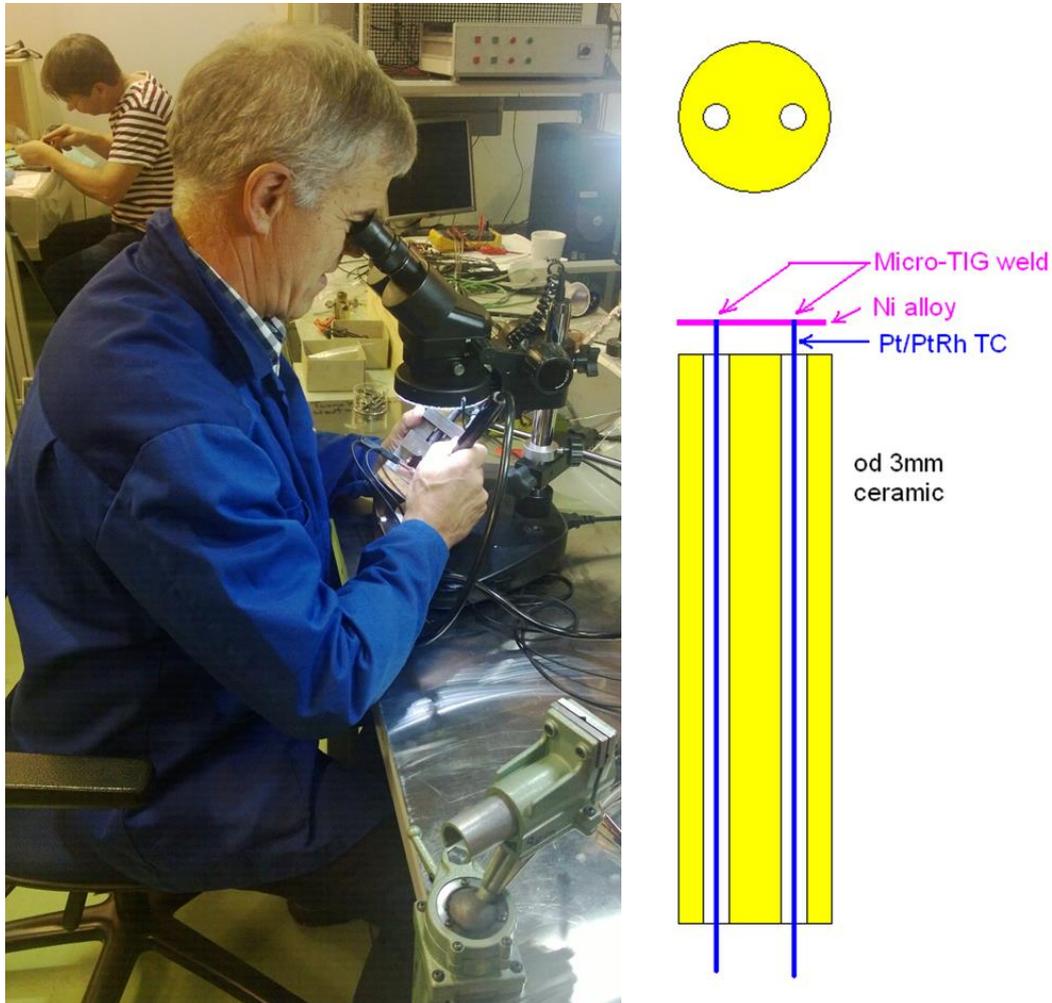

Figure 6. Welding of the thermocouple-extensometer combination by micro-TIG under microscope.