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**Procedures for Steam
Oxidation Testing of Static
and Dynamic Specimens**

**A T Fry, L J Brown, M G Gee
E Piedra**

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Materials Division

ABSTRACT

Three test methods have been developed to evaluate the properties of contacting surfaces at high temperature under static and dynamic loading conditions. The performance of these new tests has been evaluated using a range of industrially relevant materials and conditions. The results of these tests have shown that it is possible to have two oxide fronts grow into each other to form one monolithic oxide scale. Dynamic tests have shown that the addition of steam in a tribological environment clearly influences the friction between materials. Pre-ageing also influences the friction, with the majority of materials experiencing an increase in friction coefficient.

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National Physical Laboratory
Hampton Road, Teddington, Middlesex, TW11 0LW

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Approved by Dr Markys Cain, Knowledge Leader, Materials Team

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1 INTRODUCTION

The two key driving forces in the development of the global power generation industry are the increasing demand for electricity and widespread concerns about the effects of emissions of greenhouse gases on the global environment. There are many predictions for the worldwide growths in energy and electricity demands, with a range of different assumptions; for example, the latest International Energy Authority (IEA) predictions indicate there being approximately a 40% rise in primary energy demand by 2030 [1]. Concerns about the emissions of CO₂, and other greenhouse gases (GHGs), into the atmosphere have been growing during the last two decades; as a result there have been increasing efforts to limit the amount of CO₂ emitted into the atmosphere. The Kyoto Protocol committed many countries in the world to reduce their GHG emissions by 2008-2012 compared to 1990 levels, with the EU committed to a 8% reduction by 2008-2012 [2]. It is anticipated that follow-on treaties will agree higher levels of reductions. National governments are also continuing to develop policies to limit greenhouse gas emissions; for example, the latest UK government target is for a 60% reduction in CO₂ emissions by 2050 [3]. There are many sources of GHG emissions, but in the UK (and other developed countries) where a centralised power generation system has developed over a long period, large power stations represent major, easily identifiable point sources for CO₂ emissions. These therefore provide a focus for efforts in reducing CO₂ emissions.

One route available to the industry is to increase the efficiency of the power plant; carbon capture is also possible but will still require improved efficiency to make such endeavours viable. Attempts to improve plant efficiency will lead to increases in temperatures and pressures in plant and thereby place increasing higher demands on materials. Higher temperatures, pressures and working lifetime means that the materials which are currently in may not be adequate to meet these higher demands. Alternative materials, which are capable of withstanding higher, more aggressive environments, are currently being studied for next generation power plant. An essential part of this work is the assessment of oxidation of contacting surfaces and high temperature tribological performance.

High temperature sliding wear and friction testing has been carried out previously by a number of different workers [4-10]. Much of the work has been concerned with the high temperature tribological performance of ceramics [4-8], with some work on high temperature low friction coatings for dry sliding applications [6], and on tribological components for gas turbines [10]. These tests have previously been carried out at elevated temperatures under load at ambient pressure in an air environment.

More recently there has been work on assessing the tribological performance of steam generating plant materials in water environments [11,12] and at elevated temperatures in steam environments [13,14]. The results for sliding tests in a steam environment showed complex behaviour where the formation of surface glaze layers and variation in contact loads have a major effect on the results that are obtained.

To be able to evaluate the material performance under static load and dynamic conditions, two new test methodologies were required; these have been developed and are described in the following report.

2 TEST METHODS

2.1 Isothermal

Under standard isothermal testing in steam environments it is common to expose a number of test pieces to flowing steam, with and without the use of a carrier gas. Further details on this type of testing can be found in [15]. In these tests the test pieces are not restrained in any manner and all of their surfaces are exposed to the steam environment. Under such conditions the oxides grow unconstrained, and consequently no evaluation of oxide jacking, adhesion or tribological properties can be made.

2.2 Static Contacting Surfaces

2.2.1 Rationale

In industrial plant there are many surfaces that are in contact. Most of these, once made, will not need to be disturbed again. Others, usually around critical components, may need regular servicing and separation. If these contacting surfaces are in areas of extreme temperatures and aggressive environments then the surfaces will degrade and oxide jacking may occur and/or the surfaces could mechanically bond through oxide growth. To measure this and quantify the severity of the problem with respects to different materials and material combinations a test method is needed which would allow the degradation of contacting surfaces in a controlled manner.

2.2.2 Specimen Design

To perform exposures under conditions whereby two faces are in static contact under an environment of flowing steam required a new specimen geometry to be developed. Initially two approaches were considered. The first approach was to stack the surfaces and apply external forces to hold the faces together, this would require the test piece to be machined such that one flat face sat on another separated by “pips” on one of the specimen halves. By placing a mass on the top of the upper specimen the two halves could be held together. Initial trials with this approach using a mild steel specimen did not produce the adhesive effect of the oxides required for subsequent testing, in fact there was no cohesion between the faces at all, and so this approach was abandoned.

The second approach, which was subsequently adopted, consists of a pin inserted into a collar, as shown schematically in Figure 1. The insertion of the pin produced an interference fit bond that could be controlled by the tolerancing of the ‘pips’ on the pin. These pips are shown in Figure 1 at three positions around the circumference of the pin. The presence of these pips would help to maintain a constant minimum separation between the pin and the collar. Both the pin and the collar were designed to have ground surfaces to enable metrology of the oxide thickness after exposure.

Trials were performed using this geometry on stainless steel material. This combination of pin and collar was exposed to laboratory air at 750°C for 250 hours. After the exposure the specimen was sectioned, mounted and polished and examined optically using a confocal microscope. Figure 2 shows the sectioned specimen and micrographs of the oxide formed on the bottom face of the pin. These trials indicated

that the specimen design would produce the oxide growth necessary for evaluation and testing of the adhesive forces needed to remove the pin from the collar.

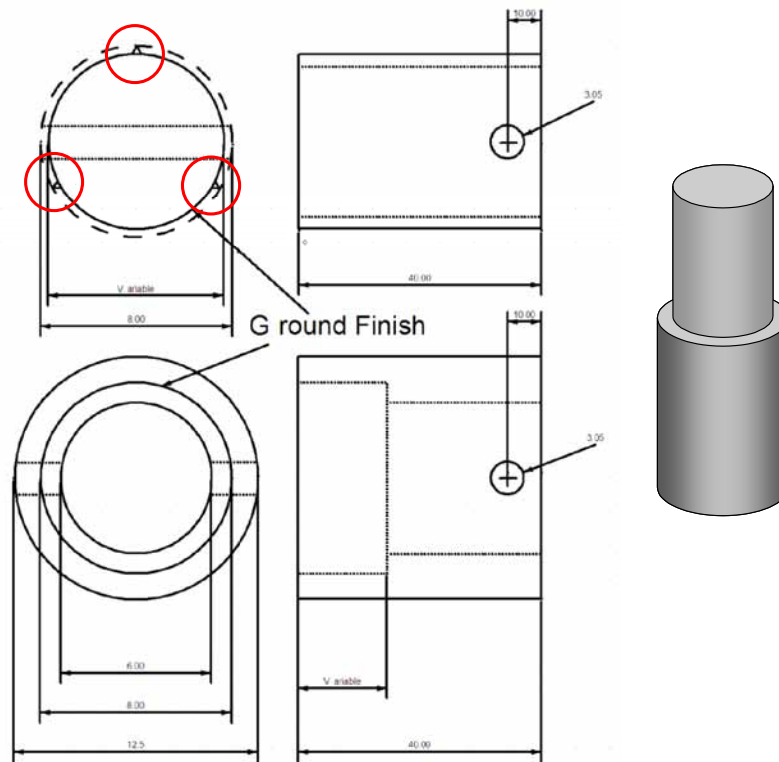


Figure 1 Schematic of the pin and collar design showing the location of the spacing pips on the pin.

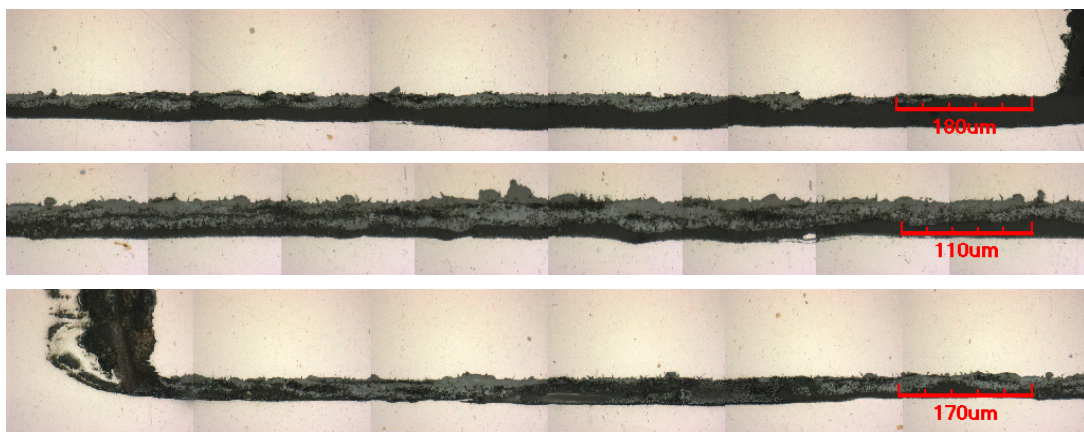


Figure 2 Stainless steel pin and collar sample used to assess the feasibility of this specimen design. Micrograph shows the oxide formed on the bottom of the pin at the two edges and the centre.

2.2.3 Exposure Procedure

Pins and collars are manufactured such that the gap between the pin and the collar is controlled via the spacing pips. The specimens, once the pin has been inserted into the collar, are placed in a furnace through which flowing steam can pass. A schematic of the furnace is shown in Figure 3.

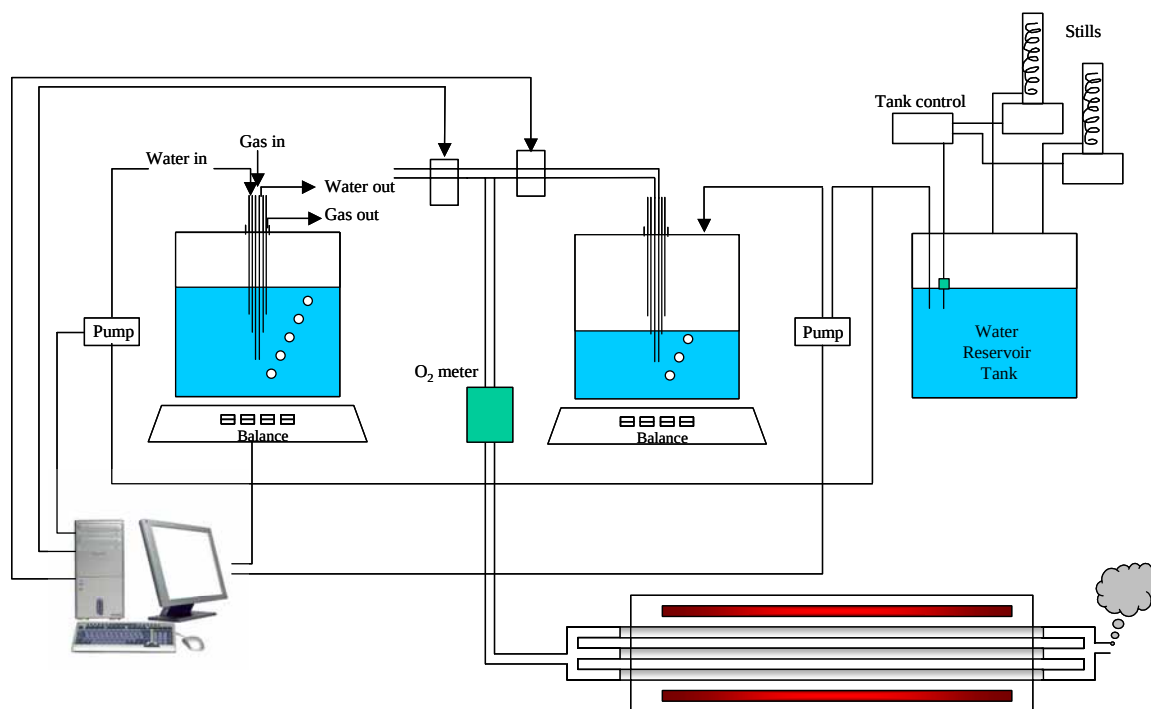


Figure 3 Schematic of the exposure facility used to expose the pin and collar specimens.

The water used to generate the steam has been deionised prior to pumping into the storage vessels. Once in the vessels N₂ gas is bubbled through the water to deoxygenate it. The water is then forced via a positive gas pressure into the furnace through a system of pipe work consisting of all metallic components. This maintains the low level of dissolved oxygen in the feed water, measured with an oxygen meter to be in the order of 10 ppb.

The furnaces used in these exposures have been adapted such that they consist of three metallic work tubes within one tube furnace. As a consequence of this modification the specimens could not be exposed in a vertical position, but were positioned on their sides and exposed in a horizontal position. On insertion of the specimen into the work tube care needs to be used to ensure that adequate flow is maintained around the specimens. The specimens are then left in a flowing steam environment for a set number of hours to allow the oxides to form and grow on both the pin and collar surfaces. As the oxides grew and met at the interface between the two surfaces, the aim is for the applied forces from the oxide growth to cause the oxides to effectively 'stick' together. This force of adhesion can subsequently be measured using a standard tensile test machine.

Once the exposure is finished the specimens are removed from the furnace gradually to avoid thermal shock. The specimen is then carefully stored prior to mechanical testing.

2.2.4 Mechanical Testing

Mechanical testing has been used by other organisations to assess the adhesive strength of oxide/oxide interfaces. Work performed by Alstom Germany used butt joint geometry where an adherand was stuck to the oxide using an adhesive. A tensile test machine was then used to separate the oxide from the substrate; this is shown in Figure 4.

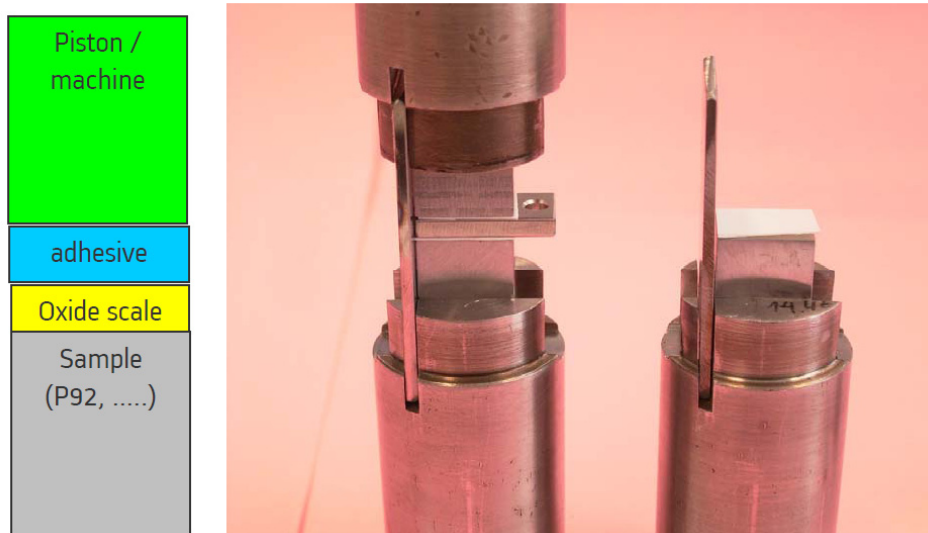


Figure 4 Geometry of specimen used by Alstom Germany [18].

The results obtained from such measurements provided a measure of the oxide strength, which was used to assess the propensity for spallation of the oxide. The aim of the test being developed at NPL was to provide a measure of the cohesive strength of oxide formed between two contacting surfaces, and so the approach used by Alstom was not appropriate. Instead, in the case of the pin and collar specimens, the pin would be mechanically loaded until the oxide bond was broken. Once there is evidence of this the test is stopped, and so the pin is not completely removed from the collar. This enables the oxide to be retained and optical measurement of the oxide thickness and structure to be made after the test. In addition to these optical measurements, ultrasonic measurements were also performed prior to mechanical testing to measure the original oxide thickness. This is reported in [19].

When considering the forces required to separate the two surfaces there is a general belief that a fairly small force would be required, and therefore an estimate of a maximum load of 3kN seemed reasonable. Consequently a loading frame (Instron Electropuls E3000, driven under velocity control) with a 3 kN load cell was selected for mechanical testing use. This allowed the collar half of the specimen to be securely gripped in the lower chuck of the machine, as shown in Figure 5. Initial trials were conducted using the specimens exposed. The specimens used did fail within the load limits of the equipment (see Figure 6), but it should be noted that these were Ni-based alloys and so oxidation was believed to be low. Figure 7 lists the material combinations used. Note that the specimen consisting of an IN617 collar with an IN939 pin was considered to be loose and so did not require much force to move the pin, as shown in Figure 6. In addition to these samples there were also samples of 9 %Cr martensitic material, which will oxidise to a greater extent and so require a higher load.

Consequently a decision was made to move to a larger load cell (10kN) on a different load frame (Instron 4507, driven under displacement control).



Figure 5 Photo showing the gripping method used for the pin and collar samples in the ElcetroPuls tensile tester.

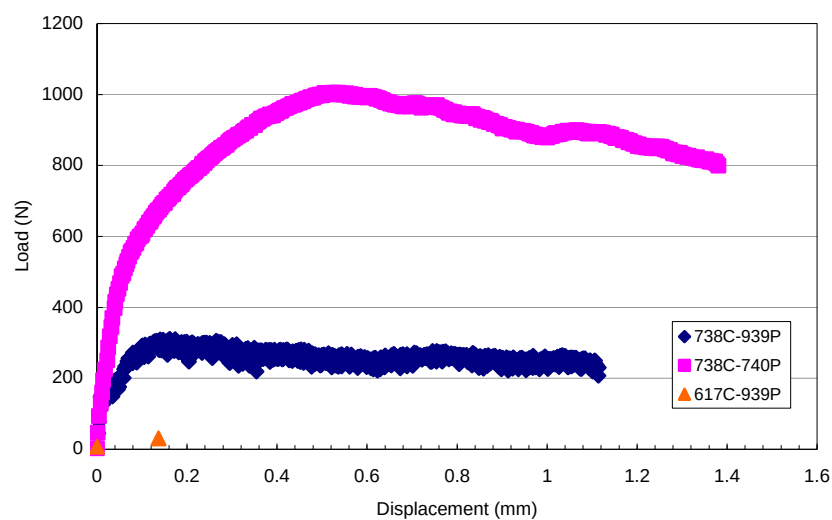


Figure 6 Load – Displacement plot for three specimen combinations.

Figure 6 shows how the force required to move the pin increases until the oxide scale is fractured, at which point the force remains fairly constant (decreasing gradually) as a result of frictional forces between the oxides on the pin and collar.

		Pins					
Collars		IN738	IN617	P92	IN939	IN740	
	IN738	X	X	X	X	X	750°C
	IN617		X	X	X	X	750°C
	P92			X	X	X	650°C

500, 1000, 2000, 3000h

Figure 7 Pin and collar combinations used in an evaluation exercise for this experimental procedure.

Due to the higher than expected forces required to remove the pin the majority of the tensile tests were conducted in an Instron 5500R test frame. The tests were controlled using a constant displacement rate, which was fixed at 1 mm/min. The mounting of the specimen is shown schematically in Figure 8.

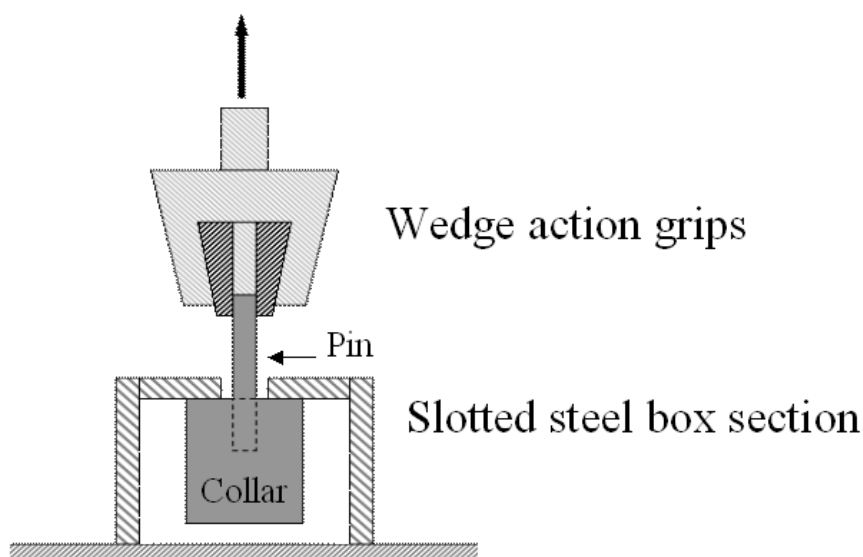


Figure 8 Schematic showing the manner in which the pin and collar specimens were gripped in the tensile test machine.

The maximum load registered during the tests was recorded, and this corresponds with the beginning of the pin movement.

A full list of the material combinations, exposure temperature and time is presented in Table I.

Table I Material combinations, exposure time and temperature.

Specimen ID		Materials		Temperature, °C	Time, h
		Pin	Collar		
AARB	001	IN617	IN617	750	500
	002				1000
	003				2000
	004				3000
AARC	001	IN740	IN617	750	500
	002				1000
	003				2000
	004				3000
AARD	001	IN939	IN617	750	500
	002				1000
	003				2000
	004				3000
AARE	001	P92	IN617	650	500
	002				1000
	003				2000
	004				3000
AARF	001	IN617	IN738	750	500
	002				1000
	003				2000
	004				3000
AARG	001	IN740	IN738	750	500
	002				1000
	003				2000
	004				3000
AARH	001	IN939	IN738	700	500
	002				1000
	003				2000
	008				3000
	007			750	500
	005				1000
	006				2000
	004				3000
AARI	001	P92	IN738	650	500
	002				1000
	003				2000
	004				3000
AARJ	001	IN738	P92	650	500
	002				1000
	003				2000
	004				3000
AARK	001	IN740	P92	650	500
	002				1000
	003				2000
	004				3000
AARL	001	P92	P92	650	500
	002				1000
	003				2000
	004				3000

2.3 Dynamic Contacting Surfaces

A test system used in earlier work to measure the dry sliding wear performance of ceramics and power generation materials [6,7] was modified to allow for testing to be conducted in a steam environment. This involved replacing the specimen holder arms and heating elements for the furnace due to existing material not being stable in a steam environment. The wear test apparatus is shown schematically in Figure 9. This set up comprises of a split furnace, which encases the arms during testing. This is easily removed by virtue of a sliding track on which the furnace is mounted. The arms hold the specimens in the steam environment within the furnace. The upper arm is attached to an actuator, allowing movement of the specimen against a fixed bottom arm. The distance travelled by the upper arm is measured using a horizontally mounted LVDT. This motion is controlled with limit switches which ensure that the movement is stopped if the travel is exceeded. The vertical displacement is measured with another LVDT and the load monitored using a vertical load cell to ensure it remains constant throughout the test. An additional horizontal load cell is positioned which enables the wear friction coefficient to be calculated. The bottom arm is pivoted to allow the lower specimen to be pressed into contact with the top specimen. The arms are water-cooled to prevent heat transfer outside of the furnace and further cooling is provided using compressed air directed onto sections of the arms outside of the furnace. The load is applied by the addition of weights added to the end of the pivot arm. The temperature of the furnace and the specimens are measured in-situ using two sheathed thermocouples.

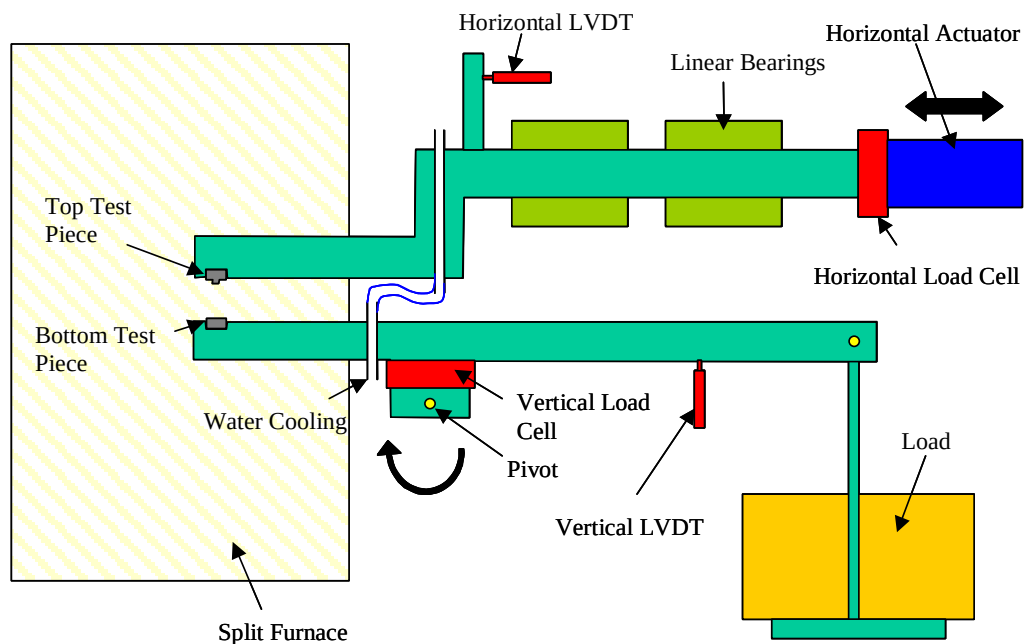


Figure 9 Schematic of the low frequency wear apparatus.

The specimens were designed to allow for a 'push' fit into the arms, with the option of an additional screw to ensure that the specimens are held firmly in place. The top specimen comprises of a block with a protruding stub which is rubbed against a flat plate specimen, see Figure 10.

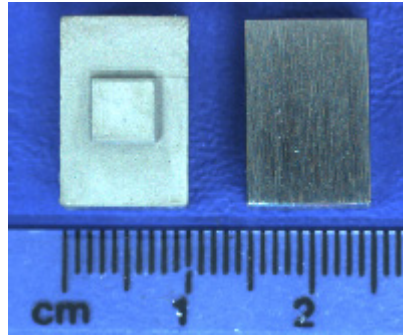


Figure 10 Specimen Dimensions.

The arms were designed specifically to allow the top and bottom surfaces to be positioned parallel to each other to ensure a flat contact between the top and bottom specimens. This was done by enabling the top arm to rotate clockwise and anti-clockwise with the lower specimen held in a holder which allowed lateral movement, see Figure 11.



Figure 11 Sample Holder Arms.

The addition of steam to the furnace was achieved by injecting a fine mist of water into the furnace. A pressurised container filled with de-ionised water was placed on a balance and the water sprayed through a nozzle (~ 10 ml/min) into the furnace through a small entrance hole. When the water level dropped below a minimum threshold, a pump connected to a water supply automatically topped up the water level, the pump then stopped when a maximum level was achieved (software monitored the balance). This ensured that there was always a constant supply of water for the duration of the test. The water mist was not injected into the furnace until the required temperature was achieved; this ensured that the mist instantly turned to steam.

Software was written specifically to control the start and end of the test, automatic top up of water, as well as collecting data for friction, load, temperature and number of cycles and time. A screen shot of the program is shown in Figure 12 presenting an example of the friction loop results that are obtained.

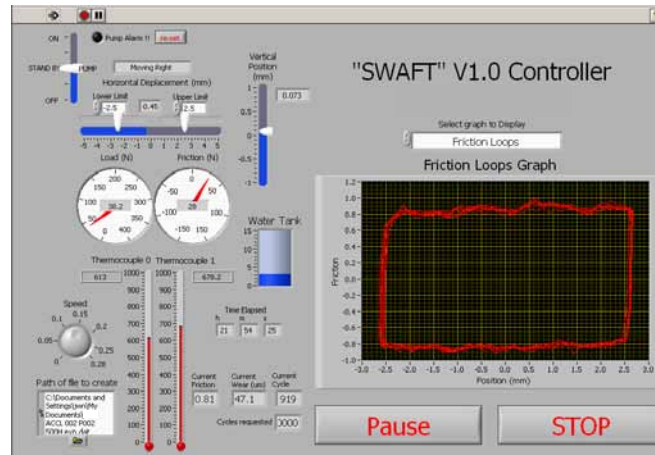


Figure 12 Software Program.

2.4 High Frequency Wear

Many of the components in power generation plant are subject to sliding contacts where the contact oscillates at high frequencies and under elevated temperature conditions. Measurement of the performance of materials is therefore required to assess the suitability of different materials for these applications, and to be able to estimate the lifetime of materials.

There are several possible approaches to carrying out this type of testing. These include systems where the loading is obtained through electro-mechanical actuation or a motor, piezoelectric loading, an electromagnetic oscillator, or servo hydraulic test systems.

Motor driven systems lack the necessary stiffness (because of bearings), the system itself is subject to fretting and fatigue, and there is a lack of adjustment while running. Piezoelectric test systems are good for low load applications, but they have a maximum stroke of about 100 μm , and the fatigue life of actuator is limited. Electromagnetic oscillators suffer due to the fact that they are force generators and since the required force is governed by friction, this force may change with time giving control difficulties. In practice there is a need to vastly over-rate oscillators to get good control. Servo-hydraulic load actuation uses the well understood technology used in servo-hydraulic test systems to get good control of loading and displacement in a test.

Figure 13 illustrates the types of test system that have been used in the past. Figure 13a shows an unbalanced system driven by a servo-hydraulic test system [16]. The disadvantage of this test system is that since only a single sample pair is used, the loading on the servo-hydraulic ram is unbalanced leading to potential issues with testing. Figure 13b shows a balanced test system that uses piezoelectric actuation [17].

After reviewing the possible approaches to carrying out this testing, it was decided to purchase a high frequency test system from Phoenix Tribology as a retrofit to an Instron

8500 servo-hydraulic test machine. The requirements that were met by the new system were:

- Accurate control of stroke
- On-line measurement of friction
- Testing at temperatures up to 1000°C

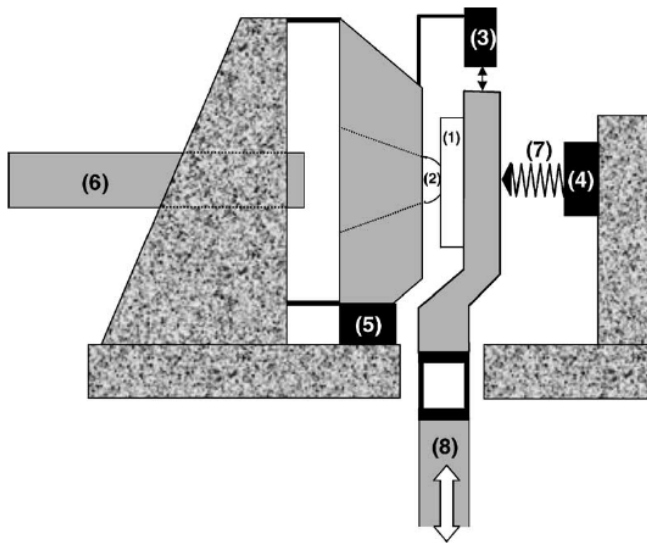
A schematic and photographs of the test system are shown in Figure 14. The system has matched pairs of samples. The central samples are moved vertically by the actuator rod from the servo-hydraulic test system. The vertical motion of this rod is measured by a capacitance displacement probe. The outside specimens are held in specimen holders at the top of the lever arms that are pivoted on flexure pivots. The outside specimen holders are pressed against the central specimens by a pneumatic loading system giving a stable loading normal to the direction of motion.

The specimen holder was designed so that the same specimens could be used for the high frequency tests as those used for the low frequency work, (Figure 10)

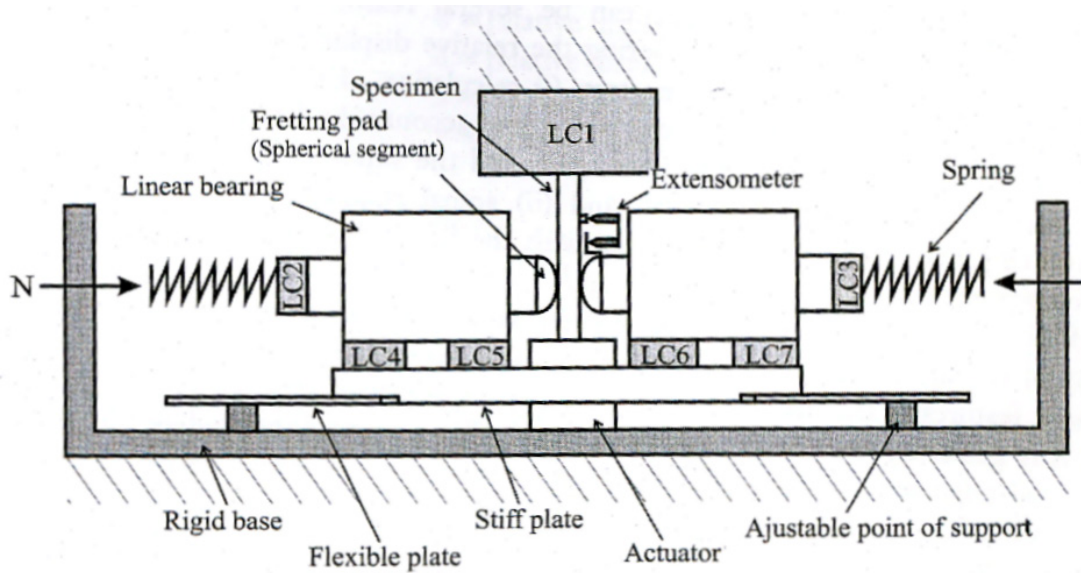
The lever arm assemblies are supported through flexures, and the motion of the levers in the vertical direction is restrained by piezoelectric load cells that thereby measure the frictional load generated in the contact. The outside samples are mounted in hemispherical bearings so that good alignment of the central and outside samples can be achieved.

A top-hat furnace is placed over the samples, actuator rod, and lever arms so that tests can be carried out at high temperature.

A program written using 'Compendex' was used to control the temperature and the applied load. The Instron servo-hydraulic system controlled the frequency of the test and also the distance travelled.



a)



b)

Figure 13 Test systems used in work by a) Lamethe et al (2003), b) Wittkowsky (1999).

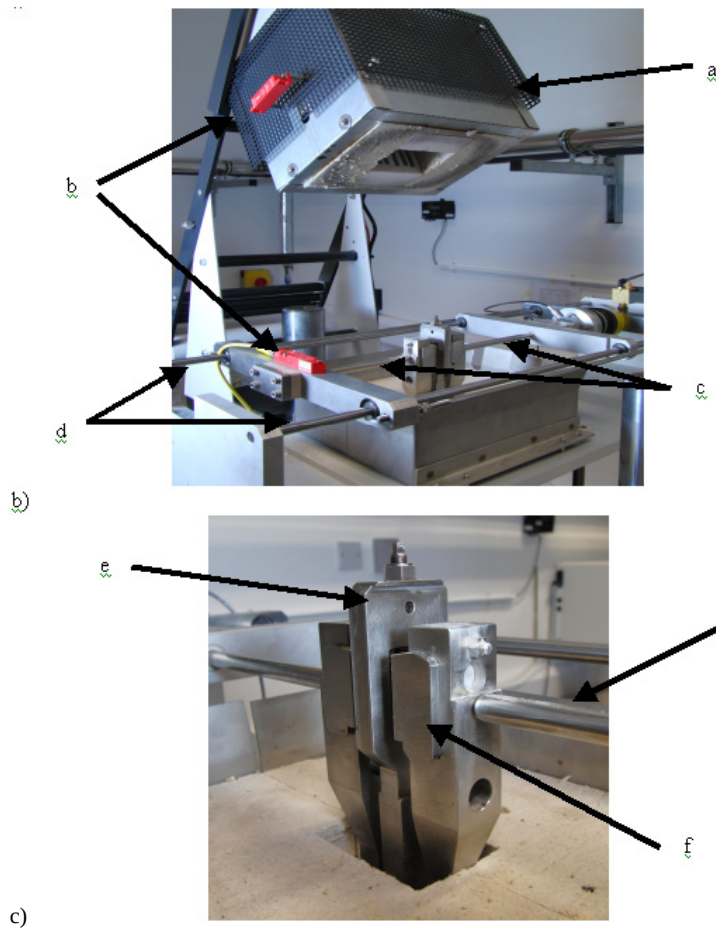
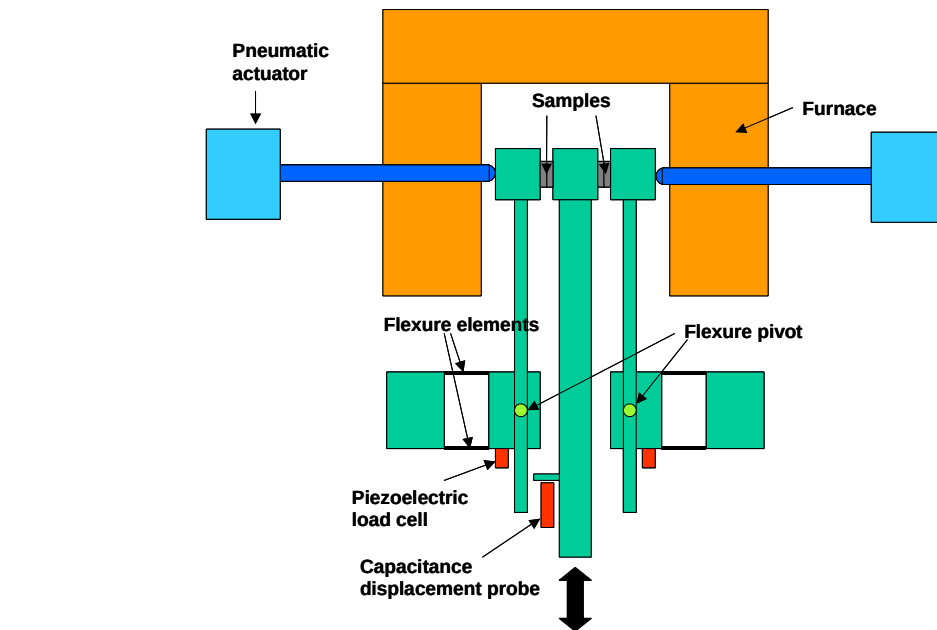


Figure 14 High frequency wear testing system, a) schematic, b) overall view of top of system, c) sample holders. In photographs a is top hat furnace, b is electromagnetic safety interlock, c and d are pneumatic loading system, e is top of actuator rod, and f is the outside sample holder.

3 RESULTS

3.1 Static Contacting Surfaces

The specimens listed in Table I were exposed to flowing steam for the specified time, a selection of these were subsequently selected for oxide thickness measurement using ultrasonic methods [19]. Once these measurements had been made the specimens were tested in the tensile test apparatus.

In some instances the growth of the oxide scale was too low to produce the adherence between the pin and the collar (loose) and in other specimens the design of the holding system was not strong enough to pull the pin out, thus it was only possible to determine a lower limit of the applied load (as in the case of series AARE, AARI, AARJ, AARK and AARL, see Table II).

After testing the pin was then held in place using epoxy resin, the specimens sectioned longitudinally and polished so that the oxide thickness between the pin and the collar could be measured optically. The results of the tensile tests are presented in Table II and shown graphically in Figures 15 – 23. Figures 24 and 25 summarise the maximum loads reached before the oxide fractured and the pin moved. Figure 24 shows the specimens exposed at 750°C, these consisted of Ni-based super alloys. These were found to have not grown particularly thick oxides, as would be expected, and consequently many of the pins were loose, or did not require a large load to move the pin. However, when these alloys were used in combination with a martensitic steel (P92), which grew a much thicker oxide scale, a large increase in the required force was found, as shown in Figure 25. In most cases here the load required exceeded that of the mounting bracket and so the test was stopped prior to removing the pin.

Table II Results of the tensile test measurements on the pin and collar specimens.

Specimen Reference		Materials		T, °C	Time, h	Maximum Load, N
		Pin	Collar			
AARB	001	IN617	IN617	750	500	Loose
	002				1000	253.8
	003				2000	Loose
	004				3000	Loose
AARC	001	IN740	IN617	750	500	Loose
	002				1000	Loose
	003				2000	Loose
	004				3000	Loose
AARD	001	IN939	IN617	750	500	980.3
	002				1000	1358
	003				2000	460.6
	004				3000	34.8
AARE	001	P92	IN617	650	500	>6000
	002				1000	>5000
	003				2000	1846.1
	004				3000	>6000
AARF	001	IN617	IN738	750	500	Loose
	002				1000	Loose
	003				2000	Loose
	004				3000	Loose
AARG	001	IN740	IN738	750	500	206.1
	002				1000	651.4
	003				2000	1251.1
	004				3000	1021.3
AARH	001	IN939	IN738	700	500	Loose
	002				1000	Loose
	003				2000	40.7
	008				3000	237.4
	007			750	500	162.1
	005				1000	Loose
	006				2000	3405
	004				3000	311.3
AARI	001	P92	IN738	650	500	>5000
	002				1000	>5000
	003				2000	Loose
	004				3000	>5000
AARJ	001	IN738	P92	650	500	>5000
	002				1000	>5000
	003				2000	>5000
	004				3000	>5000
AARK	001	IN740	P92	650	500	>5000
	002				1000	>5000
	003				2000	>5000
	004				3000	>5000
AARL	001	P92	P92	650	500	>5000
	002				1000	>5000
	003				2000	>5000
	004				3000	>5000

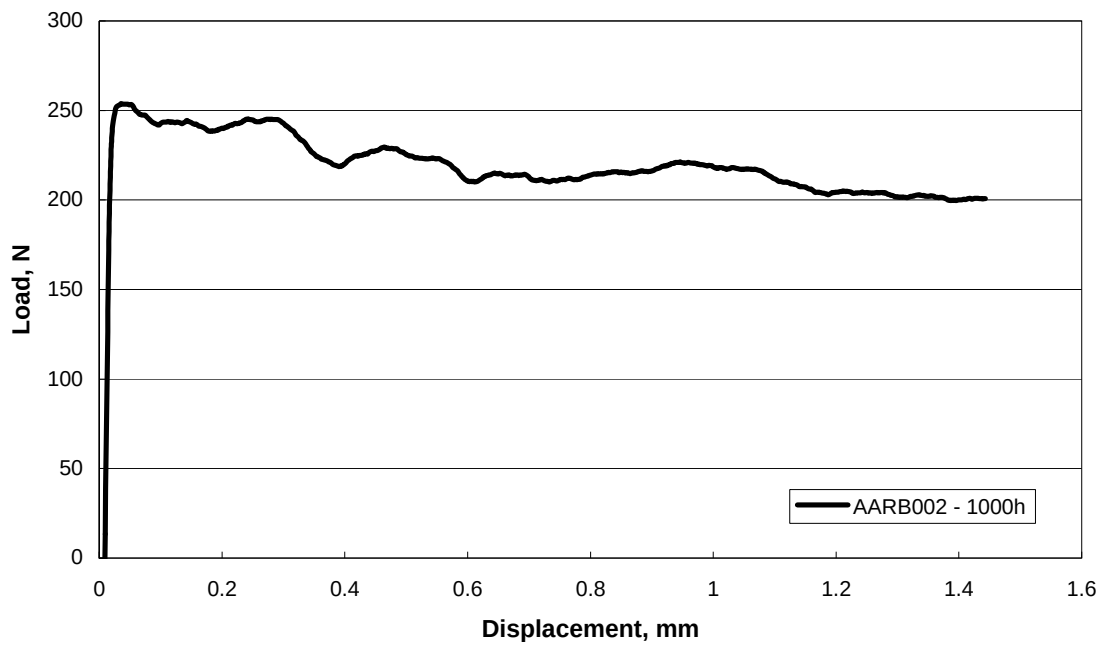


Figure 15 Load vs Displacement plot for IN939 pin in a IN617 collar exposed to flowing steam at 750°C for 1000 h.

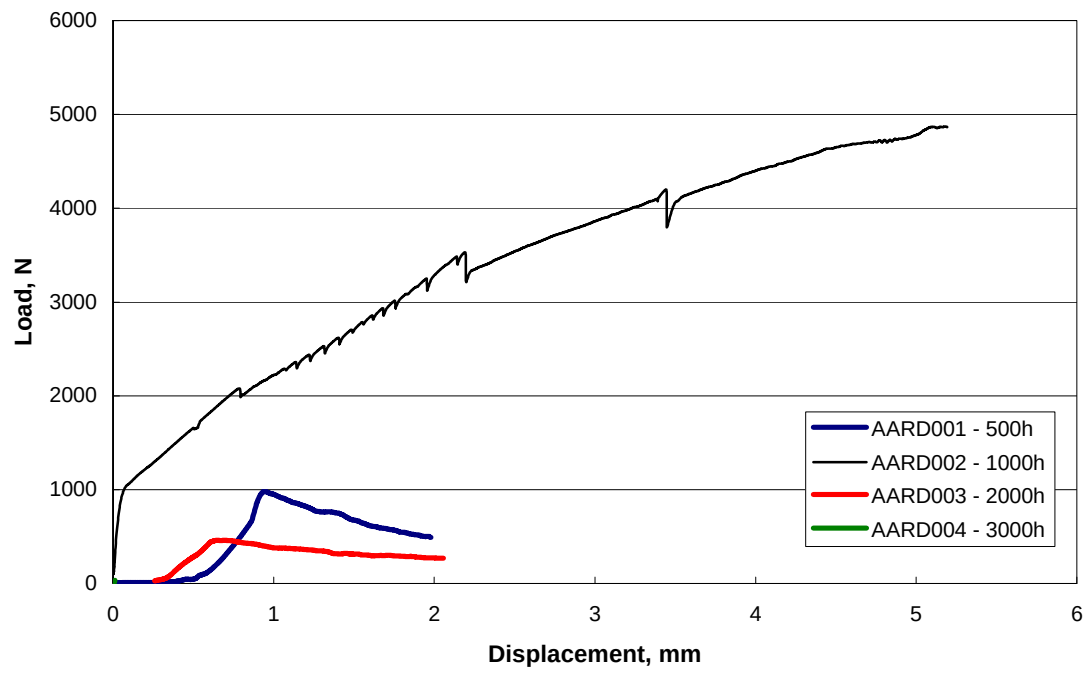


Figure 16 Load vs Displacement plot for IN617 pin in a IN617 collar exposed to steam at 750°C.

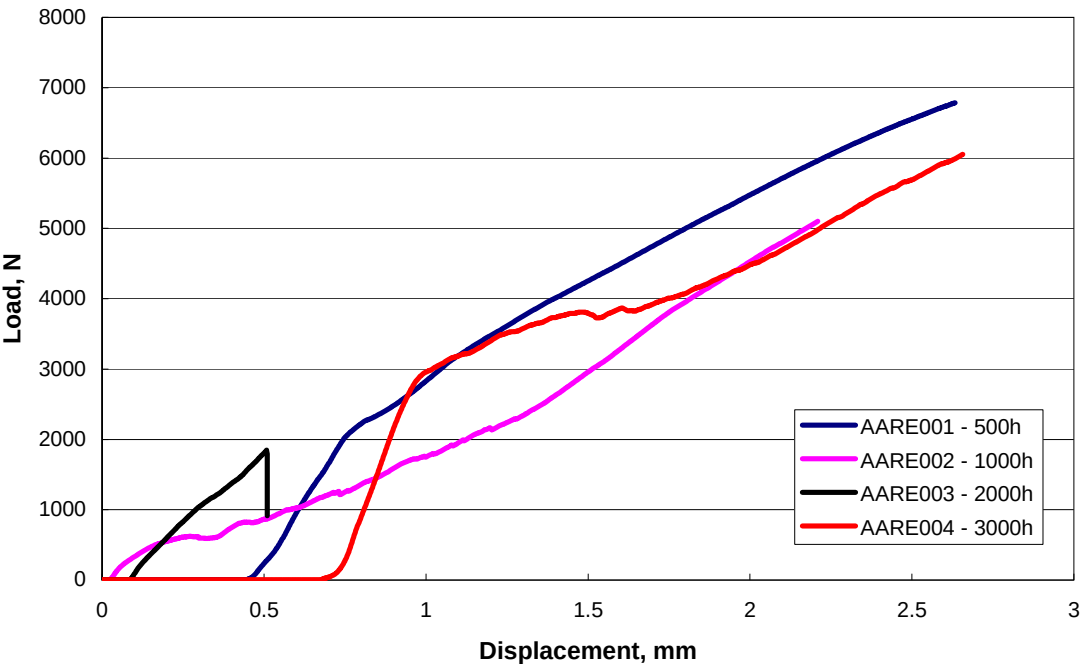


Figure 17 Load vs Displacement plot for P92 pin in a IN617 collar exposed to steam at 650°C.

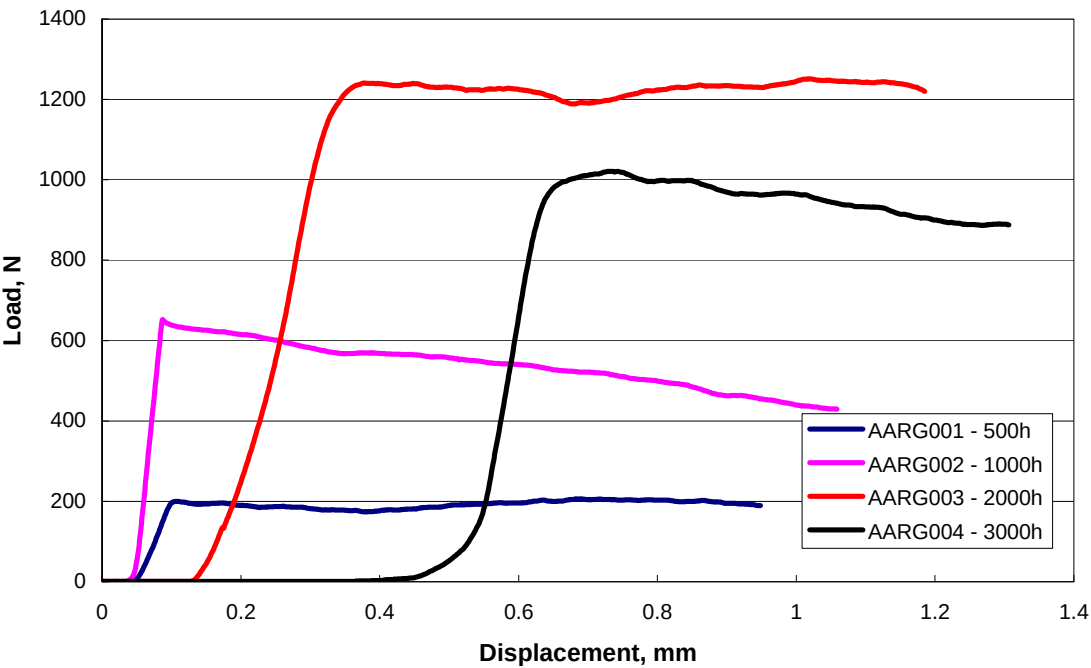


Figure 18 Load vs Displacement plot for IN740 pin in a IN738 collar exposed to steam at 750°C.

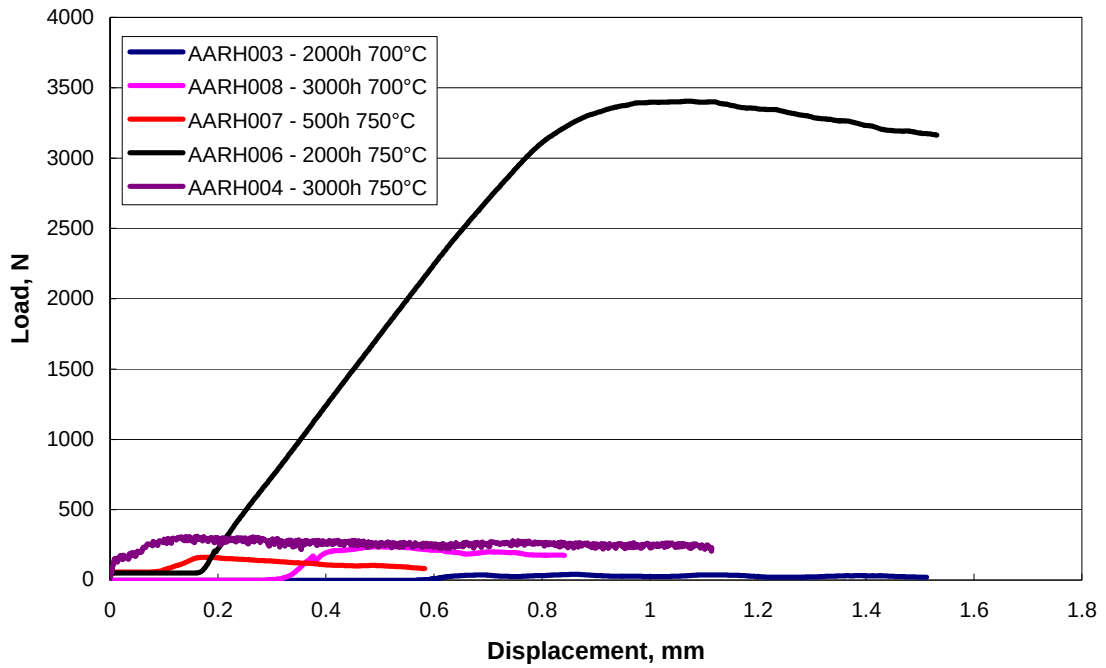


Figure 19 Load vs Displacement plot for IN939 pin in a IN738 collar exposed to steam at 700 and 750°C.

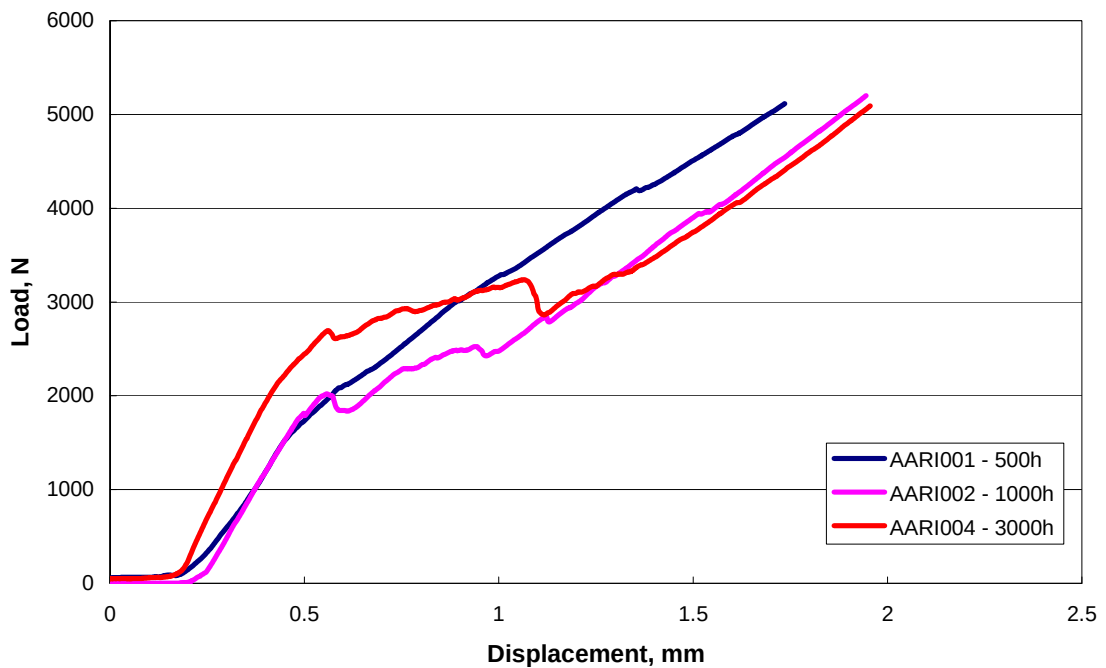


Figure 20 Load vs Displacement plot for P92 pin in a IN738 collar exposed to steam at 650°C.

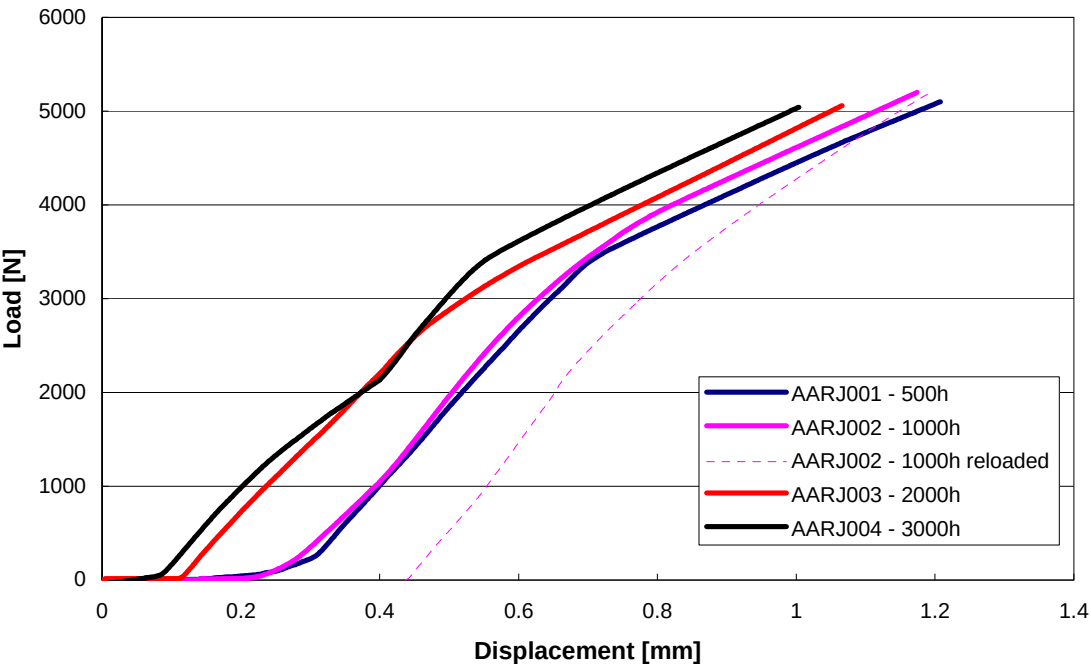


Figure 21 Load vs Displacement plot for IN738 pin in a P92 collar exposed to steam at 650°C.

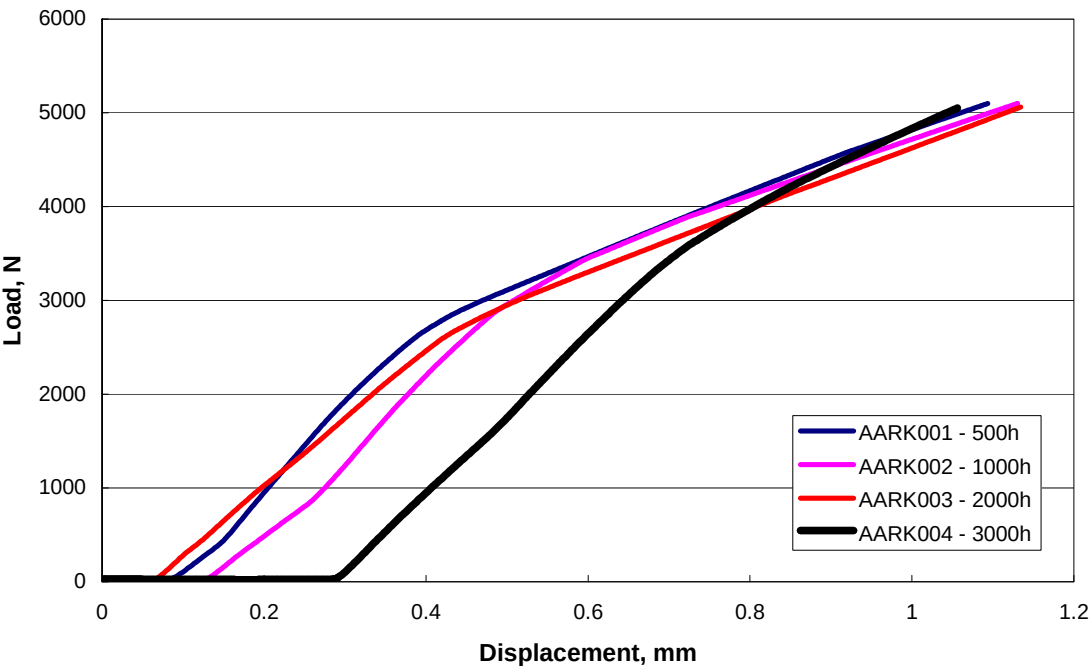


Figure 22 Load vs Displacement plot for IN740 pin in a P92 collar exposed to steam at 650°C.

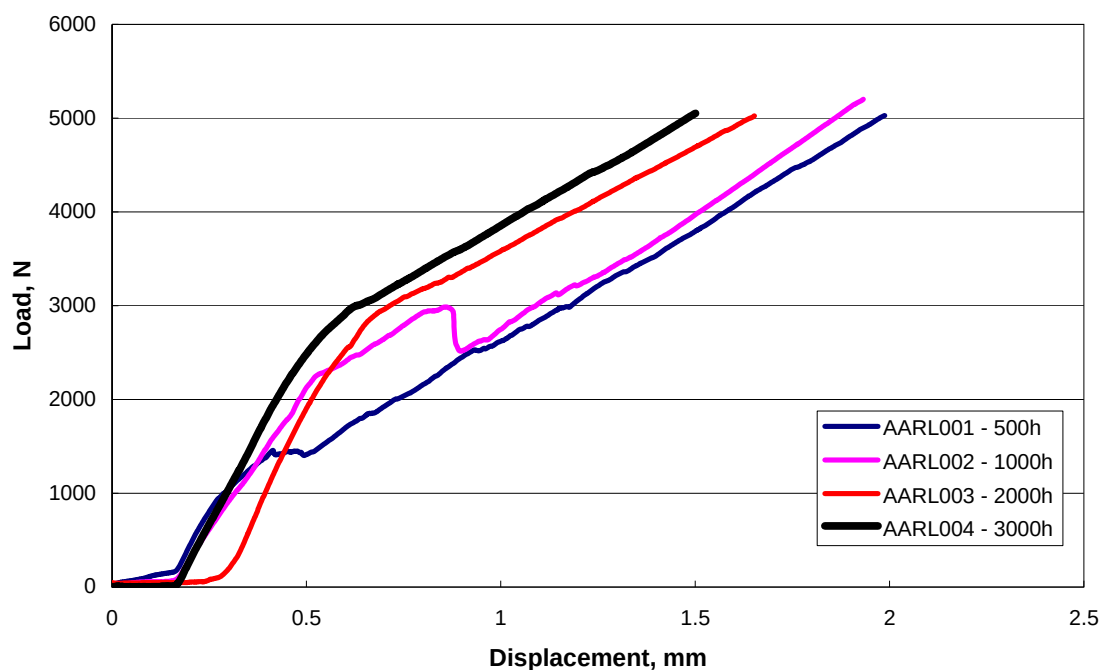


Figure 23 Load vs Displacement plot for P92 pin in a P92 collar exposed to steam at 650°C.

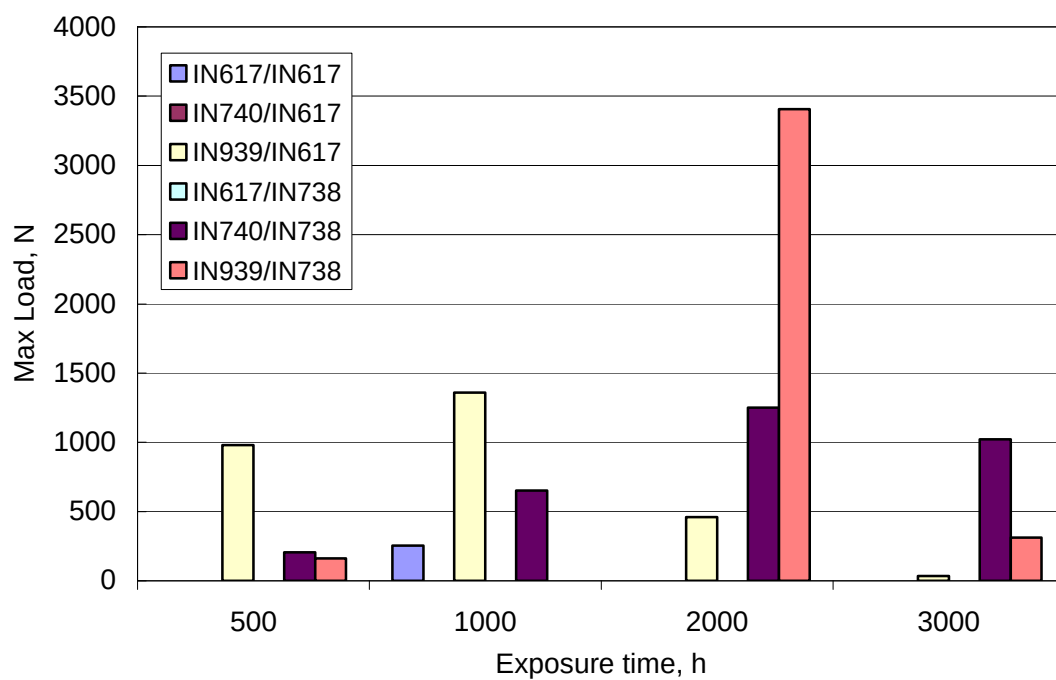


Figure 24 Maximum applied load for specimens exposed at 750°C (pin/collar).

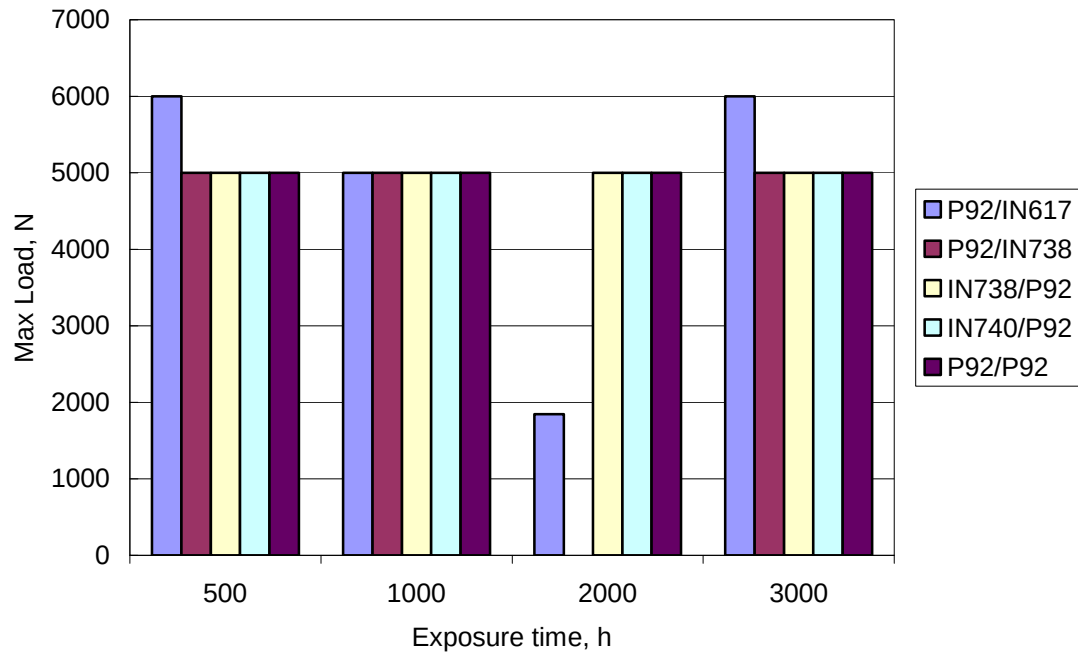


Figure 25 Maximum applied load for specimens exposed at 650°C (pin/collar).

After tensile testing the pins were glued in place and the specimens sectioned. If the specimen had been measured with ultrasound (US) then the section was made through the plane of the US measurement. The oxide thickness was then measured optically and optical micrographs obtained; these are presented in Appendix A. Figures 36 to 39 show the oxide thickness data for specimens from series AARI, AARJ, AARK, AARL and AARE. Specimens consisting of inconel pins in inconel collars did not produce oxides that could be accurately measured optically.

3.1.1 IN617 Pin / IN617 Collar

For this material combination in all but one case the pin was loose in the collar after the exposure. Specimen AARB002 which had been exposed for 1000 hours did require a small force of 250 N to move the pin. Inspection of the micrographs in Appendix A (Figures A1 to A4) shows that there whilst there were areas where the oxide grew, much of the degradation was through internal corrosive attack, as shown in Figure 26. Consequently the outward growing oxide did not achieve a sufficient thickness to bridge the gap between the pin and collar. The specimen that was not loose, AARB002, had a tighter fit in the collar, as shown in Figure A2, in the region at the bottom of the pin on the right hand side. It is conceivable that at this point the two halves were in contact and the resultant measured force was due to discrete contact points.

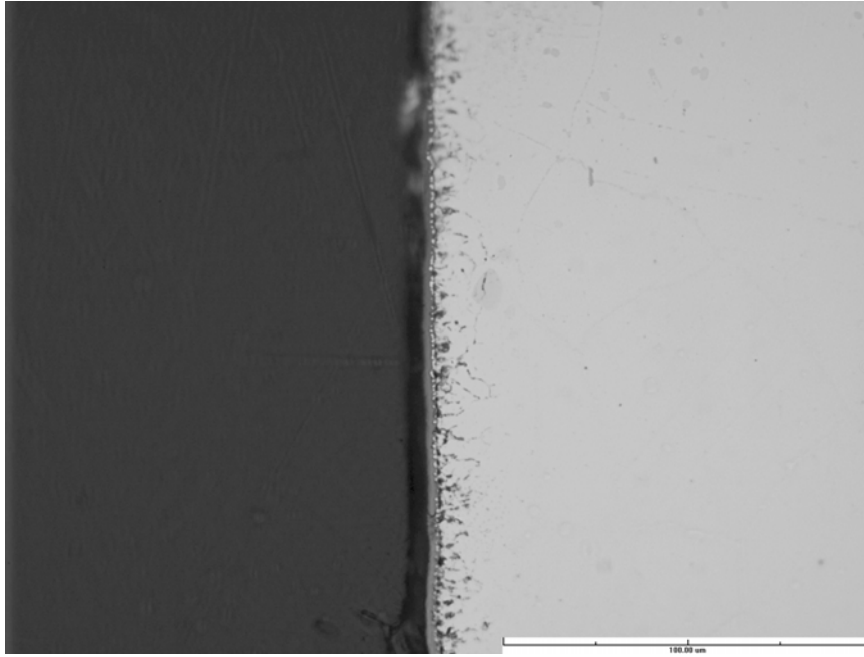


Figure 26 AARB004 (750°C for 3000 h), IN617 material shown.

3.1.2 IN740 Pin / IN617 Collar

All four specimens were loose for this material combination, although fairly extensive internal attack ($\sim 15\ \mu\text{m}$) was found on the 3000 hour specimen as shown in Figure 27. In all cases there was no optical evidence to suggest that the pin and collar had made contact.

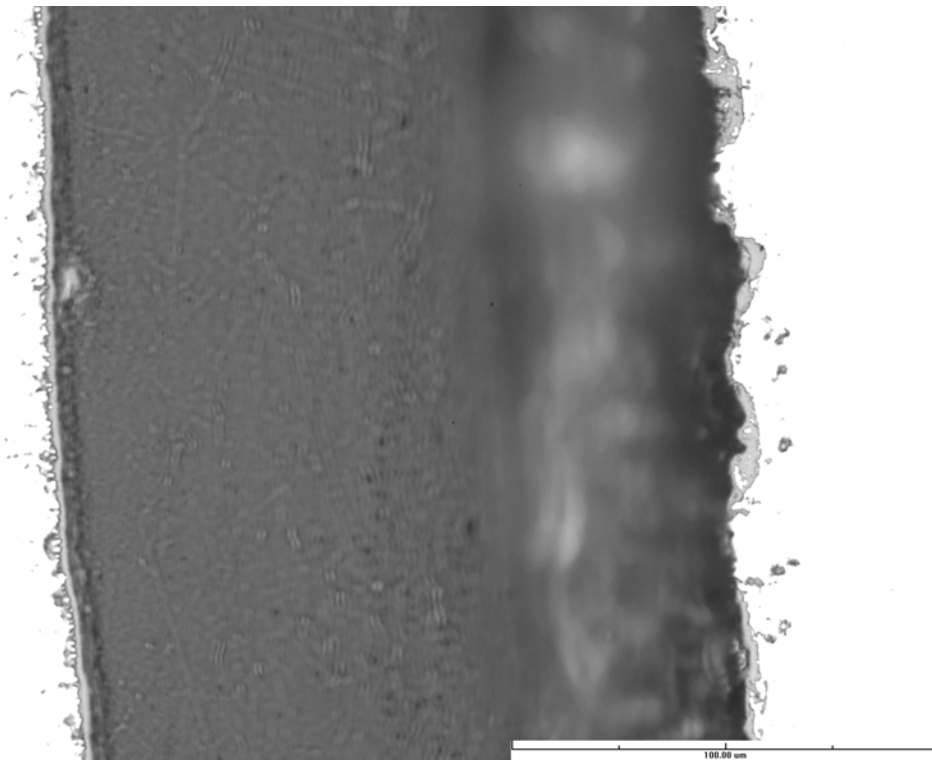


Figure 27 AARC004 (750°C for 3000 h), IN617 on the left hand side, IN740 on the right.

3.1.3 IN939 Pin / IN617 Collar

In the case of the combination of materials all four specimen sets required a load to move the pin. The specimens exposed for 500 and 1000 hours needing the greater load. Examination of the micrographs (Figures A9 to A12) does not explain why this should be the case. It is possible that contact has been made but the specimen has not been sectioned on the appropriate plane. Internal attack is once again evident and in places extends as much as 30 μm , Figure 28.

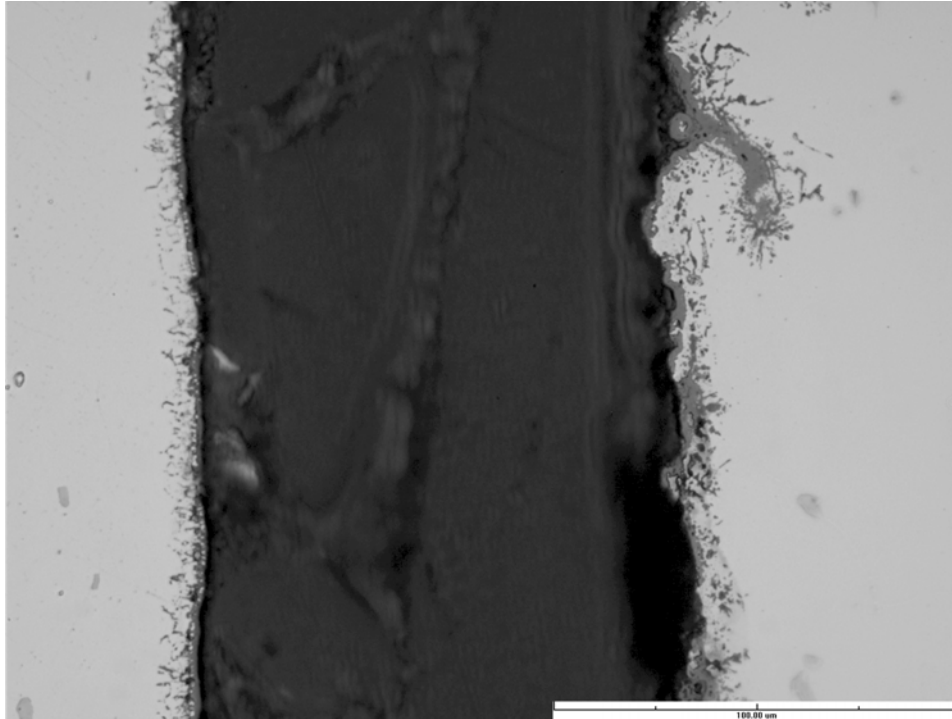


Figure 28 AARD004 (750°C for 3000 h), IN939 on the right, IN617 on the left.

3.1.4 P92 Pin / IN617 Collar

All but one of these specimens required a force greater than 5 kN to move the pin. Examination of the micrographs presented in Figures A13 to A16 show that the oxide grown on the P92 pins completely fills the gaps. The oxide scales formed on the 500 1000 and 3000 hour samples are typical of that which you would expect from this alloy. Consisting of a dense outward growing layer of magnetite and an inward growing layer of Fe-Cr spinel, with each layer being of approximately the same thickness, as shown in Figure A15. A micrograph from a standard isothermal test is shown in Figure 29 for comparison. However the oxides formed in the 2000 hour test show the outward growing magnetite layer to be very porous (Figure 30), which helps to explain the lower strength of the oxide, as shown in Figure 25 and Table II.

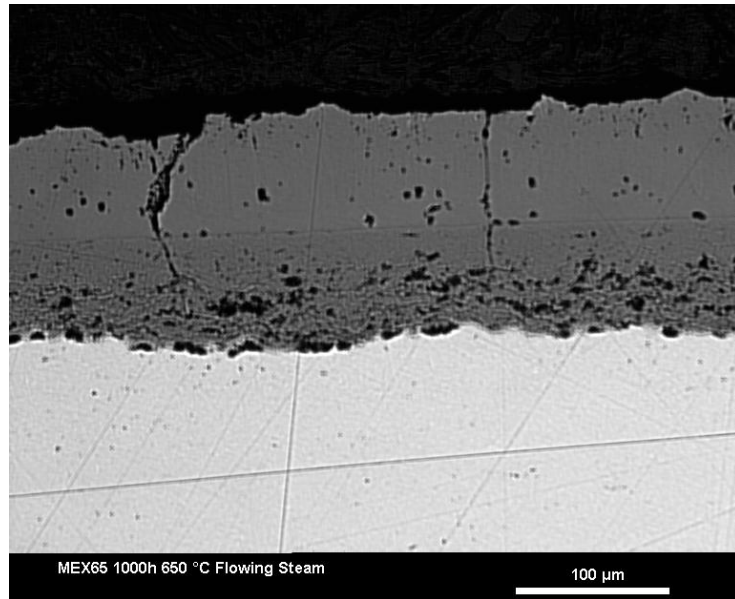


Figure 29 Micrograph of a steam oxidation test on P92 at 650°C for 1000 h.

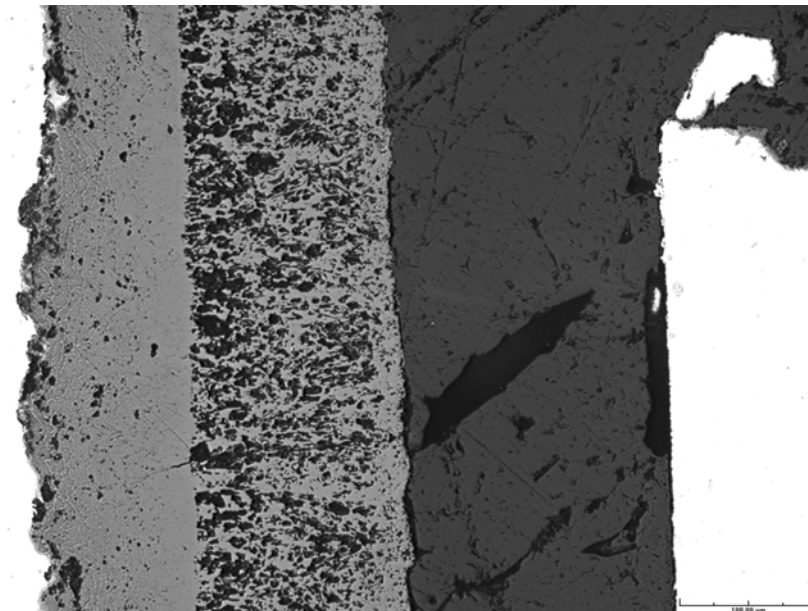


Figure 30 AARE003 (650°C for 2000 h), IN617 on the right, P92 on the left, illustrating the porous structure of the outward growing magnetite.

3.1.5 IN617 Pin / IN738 Collar

All of these samples were found to be loose. Optical examination confirmed that the oxide did not bridge the gap, Figures A17 to A20.

3.1.6 IN740 Pin / IN738 Collar

This combination produced specimens which all required a force to move the pin, this force was seen to increase with increasing exposure time up to 2000 hours. Cross sections, Figures A21 to A24, show that whilst there are similar levels of internal attack

as with other Inconel alloys the actual width of the gap, as sectioned in this plane, was much smaller than with the specimens previously discussed. This tighter fit may account for the increase in the load required to move the pins. In the case of the 3000 hour exposure there were indications of increased levels of outward growing oxide scales. SEM analysis on these specimens is required to investigate this further. An example of this is shown in Figure 31. Whilst not fully understood at this point one explanation for this increased level of oxidation may be due to the initial protective chromia scale being damaged during the exposure and breakaway oxidation attacking the chromium depleted layers below.

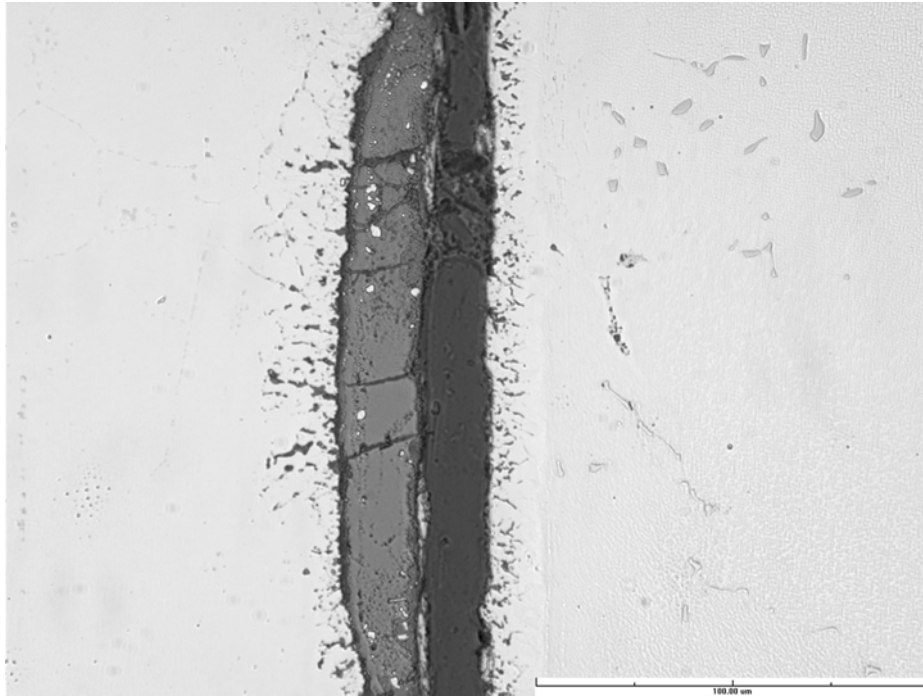


Figure 31 AARG004 (750°C for 3000 h), IN38 on the right, IN740 on the left, illustrating the increased levels of local oxidation.

3.1.7 IN939 Pin / IN738 Collar

In the case of this material pairing, it was found that some of the specimens were adhered and some were not. At 700°C specimens that had been exposed for durations of 2000 and 3000 hours had adhered. At 750°C all but the 1000 hour specimen had adhered. Examination of the cross section, Figures A25 to A32, shows that the pins did not stay central; this is likely to be as a result of the specimens being placed on their sides. This clearly gives areas where there is surface contact. Whilst this may account for the force needed to move the pins, once again there is evidence for the longer-term tests developing areas of higher corrosion, Figure 32.

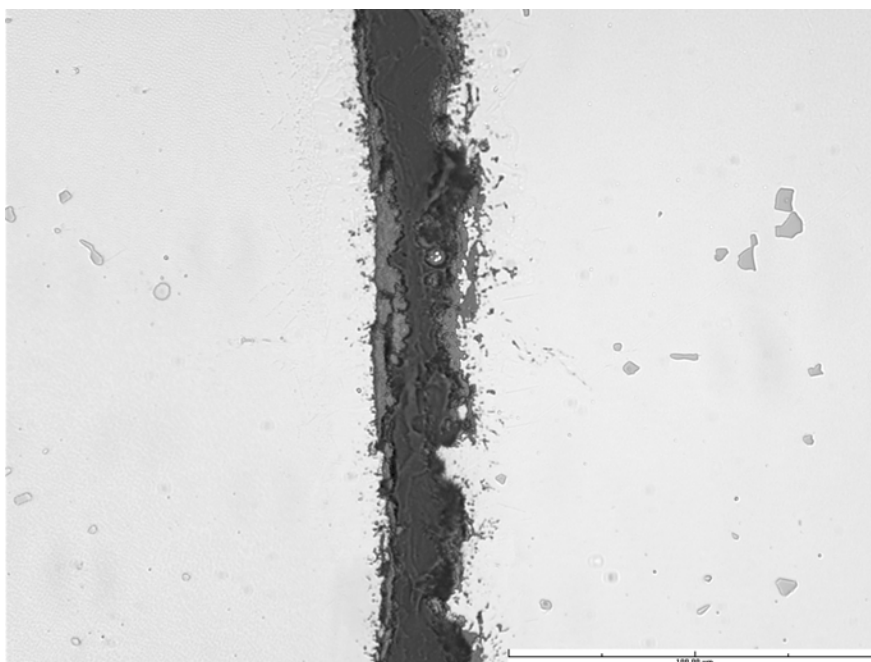


Figure 32 AARH006 (750°C for 2000 h), IN38 on the right, IN939 on the left, illustrating the increased levels of local oxidation.

3.1.8 P92 Pin / IN738 Collar

As expected the P92 oxidises readily and bridges the gap between the pin and the collar in all cases, apart from the 2000 hour exposure. In this case there is no oxide present. It is possible that the materials have become mixed and the pin is not made from P92, or that the specimen was damaged during handling and cutting, further measurement of the alloy composition would confirm whether this is the case, but for the purposes of this report this specimen will be ignored.

The three specimens that had adhered required a force greater than 5 kN to move the pin. The oxides formed were fairly dense within the hole, but towards the neck of the collar the oxide scale became more porous, Figures A33, A34 and A36.

3.1.9 IN738 Pin / P92 Collar

These specimens broadly follow the trends shown in the previous specimens. All of the IN738 pins were constrained by the oxide scale growing from the P92 collar, in all cases needing a force in excess of 5 kN to move the pin. Failure within the oxide is predominantly at the interface between the magnetite and spinel, this is replicated in the majority of the specimens involving P92. Yet again the magnetite layer is very porous especially at the neck of the specimen.

3.1.10 IN740 Pin / P92 Collar

Again there is excessive oxidation of the P92 collar which bridges the gap between itself and the pin. Close observation of the interface between the magnetite and the pin indicates that there is an interaction between the two surfaces with metal from the pin seeming to appear as inclusions in the scale. For an example of this compare the surfaces in Figure 33: this shows an area near the neck where the surfaces have not

contacted, and an area where the two surfaces have met for a specimen exposed for 2000 hours at 650°C.

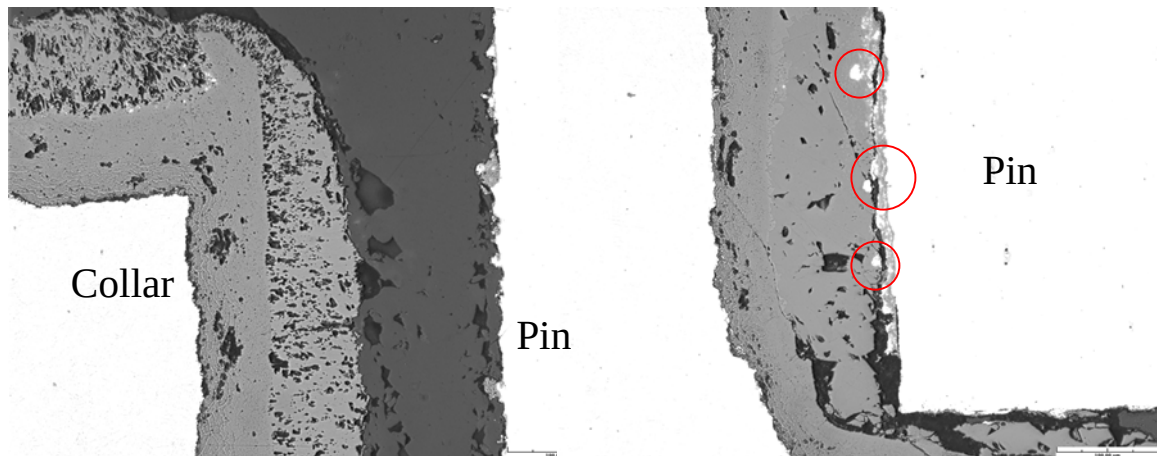


Figure 33 AARK002 (650°C, 1000 h). Image on the left is at the neck of the specimen; image on the right is at the base of the collar. Circled areas show where the IN740 appears as an inclusion in the magnetite.

3.1.11 P92 Pin / P92 Collar

This is the first instance where both components of the specimen will oxidise and potentially meet in the centre of the gap. The micrographs show (Figures A45 to A48) that the oxides grow as would be expected on both P92 surfaces. The inward growing spinel is easily identified on both halves, and the original metal surfaces can also be seen, Figure 34. The magnetite layers however appear from the images to blend together to form one layer of magnetite; the boundaries between each half not being evident.

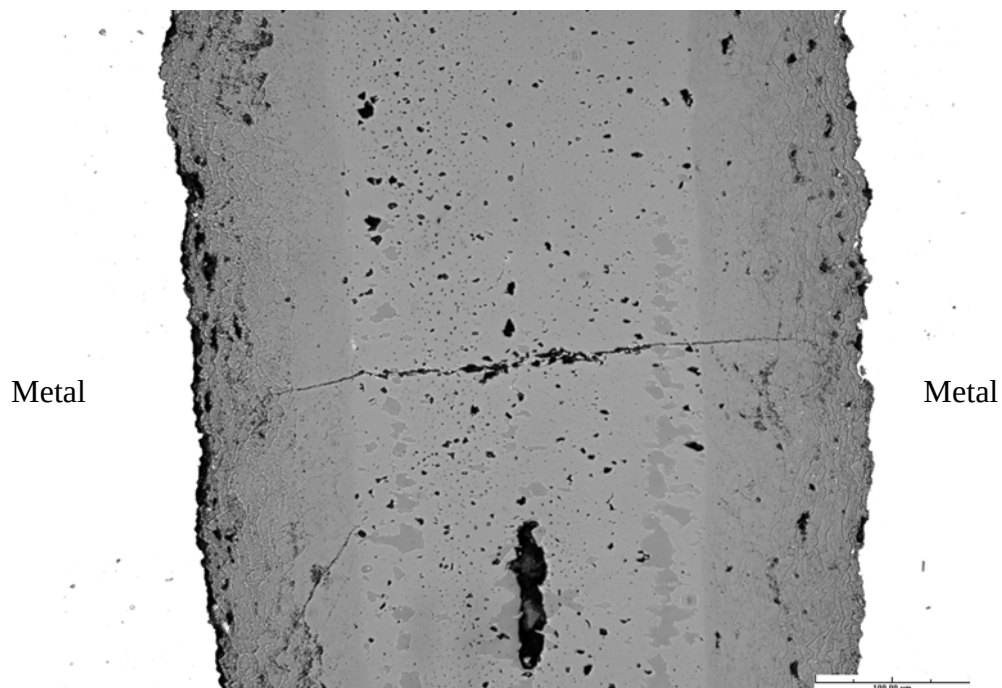


Figure 34 AARL002 (650°C, 1000 h).

3.1.12 Oxide Thickness

Figures 36 to 39 present the results of the oxide thickness measurements. These are heavily influenced by the geometry of the test pieces and the sectional plane, since the specimens were on their sides, inevitably one half will be closer to the wall than the other. Although a minimum gap will be maintained by the spacing pips shown in Figure 1.

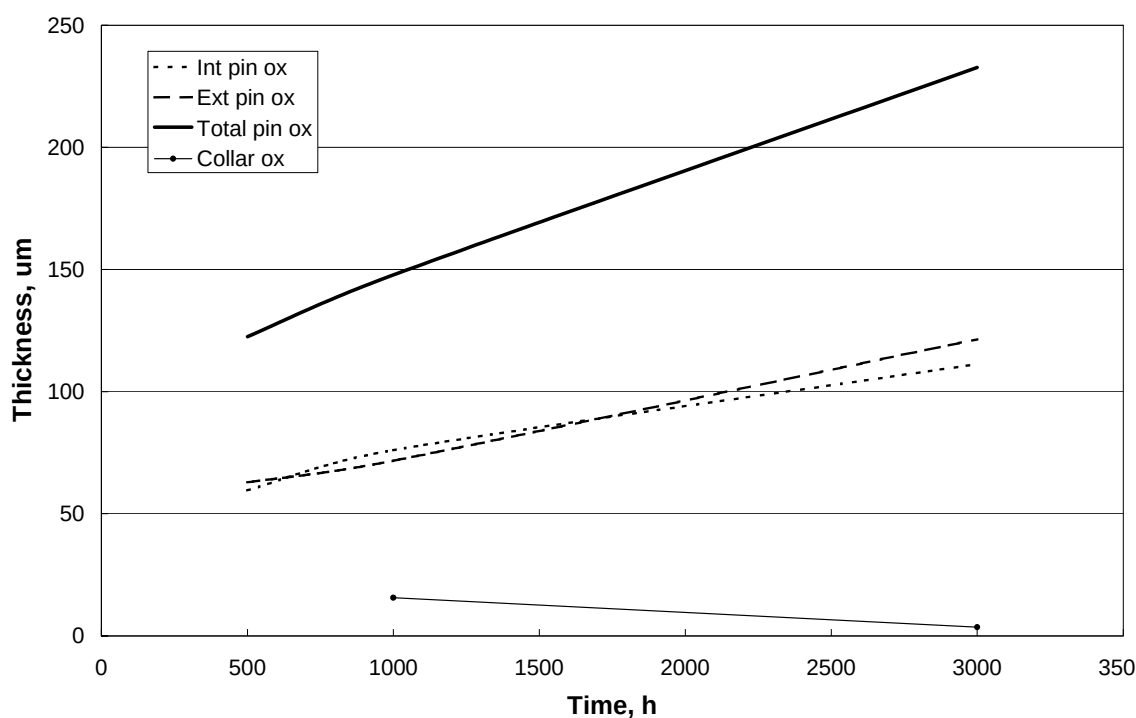


Figure 35 Oxide thickness as a function of time for a P92 pin in an IN738 collar (AARI).

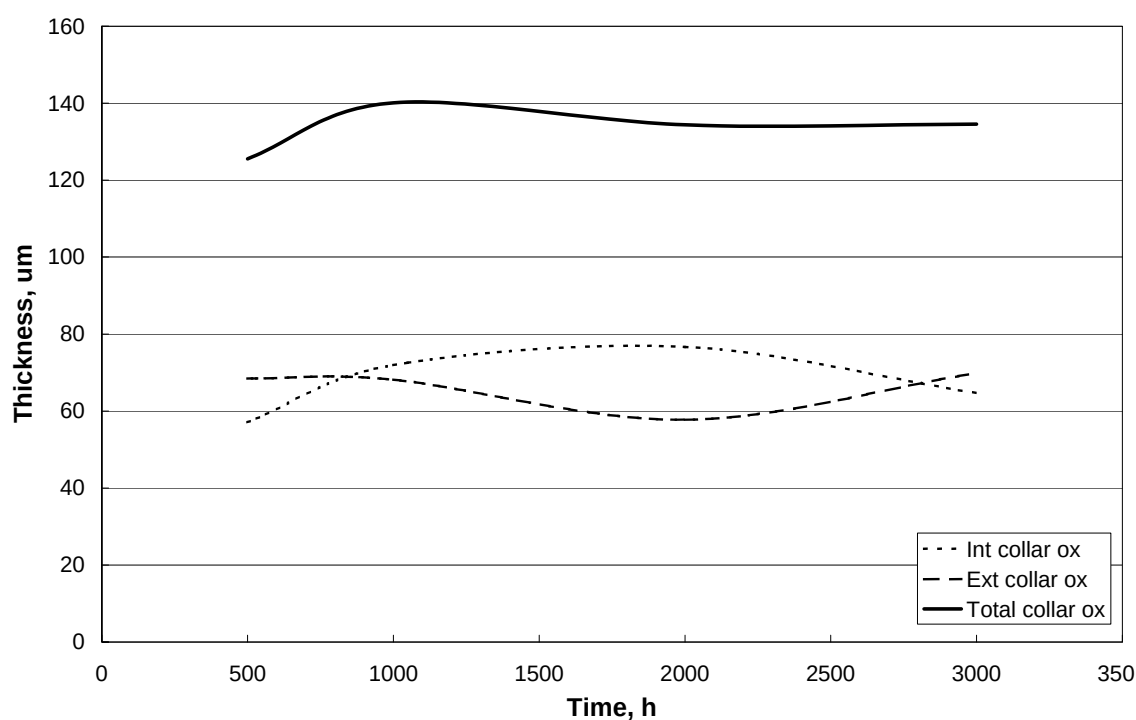


Figure 36 Oxide thickness as a function of time for an IN738 pin in a P92 collar (AARIJ).

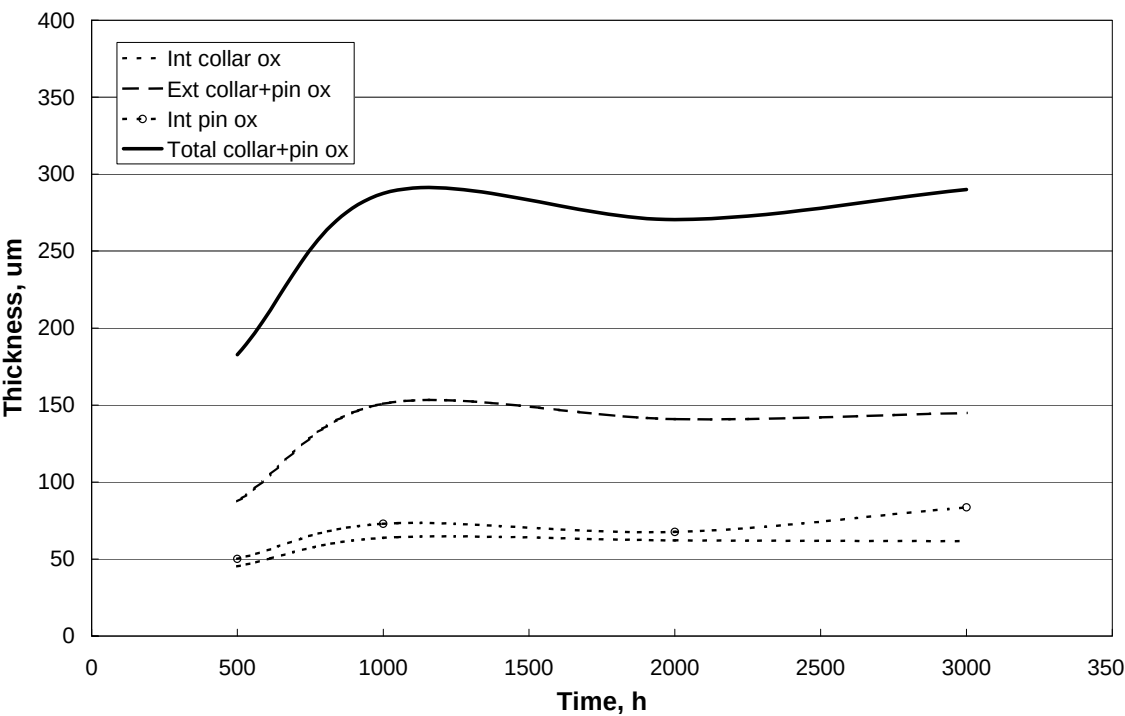


Figure 37 Oxide thickness as a function of time for a P92 pin in a P92 collar (AARL).

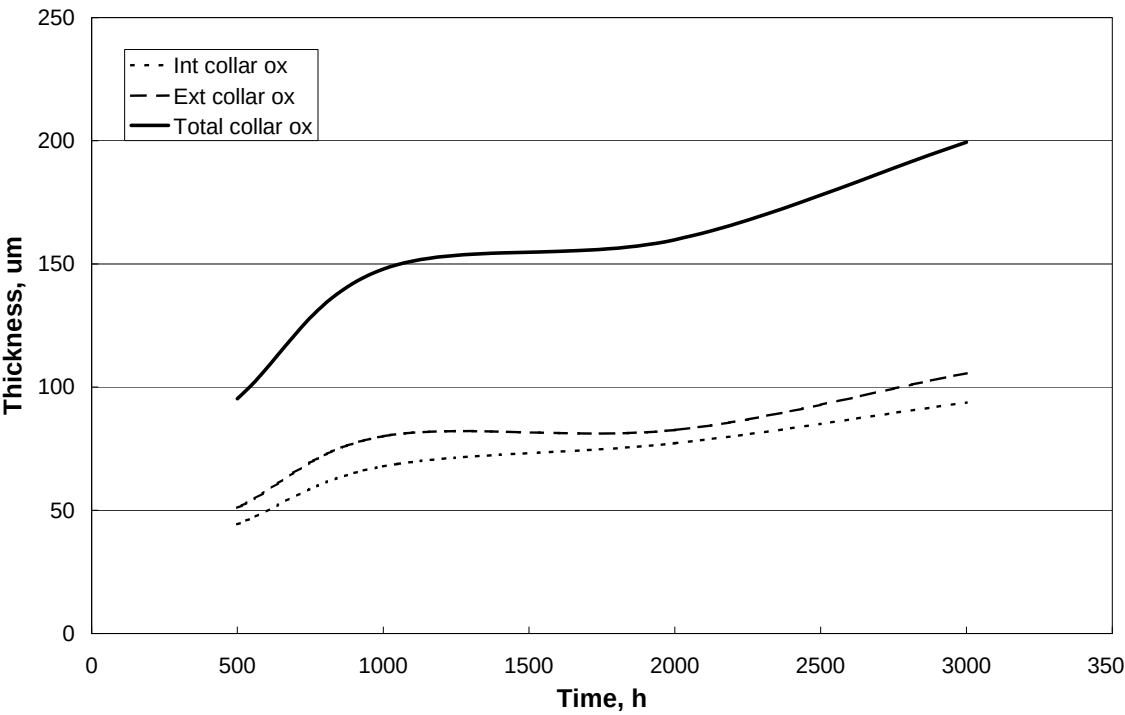


Figure 38 Oxide thickness as a function of time for an IN740 pin in a P92 collar (AARK).

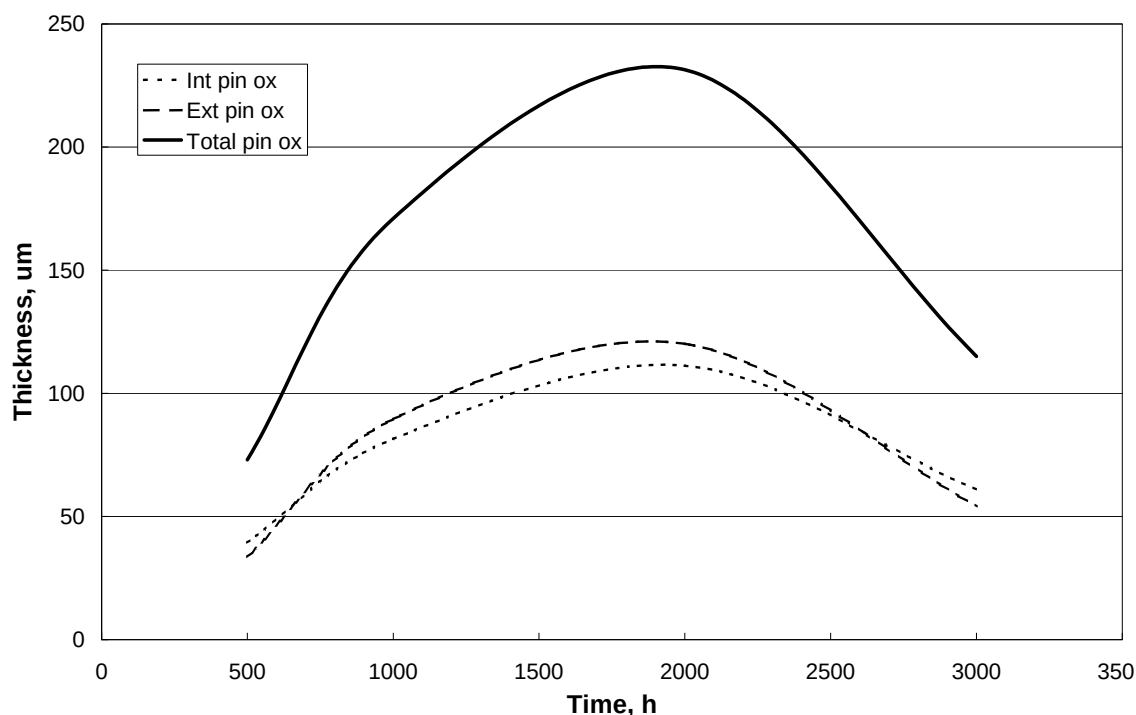


Figure 39 Oxide thickness as a function of time for a P92 pin in an IN617 collar (AARE).

The figures show that the oxide grown on the P92 material maintained the conventional 1:1 ratio of spinel to magnetite thickness. Interestingly this is still the case with the P92/P92 specimens AARL. The oxide thickness results for this series of specimens is shown in Figure 37, this shows that the spinel layer for the pin and collar grows at approximately the same rate and that the combined magnetite layer is twice the mean thickness of a single spinel layer.

The oxide thickness measurements were subsequently used to evaluate the use of ultrasonic techniques to measure the oxide thickness non-destructively [19].

3.2 Dynamic Contacting Surfaces

3.2.1 Low Frequency Tests

The materials tested are given in Table III. The stub sample (left hand side of Figure 10) was manufactured from Co-Ni-Cr-W alloy or Ni-Cr-Co alloy with some samples coated with an MCrAlY (co-deposited electroplate).

Table III Materials Examined.

Stub Material	Coating on Stub	Plate Material	Coating on Plate	Pre-exposure, h
Co-Ni-Cr-W alloy	-	Stellite	-	0
Co-Ni-Cr-W alloy	-	Stellite	-	500
Co-Ni-Cr-W alloy	-	Stellite	-	3000
Co-Ni-Cr-W alloy	-	Stellite	HVOF Cr ₂ C ₃	0
Co-Ni-Cr-W alloy	-	Stellite	HVOF Cr ₂ C ₃	500
Co-Ni-Cr-W alloy	-	Stellite	HVOF Cr ₂ C ₃	3000
Ni-Cr-Co alloy	-	Stellite	-	0
Ni-Cr-Co alloy	-	Stellite	-	500
Ni-Cr-Co alloy	-	Stellite	-	3000
Ni-Cr-Co alloy	-	Stellite	HVOF Cr ₂ C ₃	0
Ni-Cr-Co alloy	-	Stellite	HVOF Cr ₂ C ₃	500
Ni-Cr-Co alloy	-	Stellite	HVOF Cr ₂ C ₃	3000
Ni-Cr-Co alloy	MCrAlY	Stellite	HVOF Cr ₂ C ₃	0
Ni-Cr-Co alloy	MCrAlY	Stellite	HVOF Cr ₂ C ₃	500
Ni-Cr-Co alloy	MCrAlY	Stellite	HVOF Cr ₂ C ₃	3000

Both the stub (top) and plate (bottom) specimens were weighed prior to and after testing. Three sets of specimens were used for each series of tests, comparing as-received with pre-exposed specimens, which were exposed in a flowing steam (ambient pressure, distilled water, ~ 5 ml/min) furnace at an elevated temperature for either 500 or 3000 hours to provide simulation of long term ageing.

Typically tests had a duration of 50 hours in a steam environment. Some additional tests were made for a longer durations in steam, and for the same time without steam for comparative purposes. The distance of travel was 5 mm for each test and took approximately one minute to complete a cycle; a cycle is defined as one reciprocation.

3.2.1.1 Imaging and Analysis of Specimens

After approximately 50 hours of testing, specimens were re-weighed, imaged and the wear scar examined. The data collected was plotted as a function of friction coefficient against time; this will be presented in the results section along with the friction coefficient values.

3.2.1.2 Friction

During testing, the horizontal and vertical load and the horizontal and vertical displacement were logged. The vertical load showed that the dead weight remained constant throughout testing. The horizontal load gave a measure of frictional forces

generated at the contact interface that can be used to calculate friction coefficients. A good method for displaying this is through friction loops where the friction coefficient is plotted against horizontal displacement. The average friction coefficient was also calculated for a single friction loop from the integral and the trend with time plotted. A typical example of these results is shown in Figure 40 for a Ni-Cr-Co alloy stub rubbed against a flat HVOF Cr_2C_3 plate. Both specimens were tested in the as-received condition. Initial and final friction coefficient loops (25 cycles) are plotted against horizontal direction, shown in Figures 40(a) and 40(b) respectively. The average friction coefficient was plotted against time, shown in Figure 40(c). There was a general increase in friction with time, although there were occasional fluctuations in the friction.

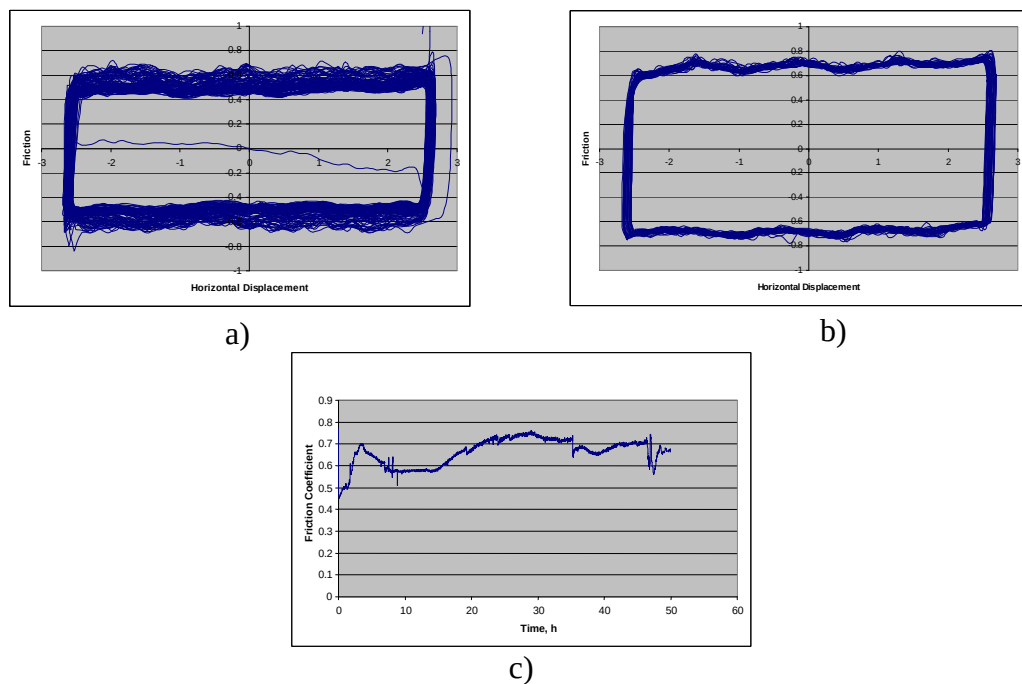


Figure 40 Plot of Friction Loops a) Initial Friction, b) Final Friction and c) Overall Friction vs. Time.

Although the majority of tests were performed in a steam environment, one test was carried out on a different material with and without steam to observe what effect the addition of steam had to the test. As shown in Figure 41 with the addition of steam it can be seen that the friction coefficient is reduced.

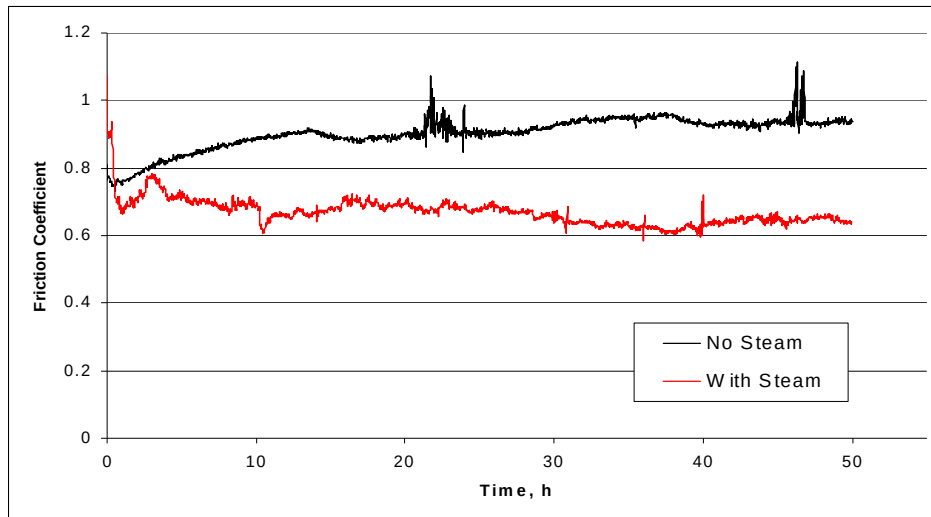


Figure 41 Comparison of Environment, Steam vs. No Steam.

Figure 41 shows a comparison of friction results for different materials in the as-received condition. The MCrAlY coating has a much higher friction coefficient, reaching a maximum of 1.4, than the other materials tested which showed a friction coefficient of between 0.5 and 0.7.

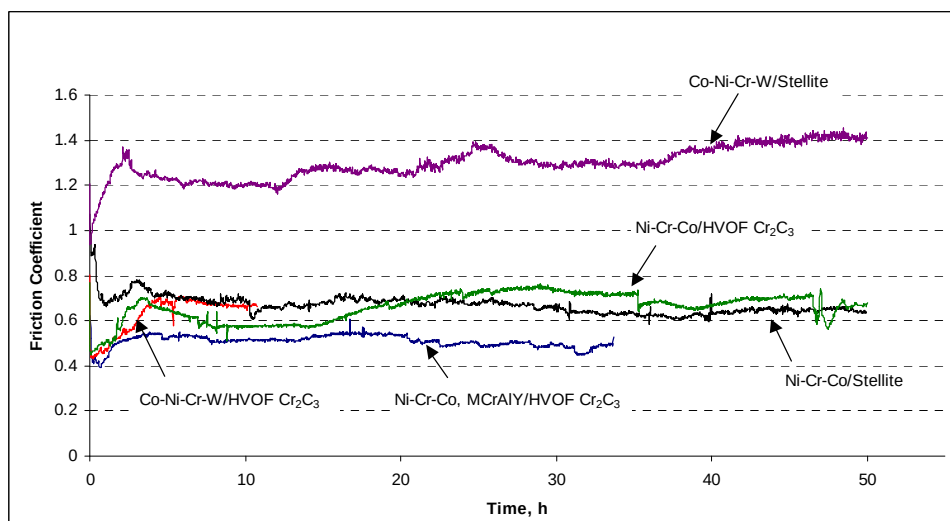


Figure 42 Comparison Between un-Aged Materials.

The results shown in Figure 42 for as-received samples can also be compared with the results shown in Figure 43 for materials that had been pre-aged for 3000 hours in a steam environment. It can be seen that the sample with the MCrAlY coating is the only test in which the friction coefficient actually decreased with pre-ageing.

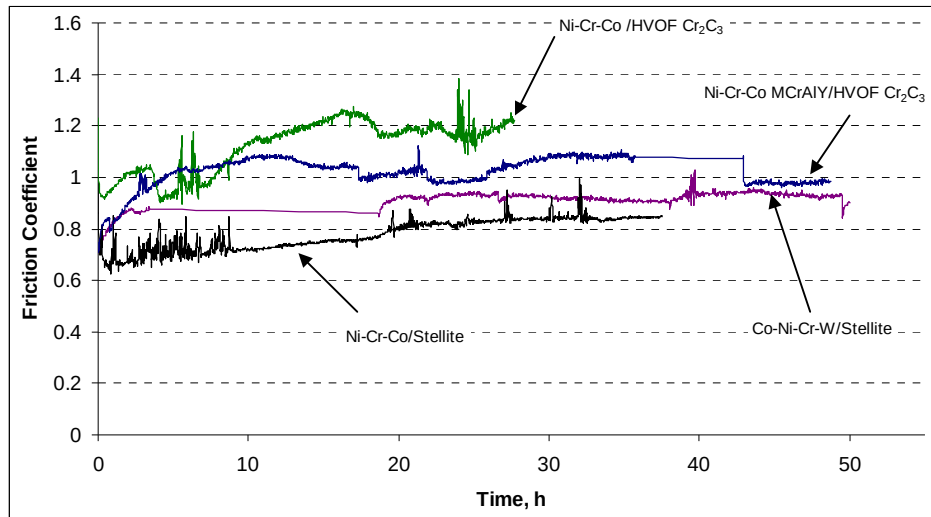
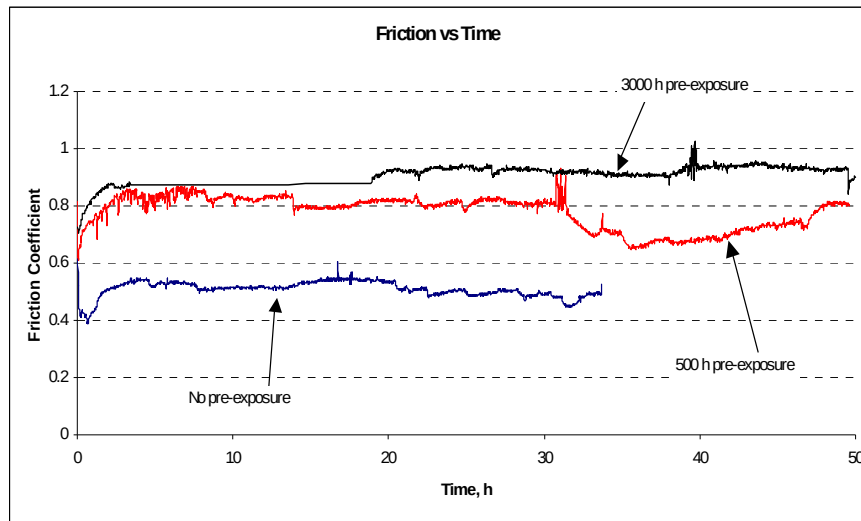
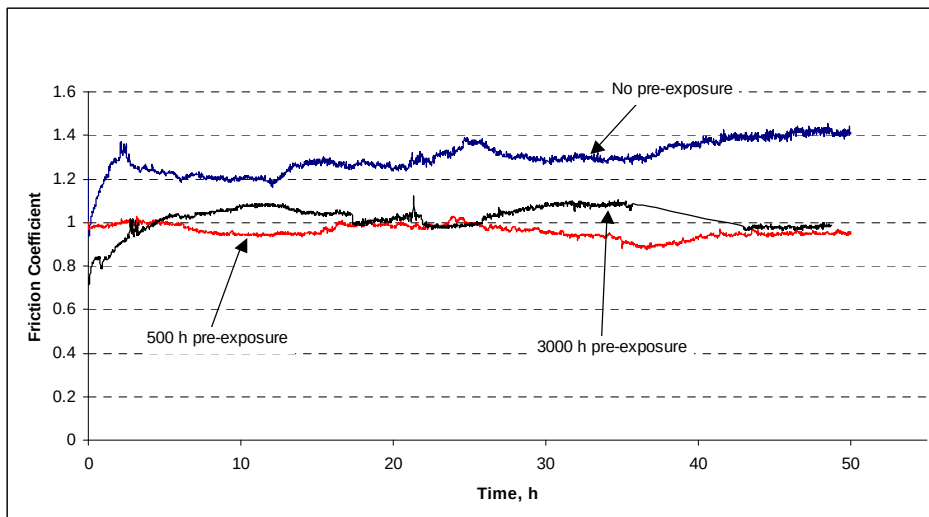


Figure 43 Comparison of Pre-Aged for 3000 h Specimens.

Tests were performed on as-received and pre-exposed specimens to compare the affect of 'aged' material. Results are presented in Figure 44. For the Co-Ni-Cr-W alloy against stellite material, ageing increases the friction. By contrast, with the MCrAlY coating, pre-ageing markedly decreases the friction coefficient.



a)



b)

Figure 44 Comparison of As-Received and Aged Material; a) Co-Ni-Cr-W alloy Against Stellite and b) MCrAlY Coated Stub Against Cr_2C_3 .

Table IV gives a summary of the final friction results with respect to material and ageing treatment.

Table IV Final Friction Coefficients.

Stub Material	Coating on Stub	Plate Material	Coating on Plate	Pre-exposure, h	Final Friction
Co-Ni-Cr-W alloy	-	Stellite	-	0	0.5
Co-Ni-Cr-W alloy	-	Stellite	-	500	0.8
Co-Ni-Cr-W alloy	-	Stellite	-	3000	0.95
Co-Ni-Cr-W alloy	-	Stellite	HVOF Cr ₂ C ₃	0	0.68
Co-Ni-Cr-W alloy	-	Stellite	HVOF Cr ₂ C ₃	500	0.9
Co-Ni-Cr-W alloy	-	Stellite	HVOF Cr ₂ C ₃	3000	-
Ni-Cr-Co alloy	-	Stellite	-	0	0.65
Ni-Cr-Co alloy	-	Stellite	-	500	0.65
Ni-Cr-Co alloy	-	Stellite	-	3000	0.85
Ni-Cr-Co alloy	-	Stellite	HVOF Cr ₂ C ₃	0	0.7
Ni-Cr-Co alloy	-	Stellite	HVOF Cr ₂ C ₃	500	0.85
Ni-Cr-Co alloy	-	Stellite	HVOF Cr ₂ C ₃	3000	1.2
Ni-Cr-Co alloy	MCrAlY	Stellite	HVOF Cr ₂ C ₃	0	0.95
Ni-Cr-Co alloy	MCrAlY	Stellite	HVOF Cr ₂ C ₃	500	0.95
Ni-Cr-Co alloy	MCrAlY	Stellite	HVOF Cr ₂ C ₃	3000	1.0

3.2.1.3 Wear

Two measures of wear have been used. Mass change results are given in Table V. These show that the mass of the stub sample always increased. This shows that the dominant contribution to mass change for the stub sample is oxidation rather than wear. In most cases the mass gain during the test decreased as the pre-exposure time increased. This is likely to be due to a protective oxide scale forming during the ageing process which provided a barrier to further oxygen diffusion. Further work to examine the oxide scale thickness and microstructure will verify if this is the case, but is outside of the scope of this report. The mass of the plate samples always decreased showing that the contribution to mass change for these samples from wear (loss) was higher than the contribution from oxidation (gain).

Table V Mass Change Results.

Stub Material	Coating on Stub	Pre-Exp, h	Mass Gain, mg	Plate Material	Coating on Plate	Pre-Exp, h	Mass Gain, mg
Co-Ni-Cr-W alloy	-	0	0.45	Stellite	-	0	-0.07
Co-Ni-Cr-W alloy	-	500	-	Stellite	-	500	-0.33
Co-Ni-Cr-W alloy	-	3000	0.19	Stellite	-	3000	-0.16
Co-Ni-Cr-W alloy	-	0	0.48	Stellite	HVOF Cr ₂ C ₃	0	-1.38
Co-Ni-Cr-W alloy	-	500	0.15	Stellite	HVOF Cr ₂ C ₃	500	-23.84
Co-Ni-Cr-W alloy	-	3000	0.1	Stellite	HVOF Cr ₂ C ₃	3000	-41.84
Ni-Cr-Co alloy	-	0	0.37	Stellite	-	0	-0.06
Ni-Cr-Co alloy	-	500	0.07	Stellite	-	500	-0.24
Ni-Cr-Co alloy	-	3000	0.03	Stellite	-	3000	-0.3
Ni-Cr-Co alloy	-	0	0.36	Stellite	HVOF Cr ₂ C ₃	0	-3.5
Ni-Cr-Co alloy	-	500	0.05	Stellite	HVOF Cr ₂ C ₃	500	-23.28
Ni-Cr-Co alloy	-	3000	-	Stellite	HVOF Cr ₂ C ₃	3000	-
Ni-Cr-Co alloy	MCrAlY	0	1.11	Stellite	HVOF Cr ₂ C ₃	0	-0.32
Ni-Cr-Co alloy	MCrAlY	500	0.14	Stellite	HVOF Cr ₂ C ₃	500	-22.04
Ni-Cr-Co alloy	MCrAlY	3000	0.23	Stellite	HVOF Cr ₂ C ₃	3000	-9.96

The results of the measurements using the LVDT to monitor vertical displacement showed little change during tests.

3.2.1.4 Visual Inspection of Wear Scar

In many cases, despite the attention given to sample alignment in the design of the test system, it was found that full contact was not achieved over the sample surfaces. Thus in Figure 45 wear is only observed at the bottom corner of the stub, with the wear scar on the plate which runs across the bottom of the sample consistent with this. In Figure 46 there is severe scoring along the bottom of the stub. Close examination of the sample shows that the stub was in complete contact at the beginning of the test, however, it appears that particles may have been ripped out of the plate by the stub and then ploughed back into the base plate forming scores that are visible along the bottom of the contact area. Figure 47 shows a sample set with even wear across the stub, indicating that the contact between the surfaces was flat. Figure 47b shows a particle at the right edge of the wear scar which has evidently caused the heavy scoring to the right of the particle.

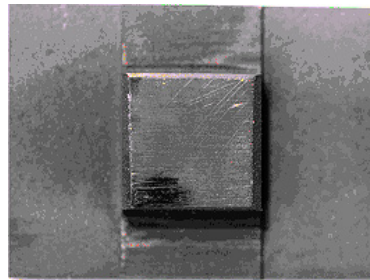
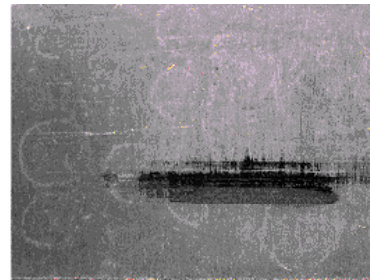
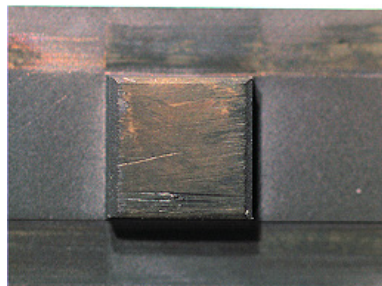


Figure 45

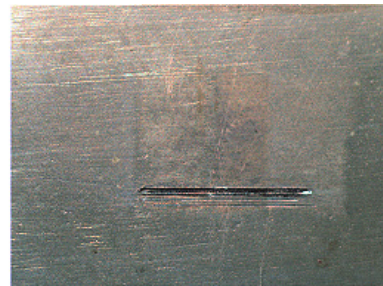


b)

Figure 45 Wear to the bottom corner of (a) stub and (b) along plate.

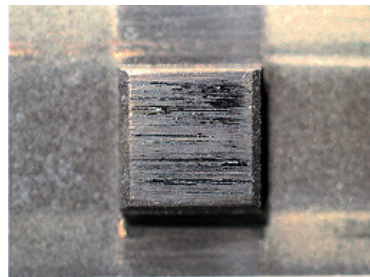


a)

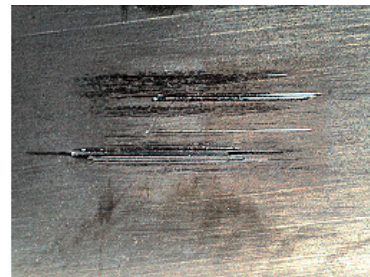


b)

Figure 46 Scoring to the bottom of (a) stub and (b) along plate.



a)



b)

Figure 47 Image of worn specimens a) top stub and b) bottom plate.

3.2.2 High Frequency Tests

Only preliminary tests were carried out with the high frequency test system. These showed that good control of the test system could be achieved. A typical friction result of a test of Nimonic 101 against Waspaloy tested at a temperature of 720°C is shown in Figure 48.

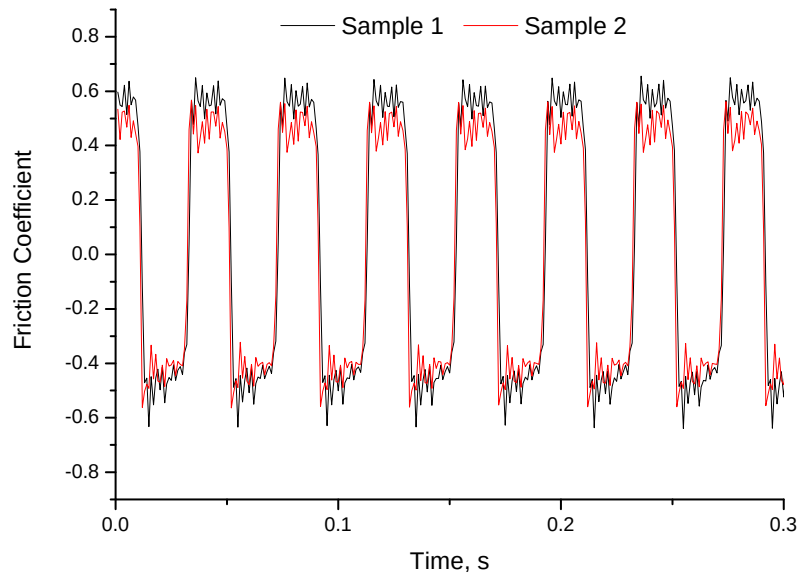


Figure 48 Friction coefficient results for test of Nimonic 101 versus Waspaloy at 720°C.

The tests showed that sample alignment was improved with a greater contact area than in the low frequency tests.

4 Discussion

Two novel test methods have been developed for the evaluation of the steam oxidation properties of contacting surfaces under static and dynamic conditions. Tests performed under static conditions have shown that the test geometry used can produce adequate results. However, there are also clear indications that this test is not suitable for all materials and that the test geometry can be improved. One of the aims of these tests was to produce samples that not only could be used to measure the strength of oxides, but also to produce a geometry that was amenable to oxide thickness measurement through non-destructive means. The results presented in this report for the static contact tests show that the specimen geometry is generally too long, and that rather than using a pin in a collar geometry, a cuboid in a slot would provide an improved solution, Figure 49. This would consist of two specimen halves which are supported in an inert holder, thus removing the use of spacing lugs and maintaining an even separation around the specimen. Such a geometry would be easier to expose in furnaces and would be easier to measure ultrasonically (see [19] for details on the issues encountered), although may be prone to edge effects.

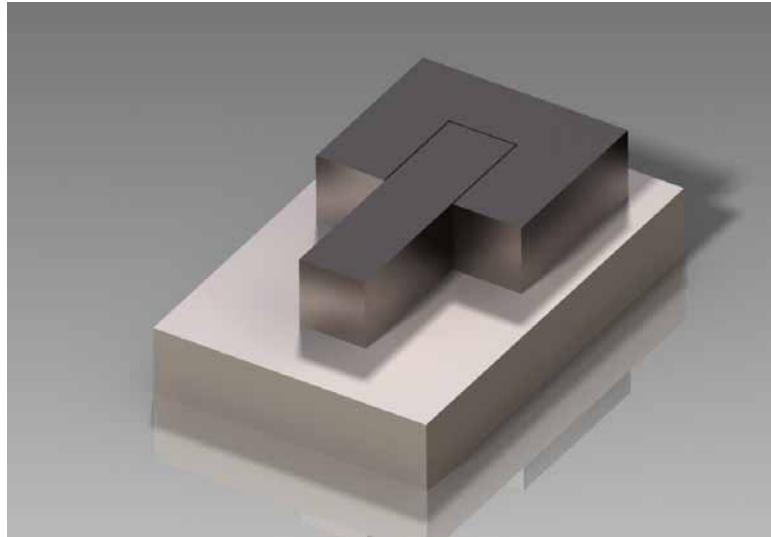


Figure 49 Proposed design of the modified contacting surfaces specimen.

The results of the load and oxide thickness, clearly shows a link between the two, with an increasing load being required as the oxide thickness increases. Unfortunately the geometry of the test pieces prevented securing gripping in the tensile test apparatus and so for many samples the load essentially saturated before clear sliding of the pin was seen. However, closer inspection of the load/displacement plots shows either perturbations in the curves or changes in slope. If we infer that these are an initial failure in the oxide and then plot these values (where they can be clearly identified) against the mean oxide thickness then it is possible to obtain a reasonably straight-line correlation between the oxide thickness and strength. This is shown in Figure 50.

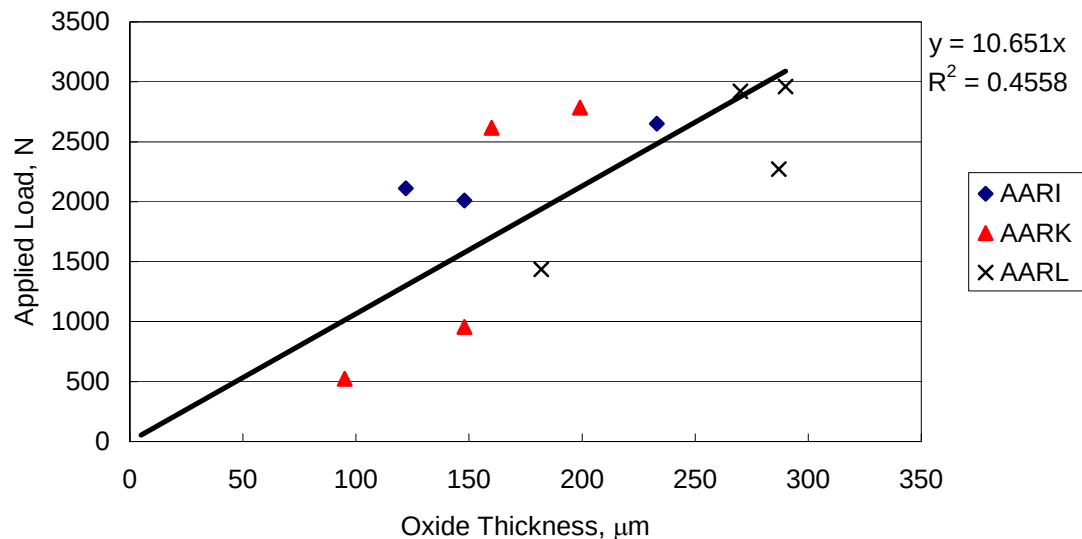


Figure 50 Relationship between the oxide thickness and the estimated load required to cause initial failure in the oxide.

Although there were issues with the specimen design and not maintaining a fixed separation, the results suggest that with better control of the specimen, though the new design, then quantitative data on the oxide strength as a function of thickness is

obtainable. Moreover, with the modified design, it is possible to better control the separation which could make the test applicable to the more corrosion resistant materials.

In the case of surfaces under dynamic contact, a low frequency rig has been developed and has proved to be a useful tool in the evaluation of the tribological performance of materials intended for use in high temperature operation in steam turbines. The water injection system performed well, proving to be a reliable way to generate the necessary steam environment for the testing. This test system has the capability for operation at temperatures up to about 900°C with nimonic sample holders, and up to 1400°C with ceramic sample holders.

The friction force measurements are converted to friction coefficient values and are initially displayed as friction loops. No sign of stiction was observed in the tests (initial peak of friction when samples are stationary with respect to one another), showing that the dynamic friction coefficient is as large in these experiments as the static friction coefficient.

When average friction trends were calculated from the friction loop data, it was clear that although overall trends for the evolution of friction with time were clear in many tests, there were also many sudden changes or fluctuations in friction that were observed. These sudden changes are likely to be related to specific tribological events such as build up and sudden breakaway of oxide films or transfer layers at the contact interface, or increases due to the scoring and grooving events that were observed.

The presence of steam appears to affect the behaviour of the friction coefficient. As shown within this work, when steam is present, the friction decreases. As condensation does not occur between the specimens the steam cannot act as a lubricant. It is quite likely that this effect of steam is due to the influence of water vapour on the formation of oxides at the contact interface. For example the oxidation of the contact area may well lubricate the wear scar.

The pre-aged specimens have a higher friction coefficient compared with the same material tested in the as-received condition. This is also likely to be due to the formation of oxide layers for the pre-aged samples that lead to higher friction coefficients. The exception to this behaviour is the MCrAlY coating which showed a higher friction coefficient than any of the other materials and is the only material tested that shows a decrease in friction coefficient with pre-ageing. The MCrAlY coating is well known to form lubricious oxide layers that contain cobalt when aged.

The lack of full contact over the sample faces in many of the tests was disappointing, but in practice this was due more to the low wear that was encountered in the testing rather than alignment as such. Obtaining full contact between two flat samples is very difficult in tribological testing, so that full contact is only achieved if the samples undergo considerable wear, or the test duration is very long.

A major feature of many of the worn surfaces that were examined is the presence of many scores across the surface that had caused quite deep damage and had roughened the surface locally. These scores were often associated with particles that were attached to the surface of the wear scar at the end of one of these scores. These observations

suggest that strong bonds developed at localised points in contact between the two surfaces. Particles from one surface were pulled out from the surface to form hardened protuberances that then were dragged across the opposing surface to cause the deep scoring and damage that was observed.

Additional work is required to fully elucidate the mechanisms of wear that have been occurring in the tests that have been carried out and reported here, thus further examination of the test pieces is necessary. This will consist of closer examination using the SEM and EDX of the surfaces to observe if any material exchange between specimens has occurred and to study formation of oxides. Cross sections of specimens will permit closer examination of the wear scar and allow depth measurements of the thickness of any oxide scales that have been formed to be made.

5 Conclusions

Three test methods have been developed to evaluate the properties of contacting surfaces at high temperature under static and dynamic loading conditions. The performance of these new tests has been evaluated using a range of industrially relevant materials and conditions. Generally the tests were found to produce the effects and results anticipated at the outset of this work, but some limitations in the test design have been highlighted, particularly in the static test. As a consequence an improved specimen design has been recommended which will provide better control of the test and produce specimens better suited to mechanical and non-destructive testing.

The results of the static tests have shown that it is possible to have two oxide fronts grow into each other to form one monolithic oxide scale. Also as the oxides thicken the force required to crack the oxide/move the pin increases. This appears to be a broadly linear relationship.

Dynamic tests have shown that the addition of steam in a tribological environment clearly influences the friction between materials. Pre-ageing of specimens also influences the friction, with the majority of materials experiencing an increase in friction coefficient, the only exception being the MCrAlY coated stub, due to its lubricious properties which form during testing.

6 Acknowledgement

The authors are grateful to Jim Banks, David Laing and John Nunn for their technical expertise, and would like to acknowledge funding from the Technology Strategy Board and from the Department of Business Innovation and Science for the work reported in this document.

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Appendix A – Micrographs of Pin and Collar Specimens

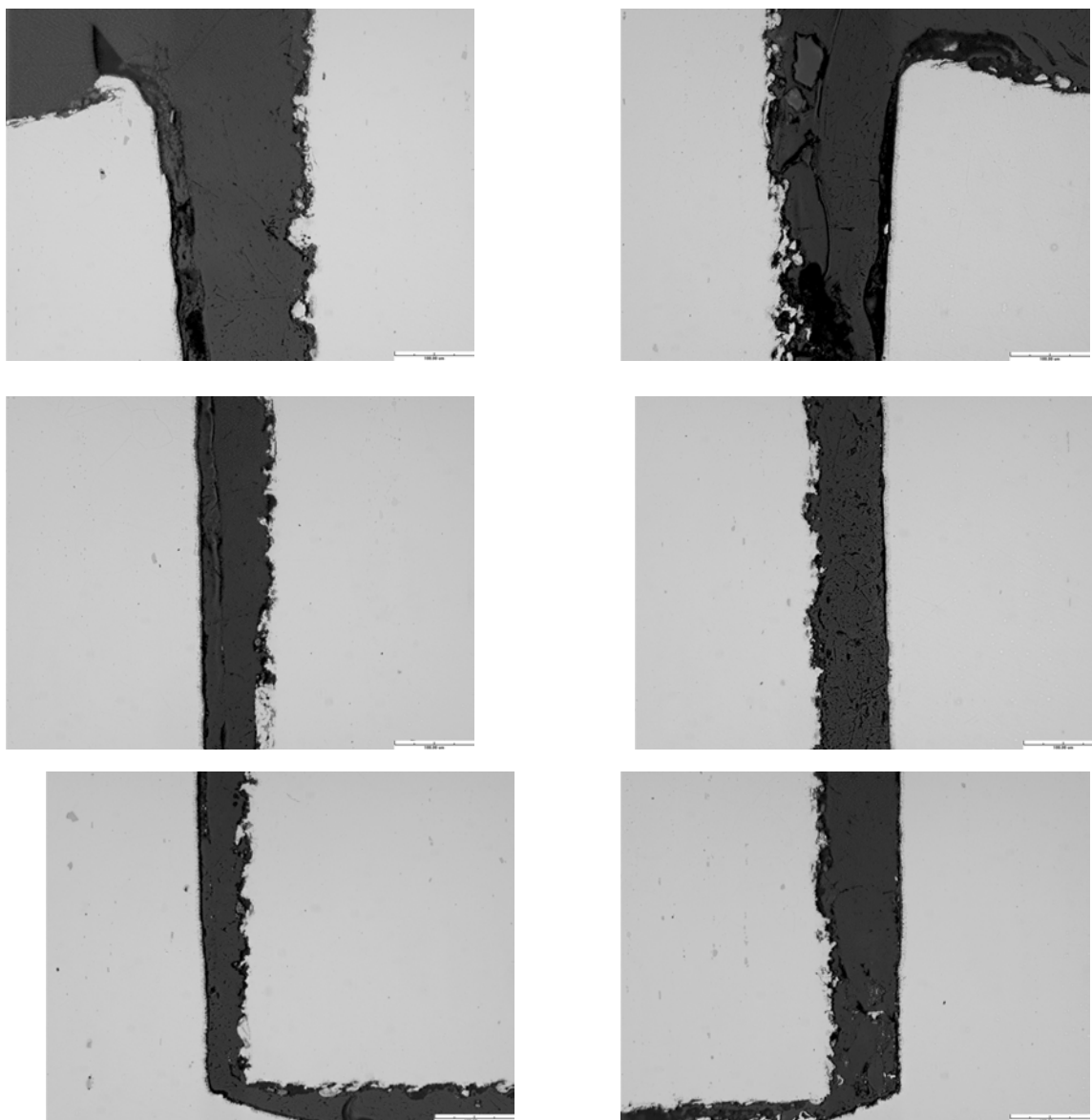


Figure A1 AARB001 – IN617 Pin in a IN617 Collar, 500h at 750°C.

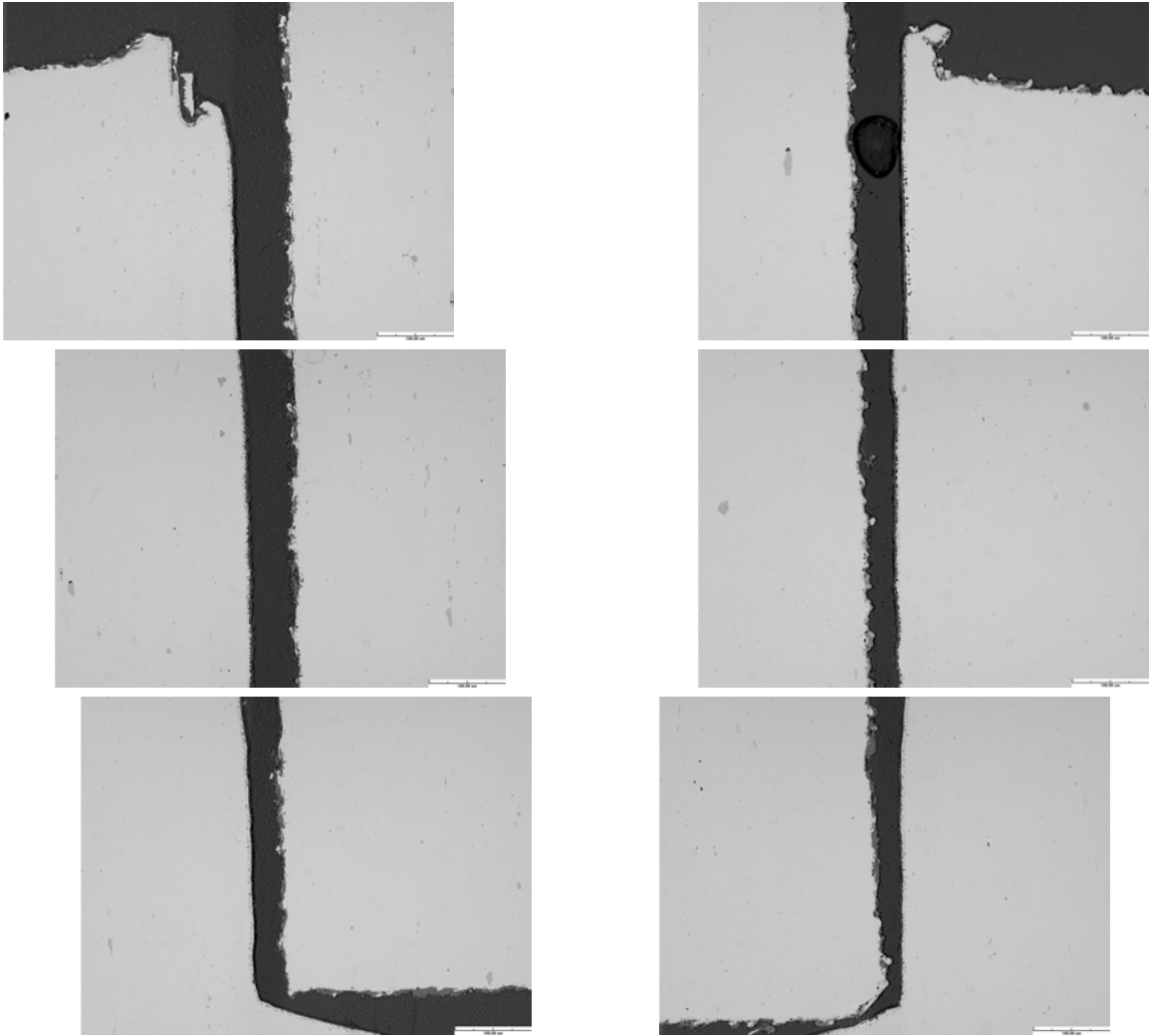


Figure A2 AARB002 – IN617 Pin in a IN617 Collar, 1000h at 750°C.

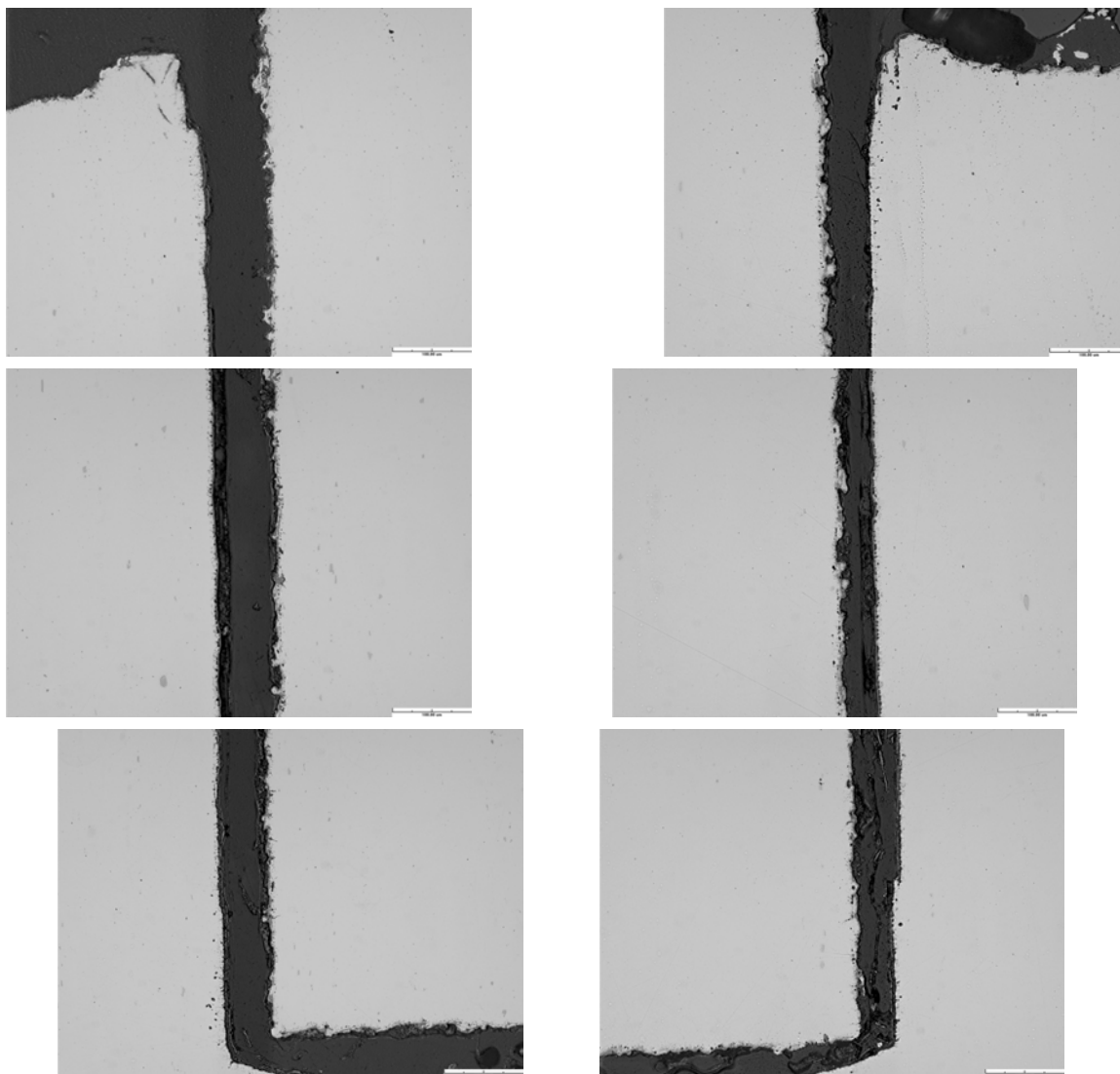


Figure A3 AARB003 – IN617 Pin in a IN617 Collar, 2000h at 750°C

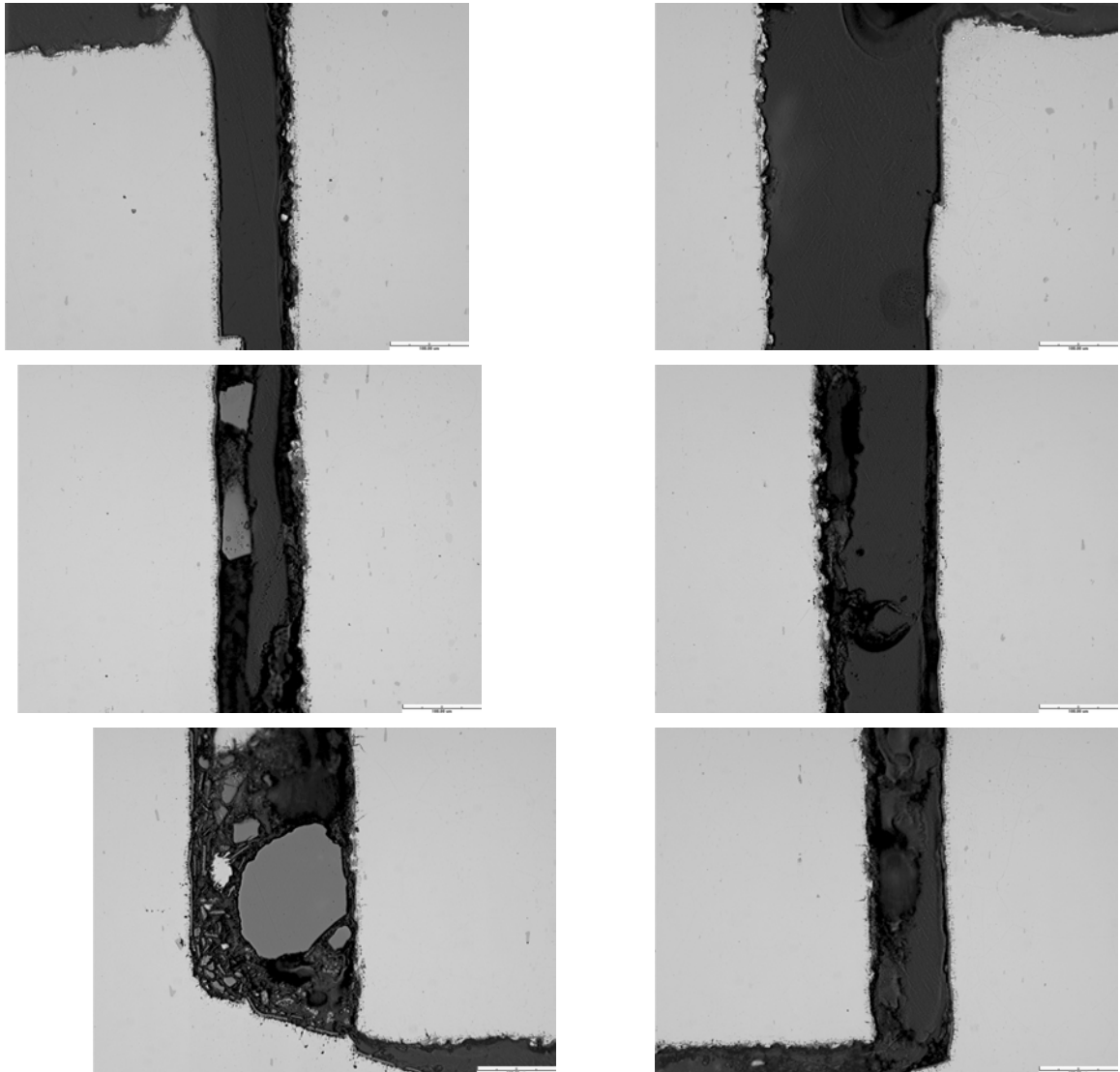


Figure A4 AARB004 – IN617 Pin in an IN617 Collar, 3000h at 750°C

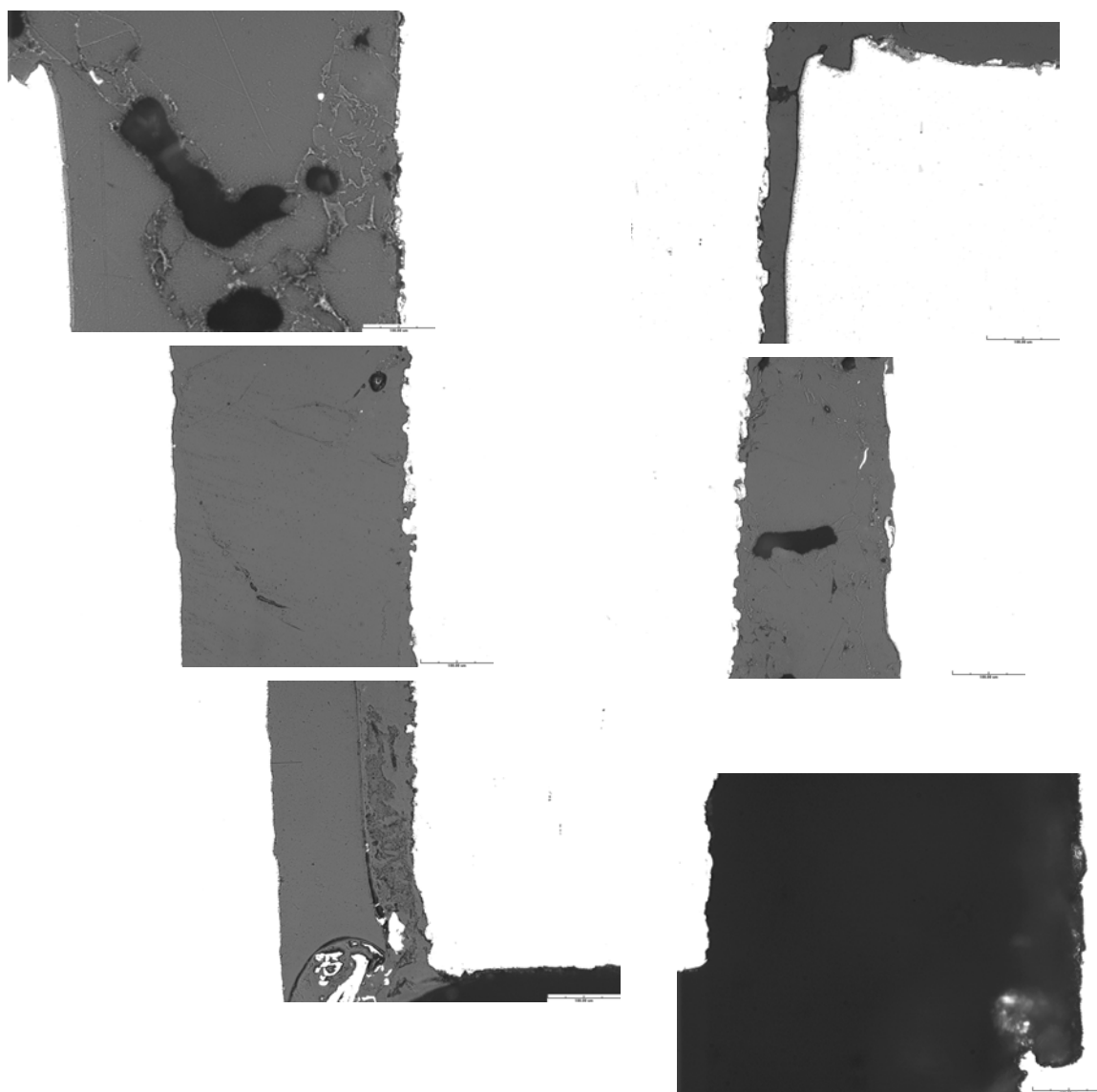


Figure A5 AARC001 – IN740 Pin in an IN617 Collar, 500h at 750°C



Figure A6 AARC002 – IN740 Pin in an IN617 Collar, 1000h at 750°C.

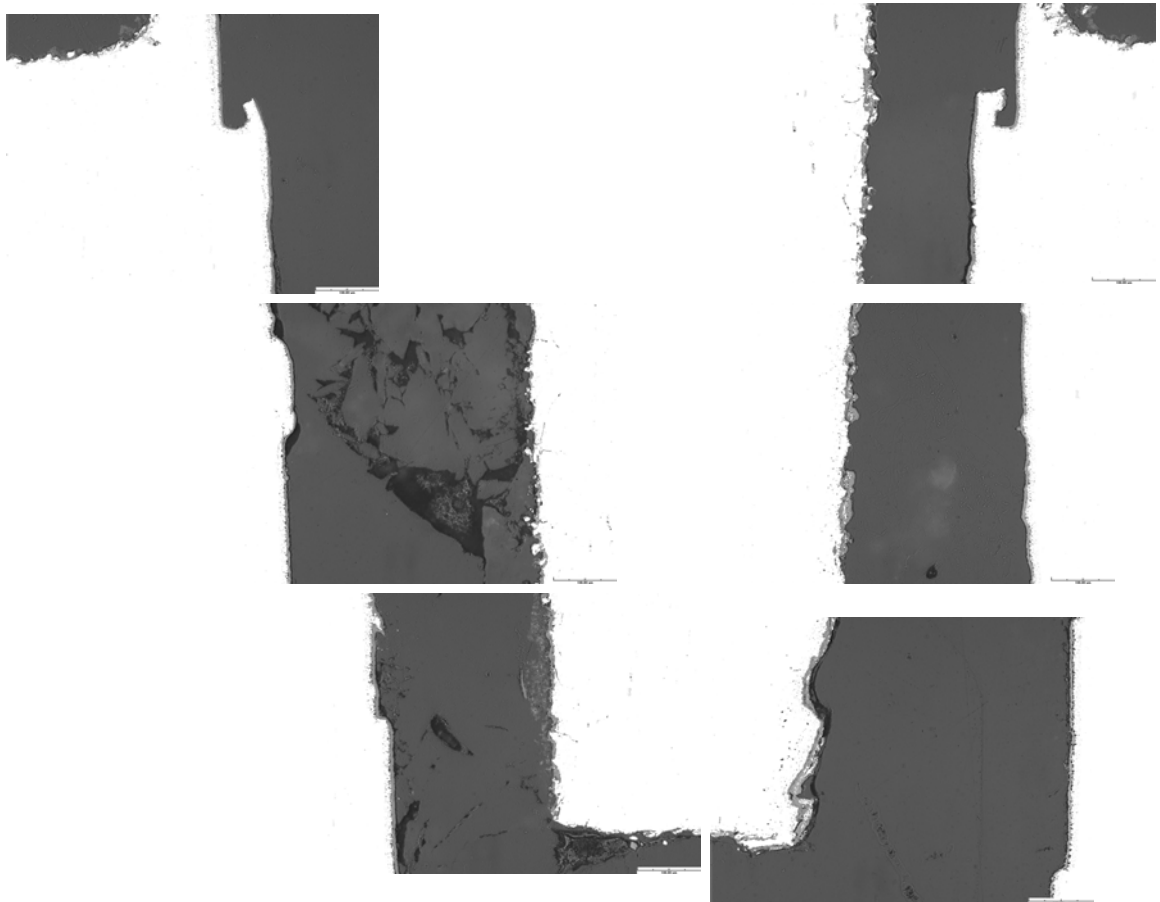


Figure A7 AARC003 – IN740 Pin in an IN617 Collar, 2000h at 750°C.



Figure A8 AARC004 – IN740 Pin in an IN617 Collar, 3000h at 750°C.

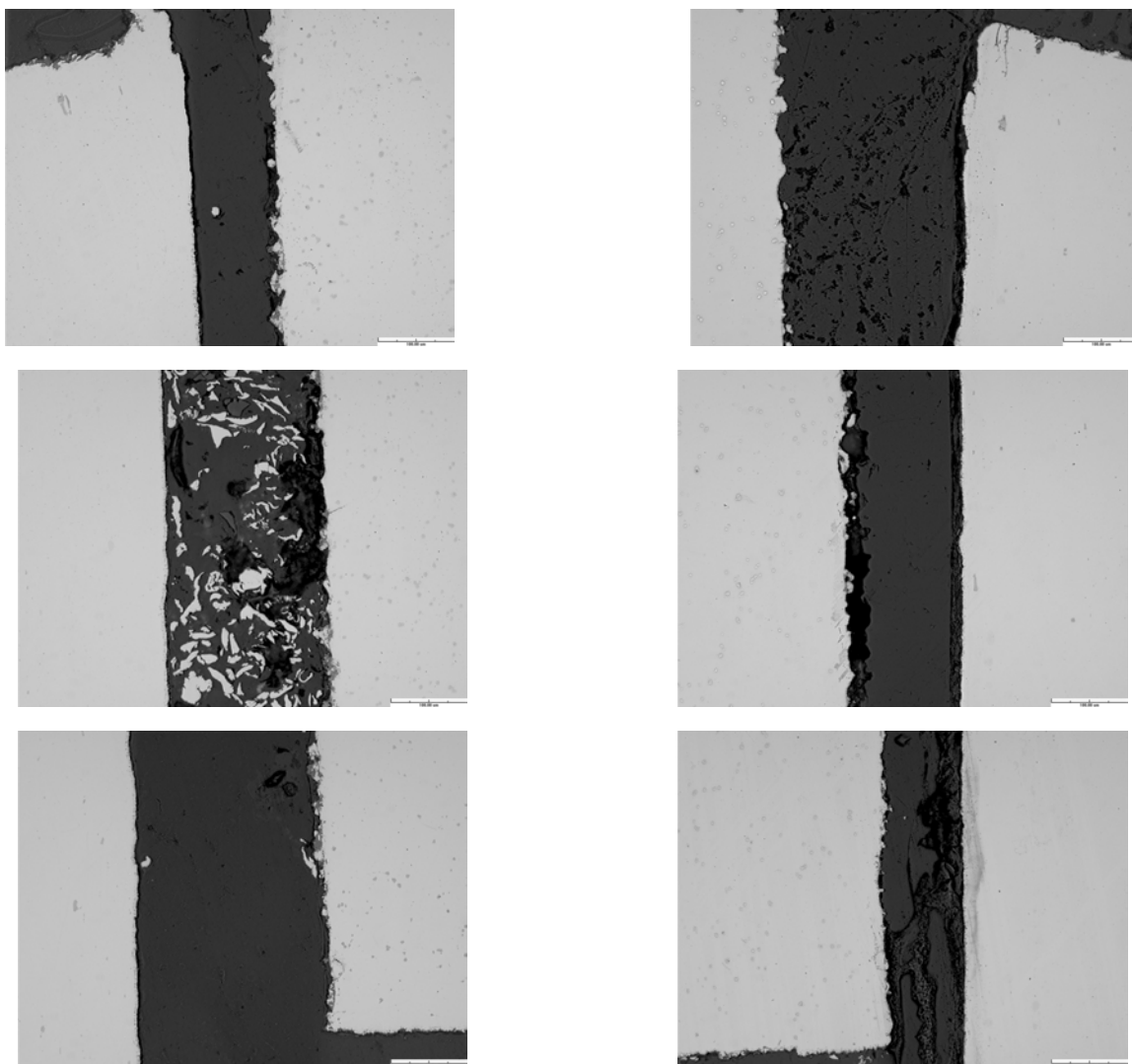


Figure A9 AARD001 – IN939 Pin in an IN617 Collar, 500 h at 750°C.

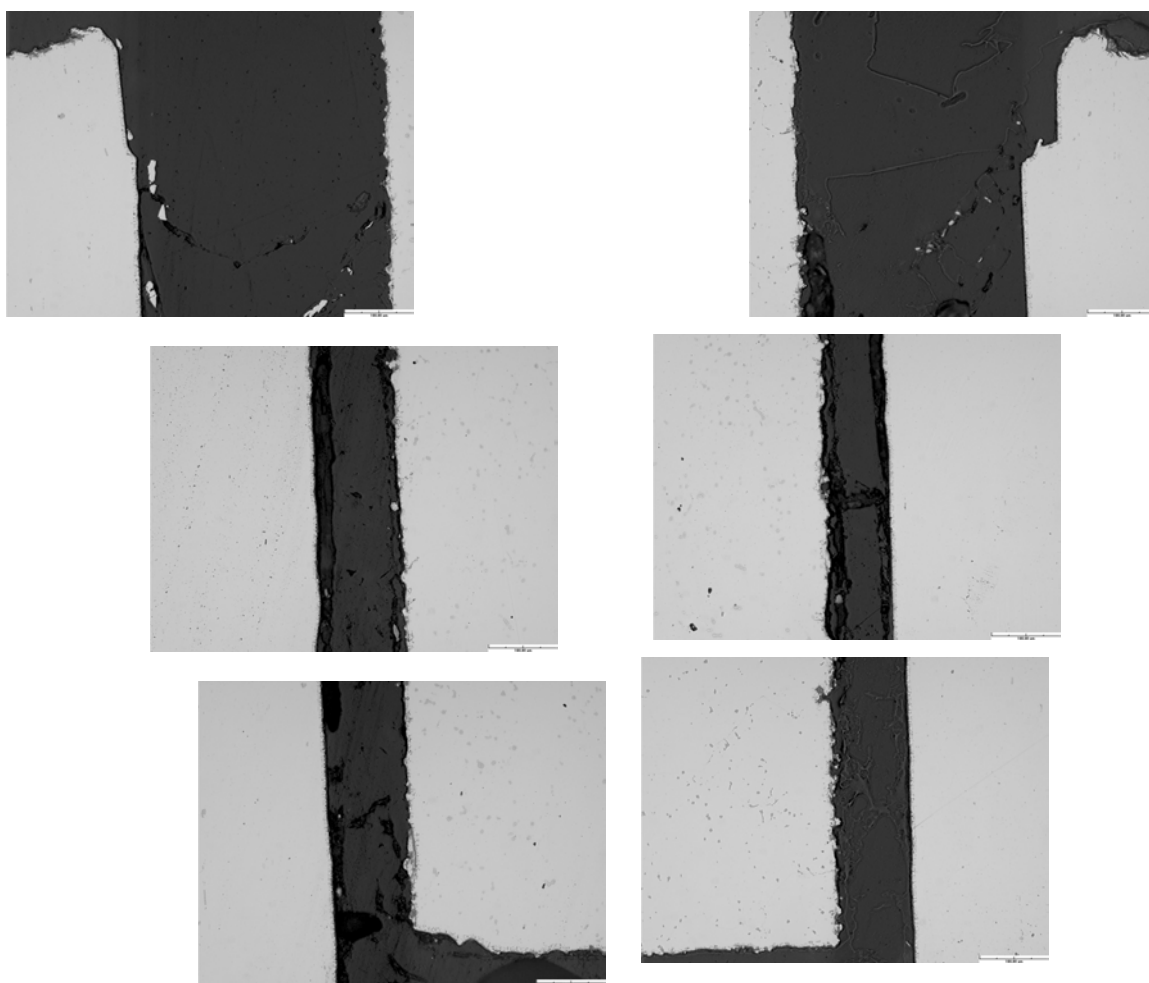


Figure A10 AARD002 – IN939 Pin in an IN617 Collar, 1000h at 750°C.

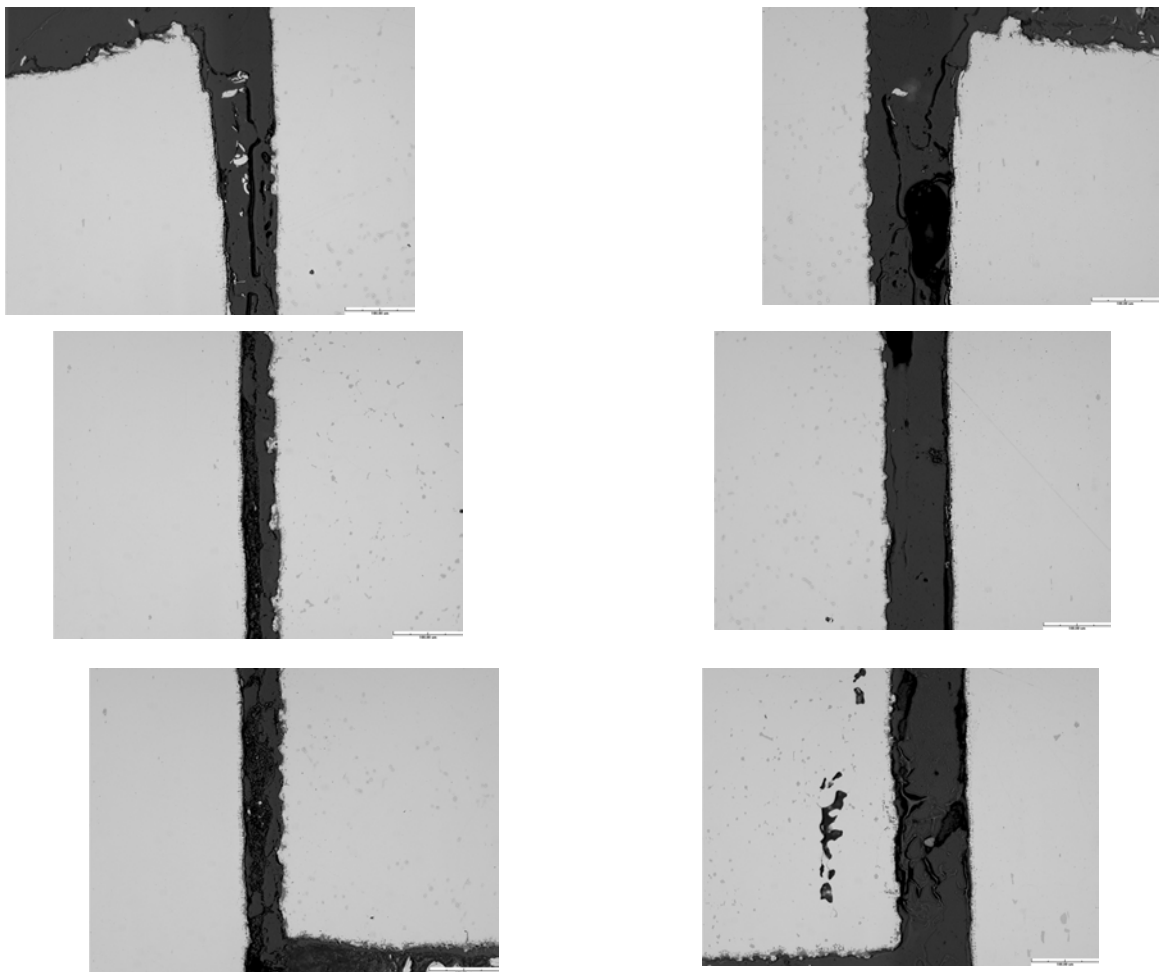


Figure A11 AARD003 – IN939 Pin in an IN617 Collar, 2000 h at 750°C.

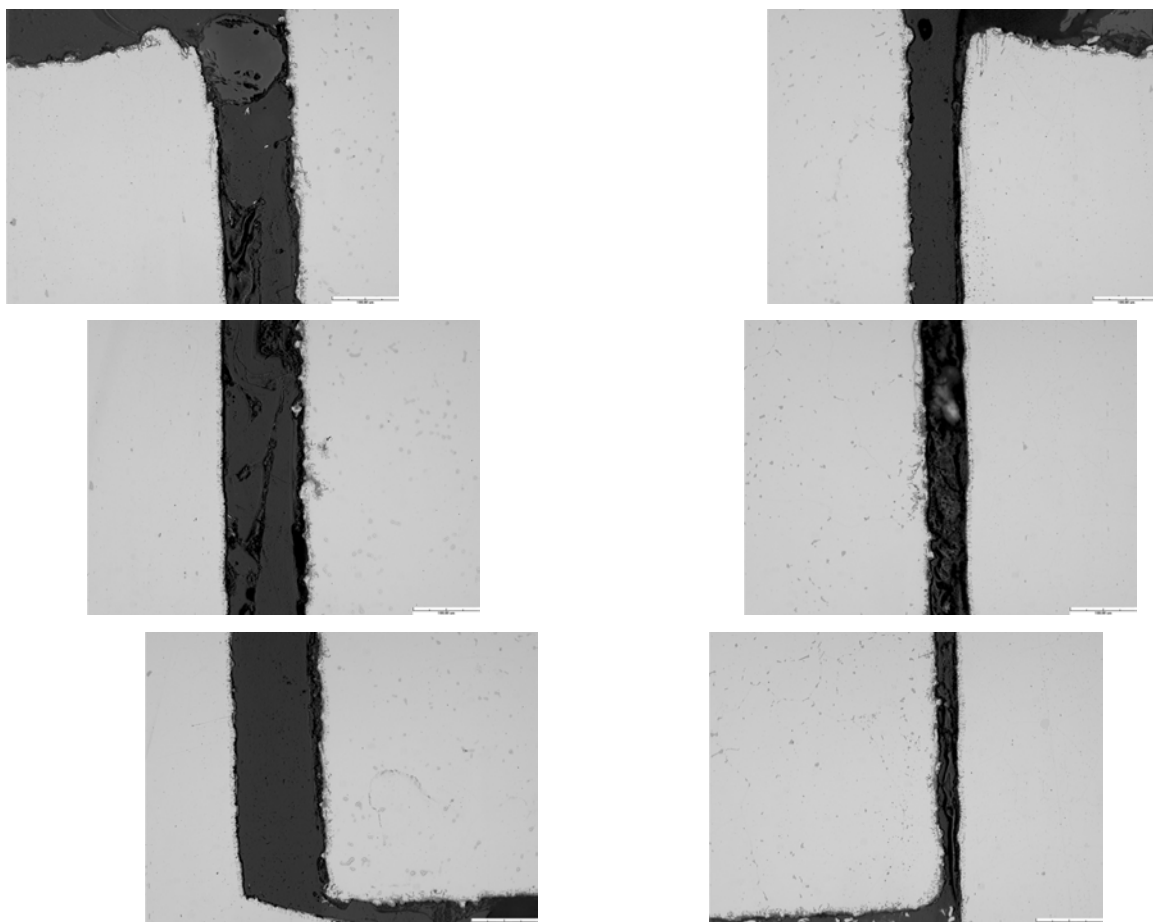


Figure A12 AARD004 – IN939 Pin in an IN617 Collar, 3000 h at 750°C.

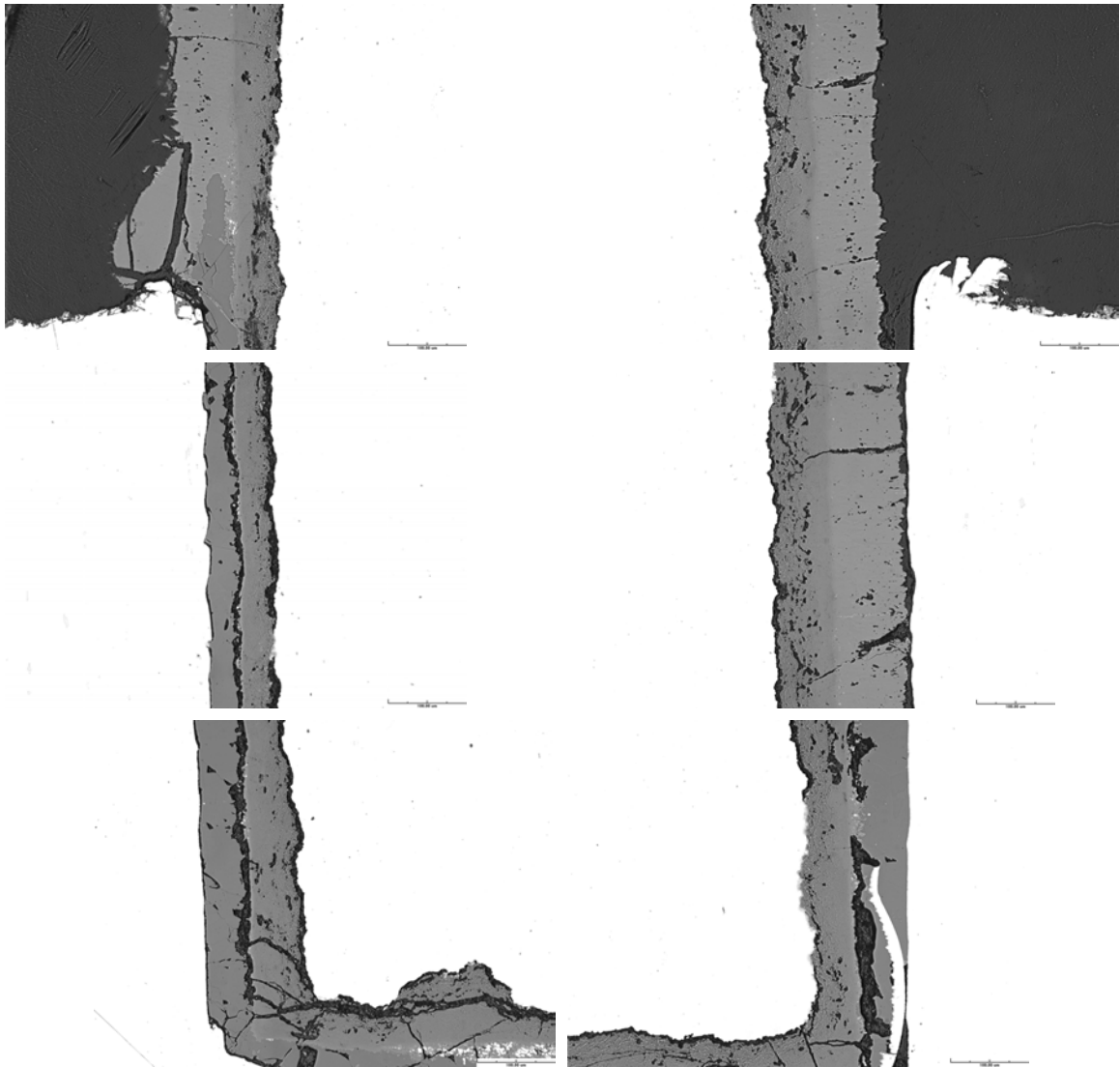


Figure A13 AARE001 – P92 Pin in an IN617 Collar, 500 h at 650°C.



Figure A14 AARE002 – P92 Pin in an IN617 Collar, 1000h at 650°C.

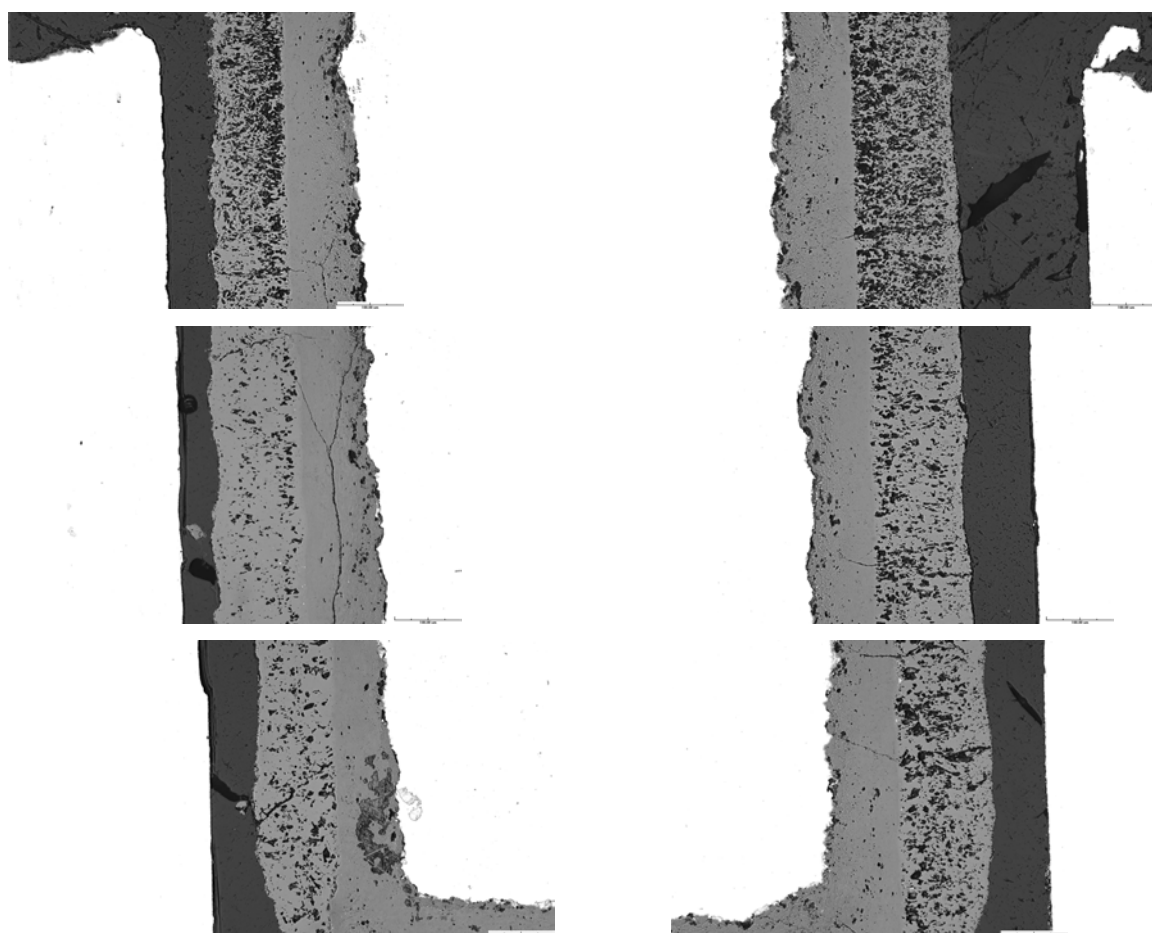


Figure A15 AARE003 – P92 Pin in an IN617 Collar, 2000 h at 650°C.

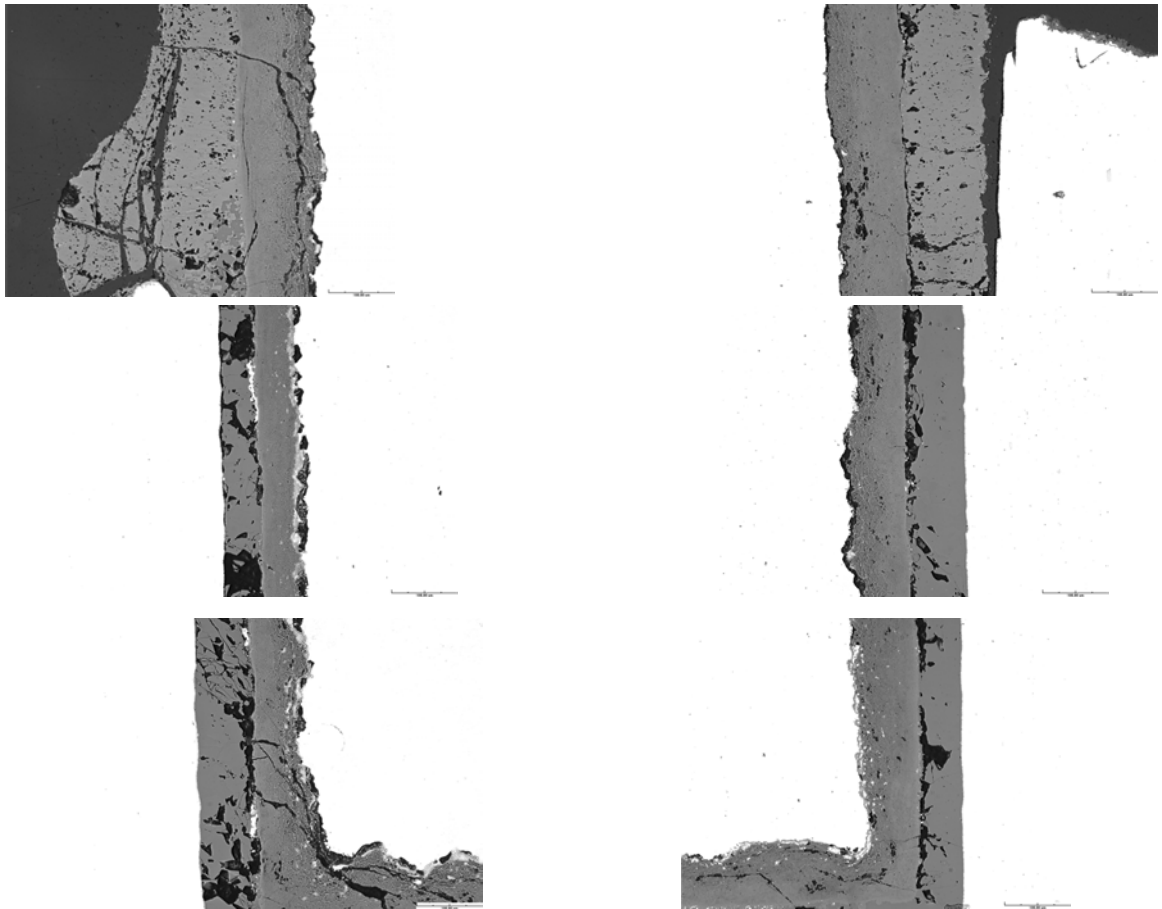


Figure A16 AARE004 – P92 Pin in an IN617 Collar, 3000h at 650°C.

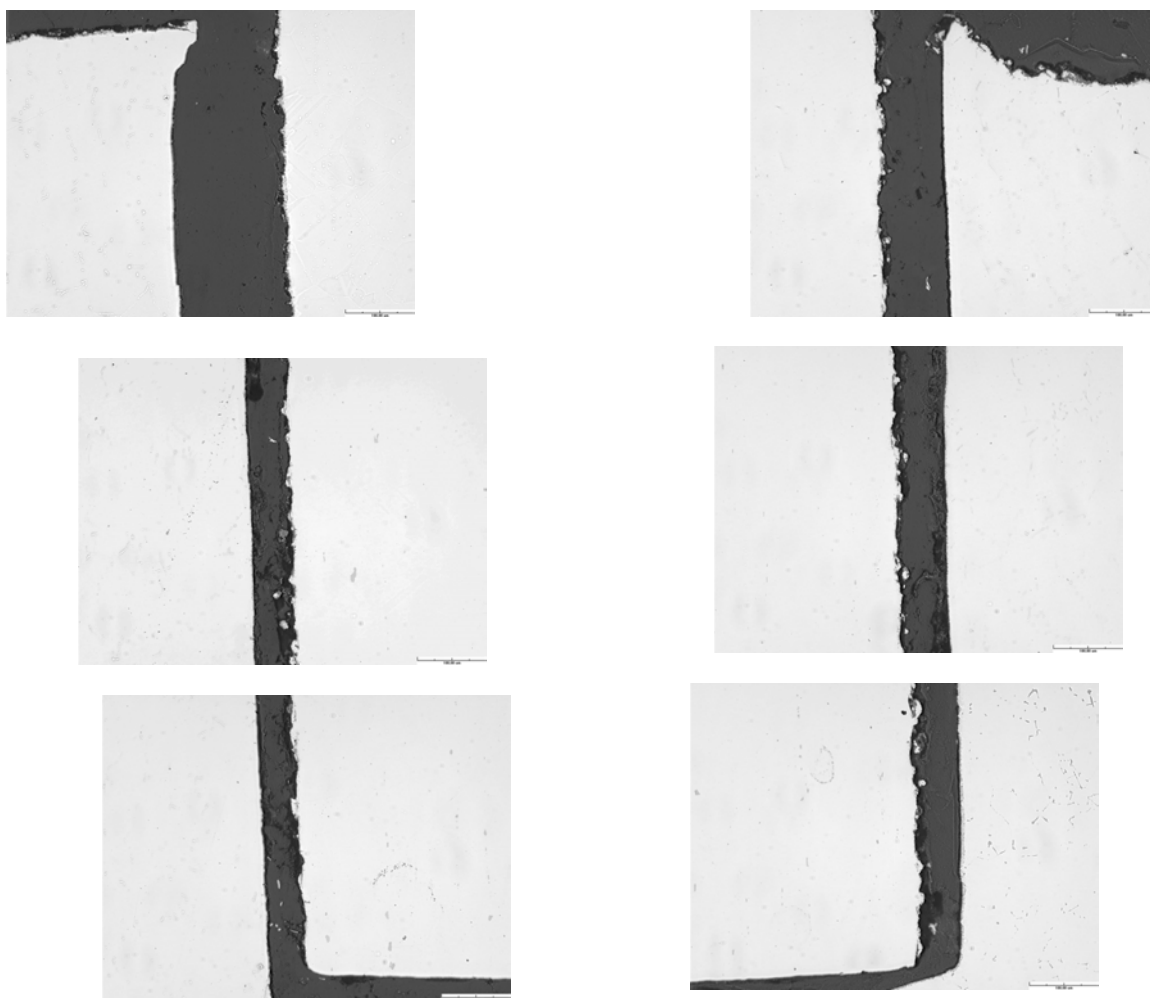


Figure A17 AARF001 – IN617 Pin in an IN738 Collar, 500h at 750°C.

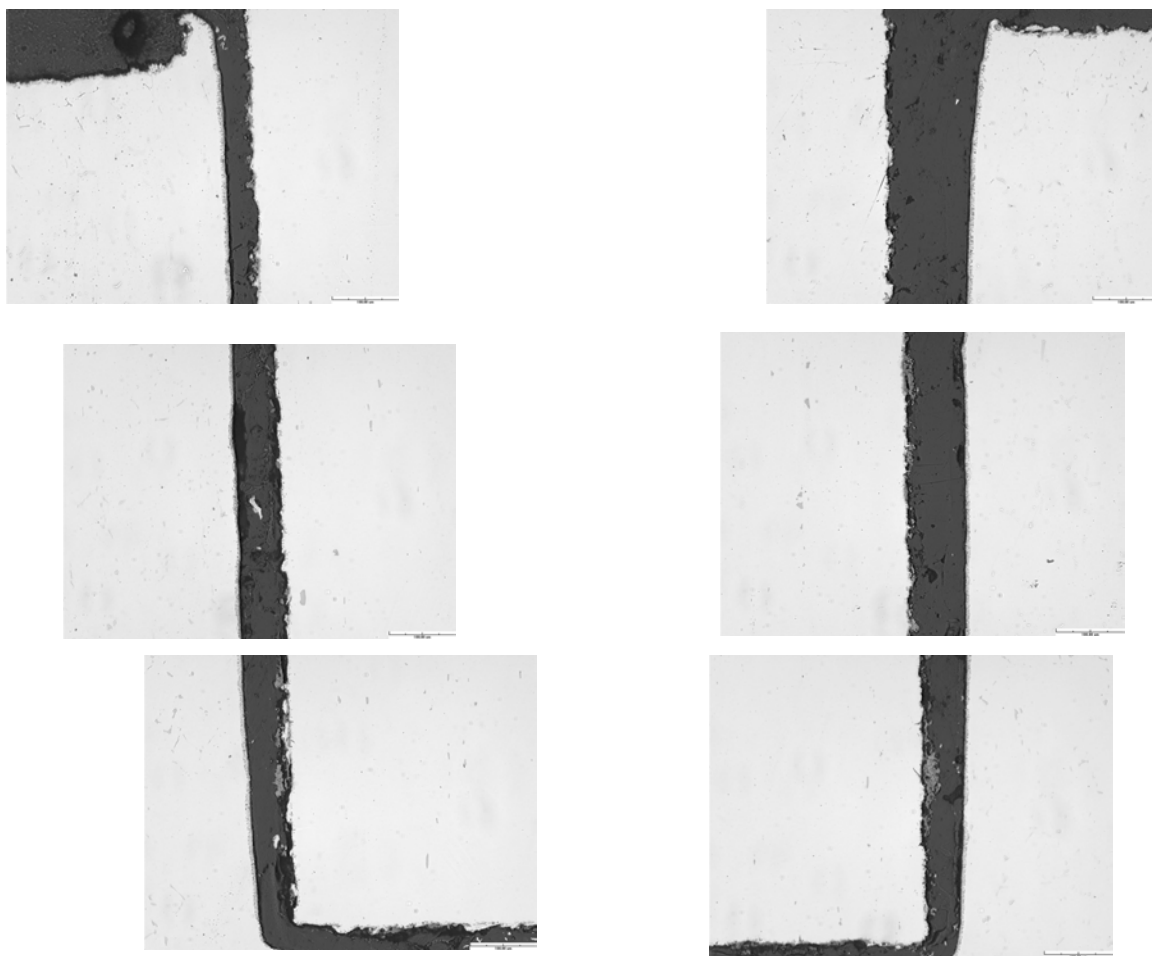


Figure A18 AARF002 – IN617 Pin in an IN738 Collar, 1000h at 750°C.

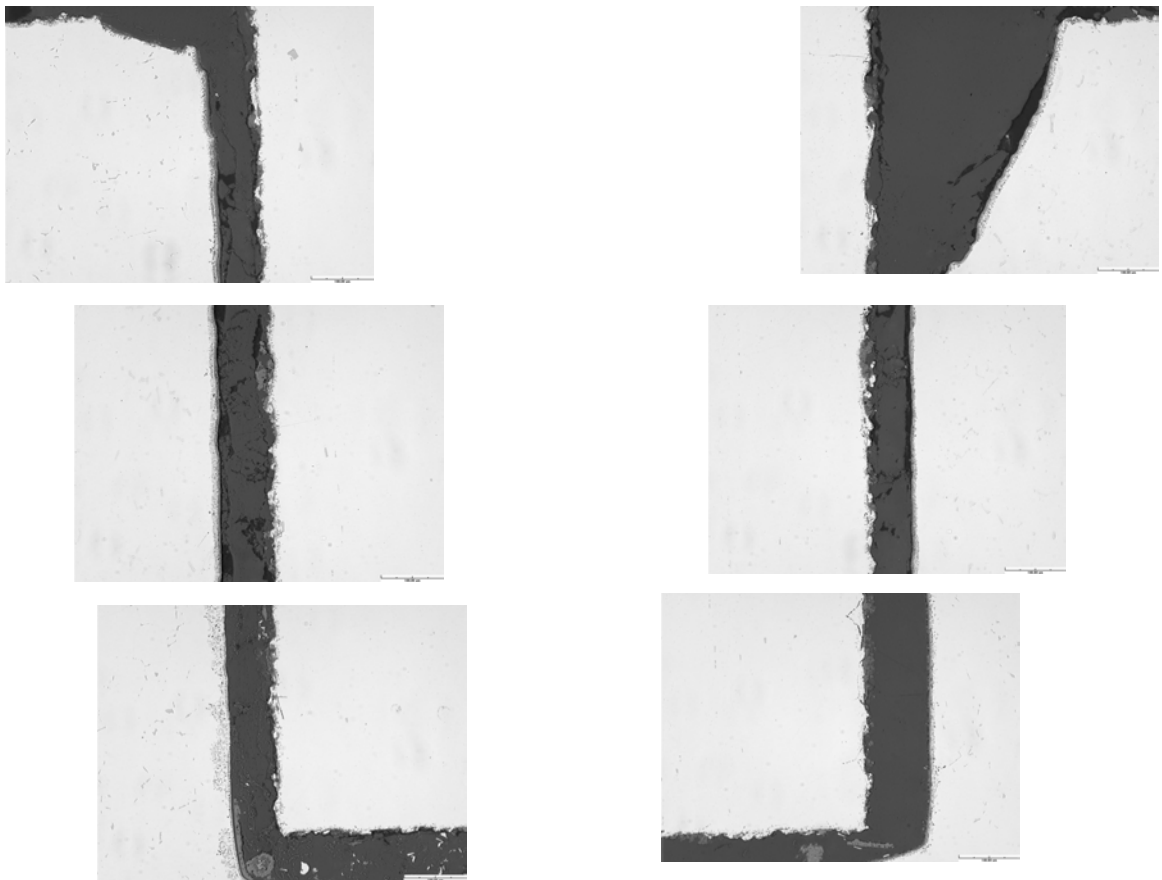


Figure A19 AARF003 – IN617 Pin in an IN738 Collar, 2000h at 750°C.

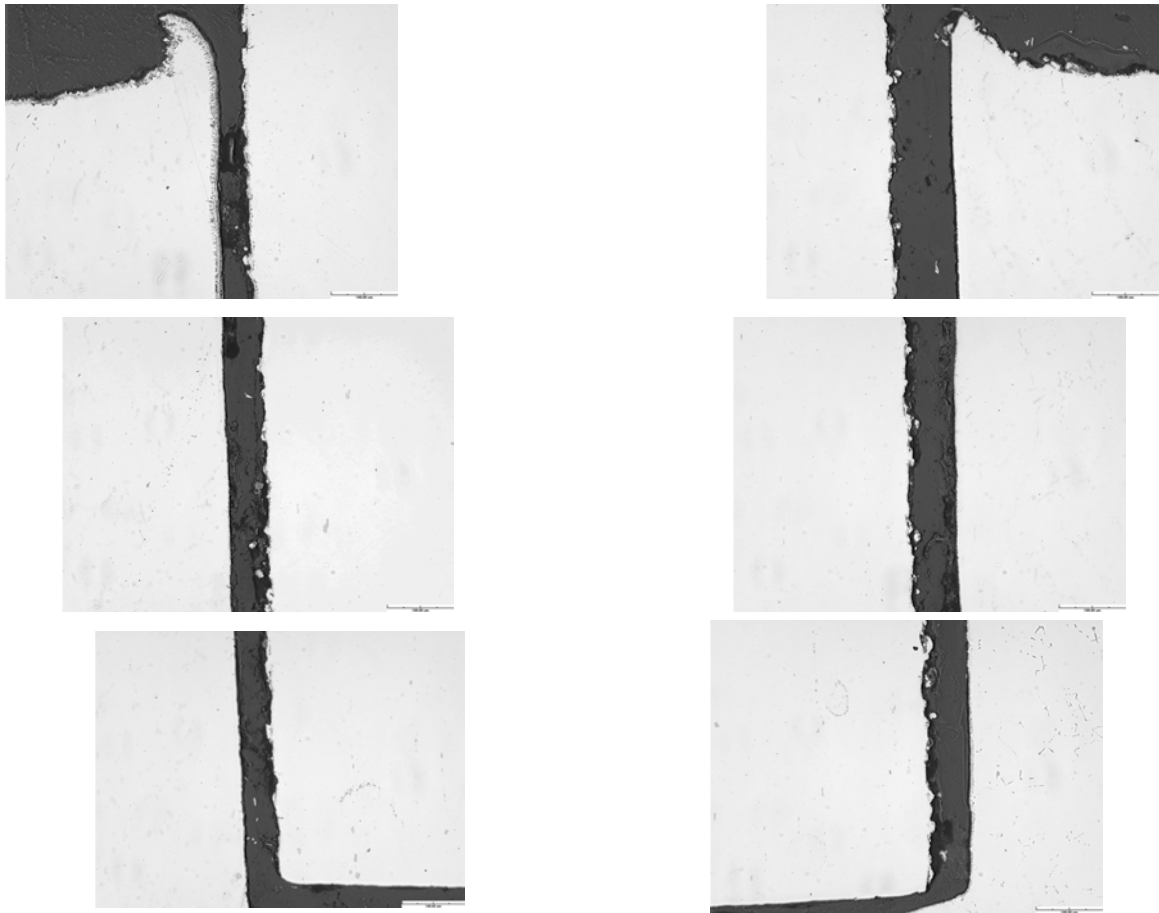


Figure A20 AARF004 – IN617 Pin in an IN738 Collar, 3000h at 750°C.

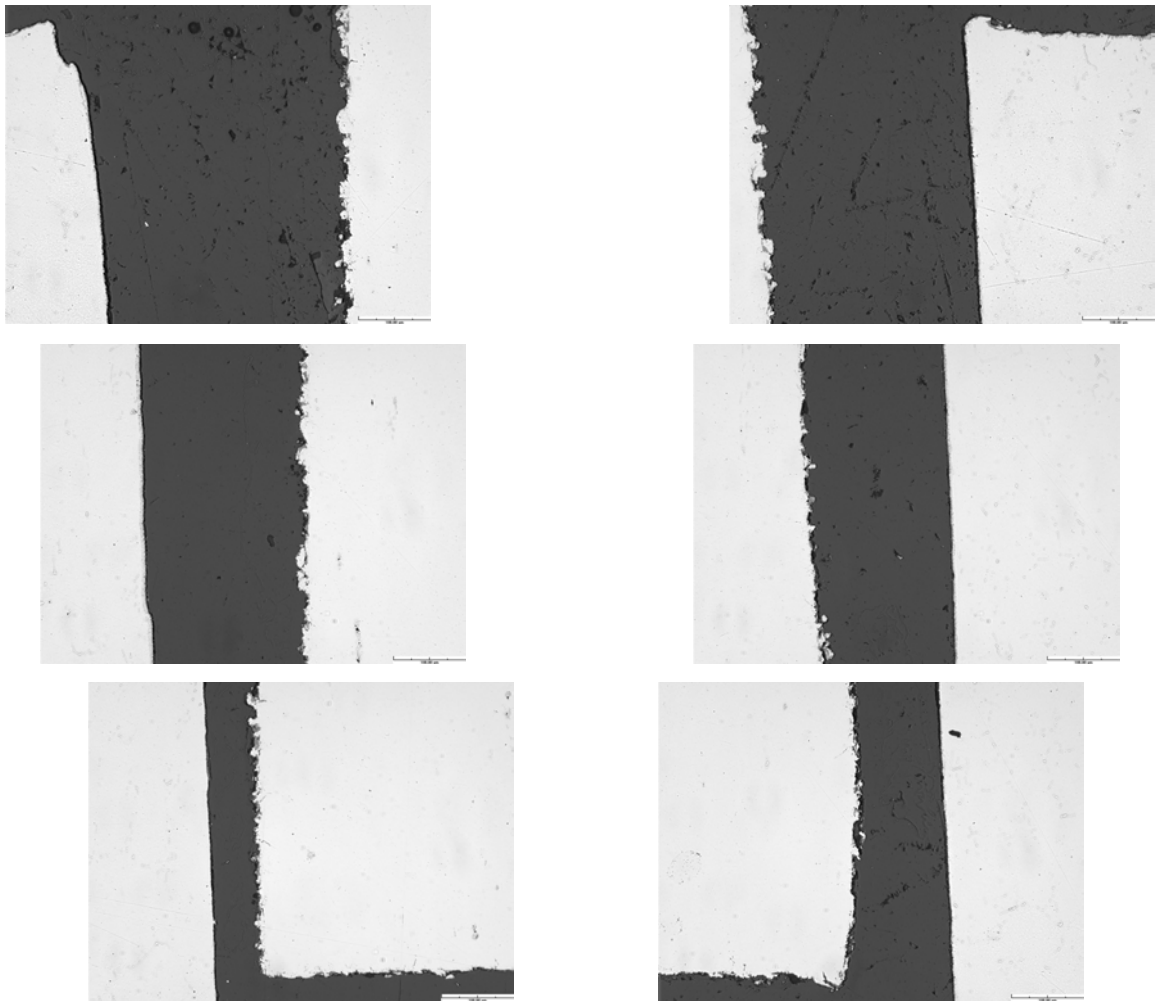


Figure A21 AARG001 – IN740 Pin in an IN738 Collar, 500h at 750°C.

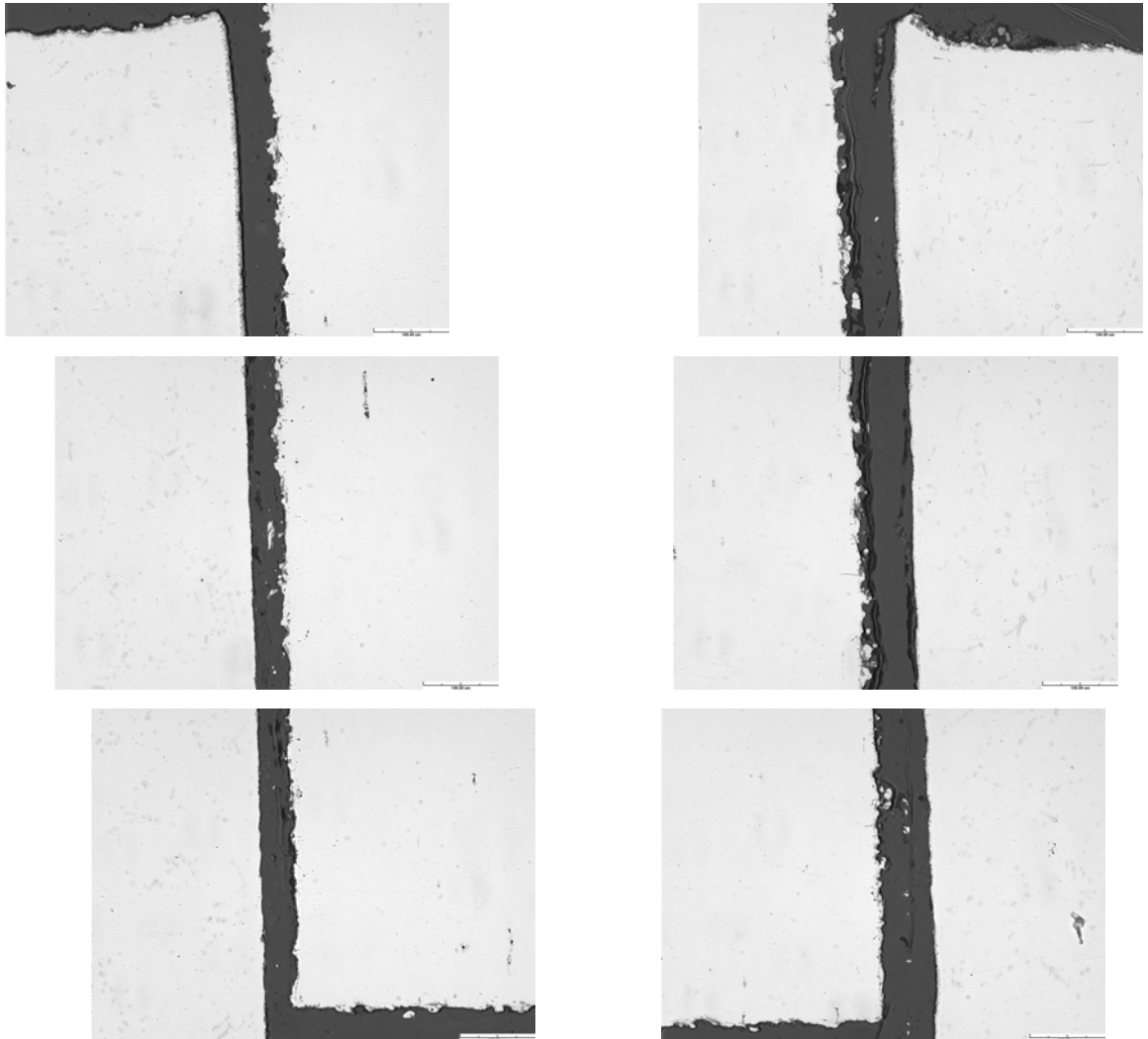


Figure A22 AARG002 – IN740 Pin in an IN738 Collar, 1000h at 750°C.

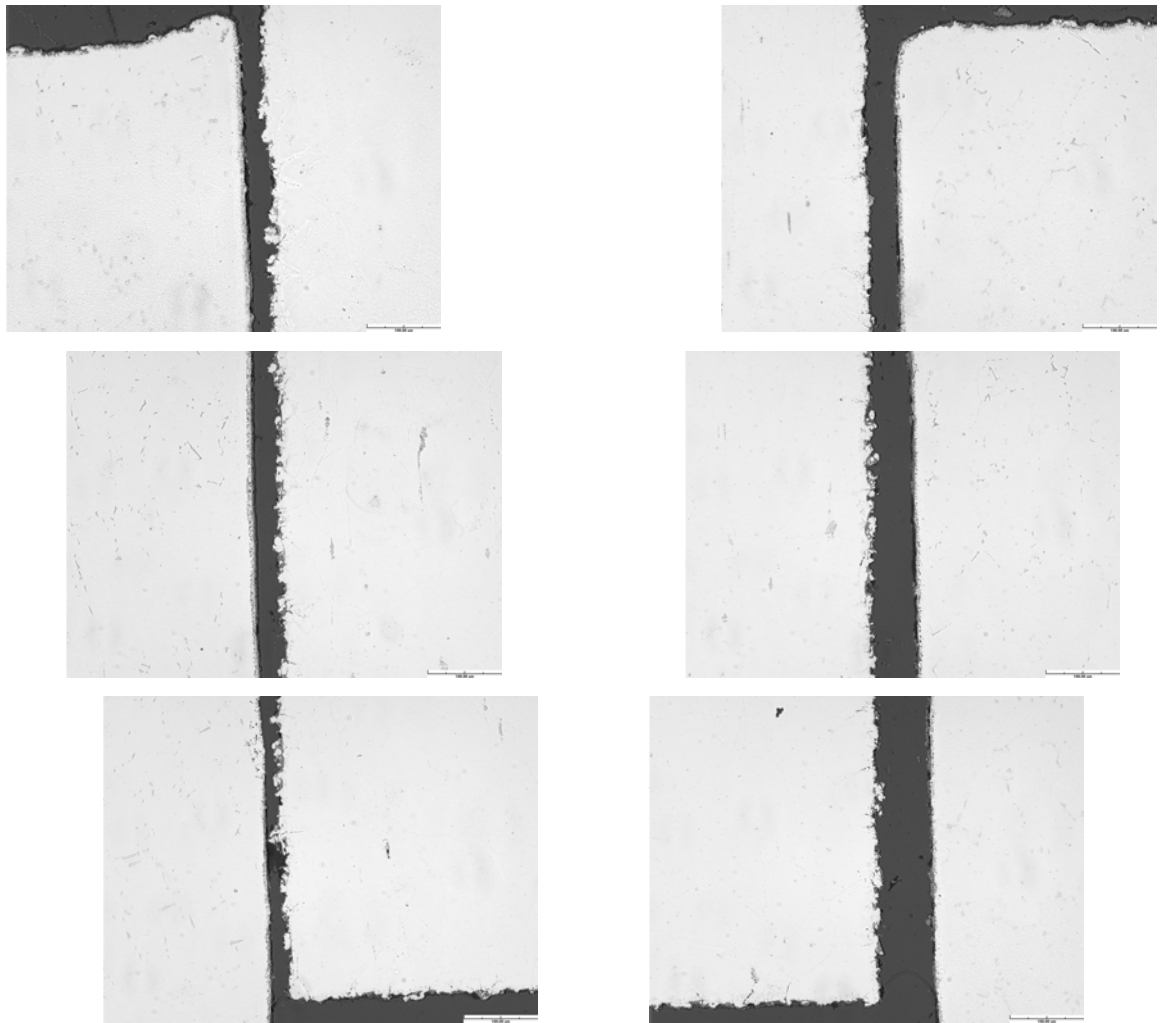


Figure A23 AARG003 – IN740 Pin in an IN738 Collar, 2000h at 750°C.

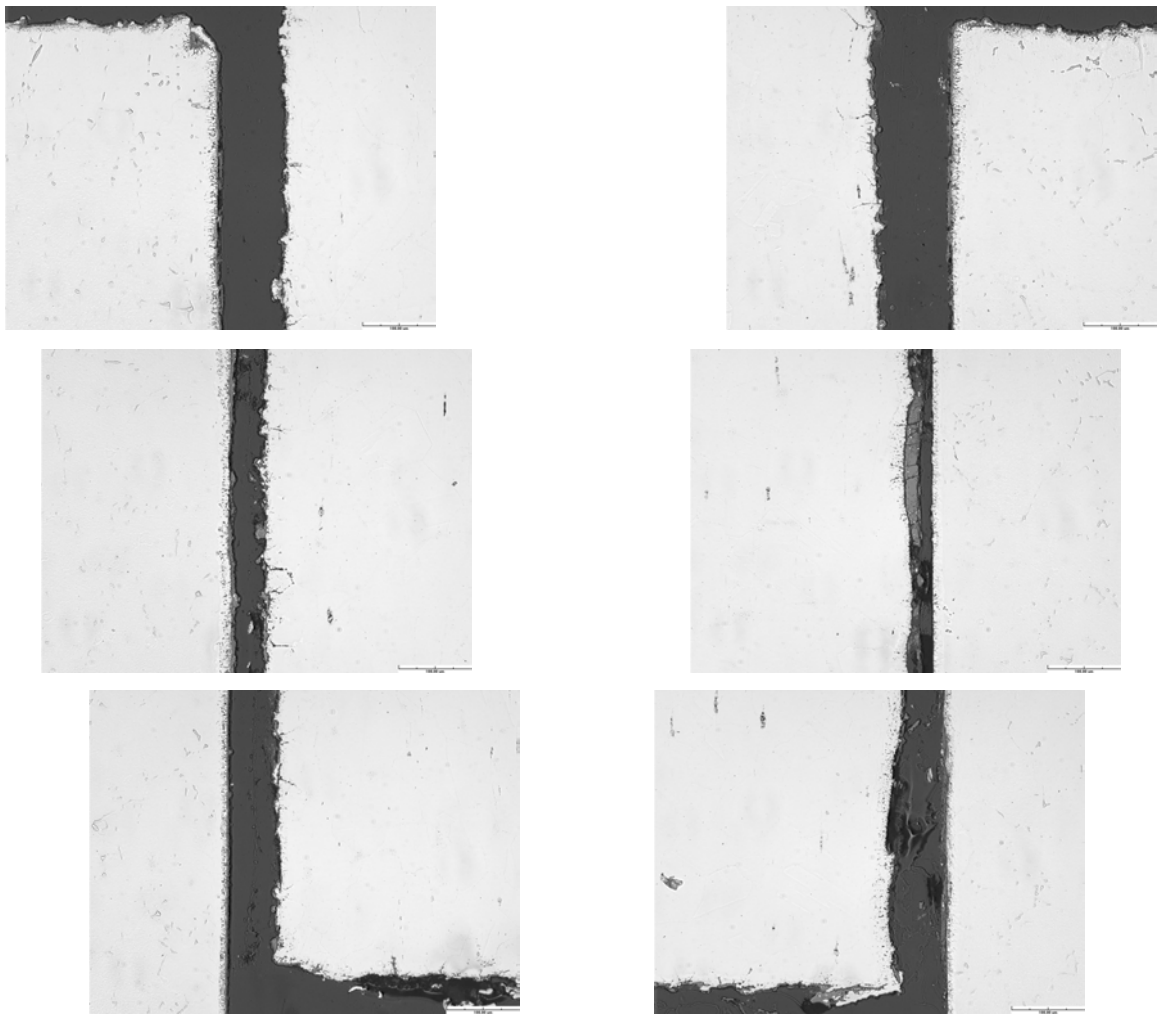


Figure A24 AARG004 – IN740 Pin in an IN738 Collar, 3000h at 750°C.

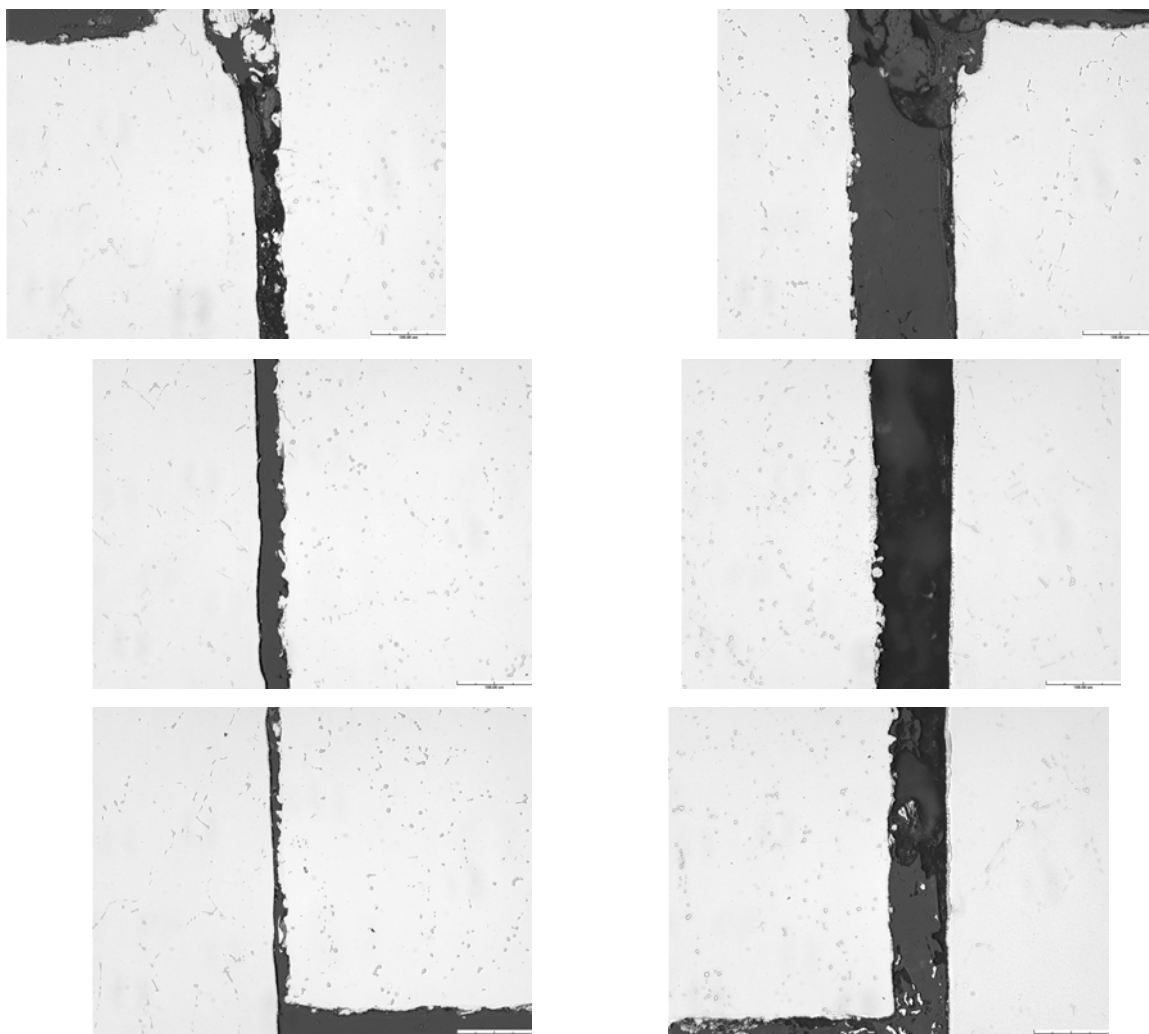


Figure A25 AARH001 – IN939 Pin in an IN738 Collar, 500h at 700°C.

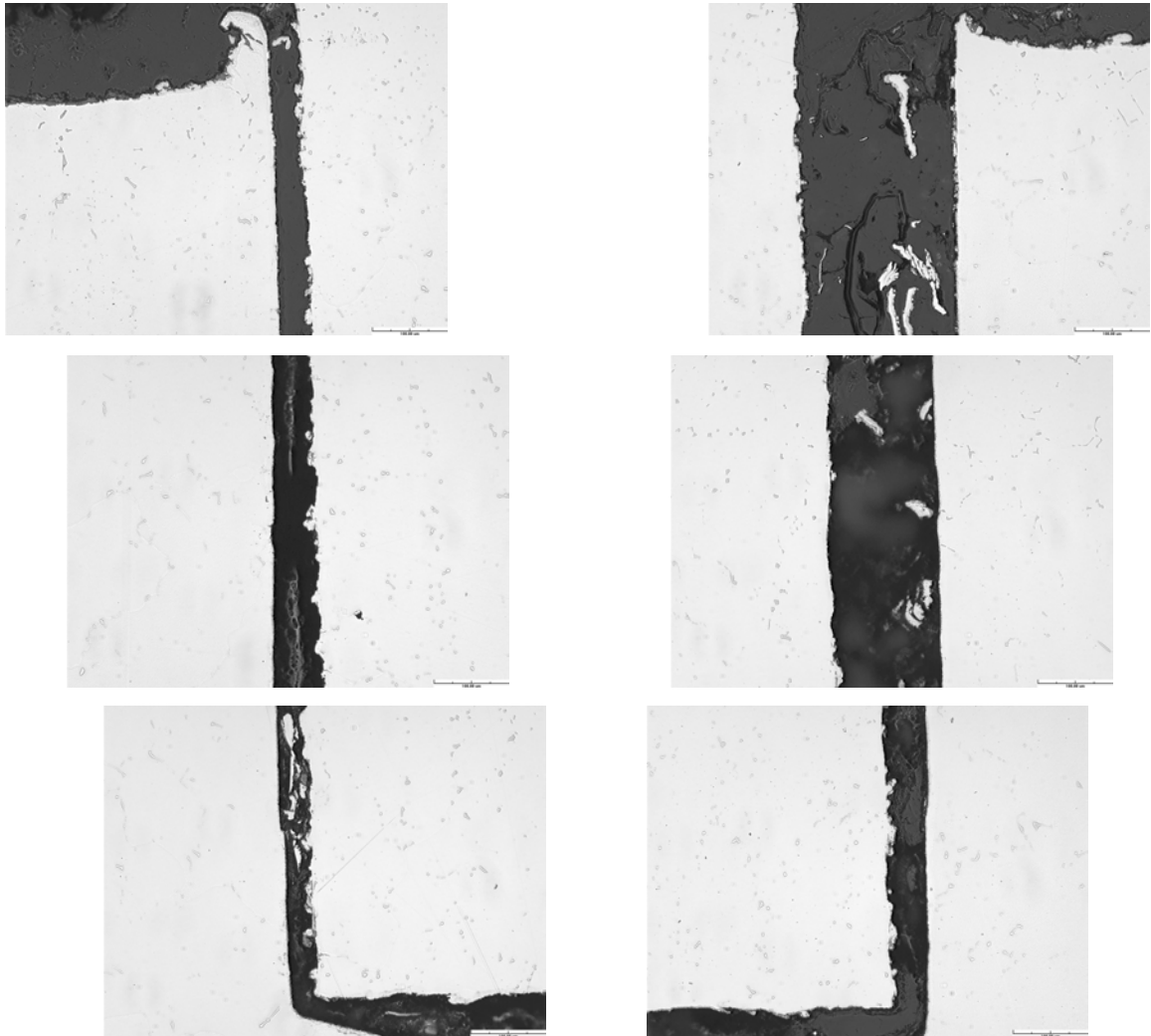


Figure A26 AARH002 – IN939 Pin in an IN738 Collar, 1000h at 700°C.

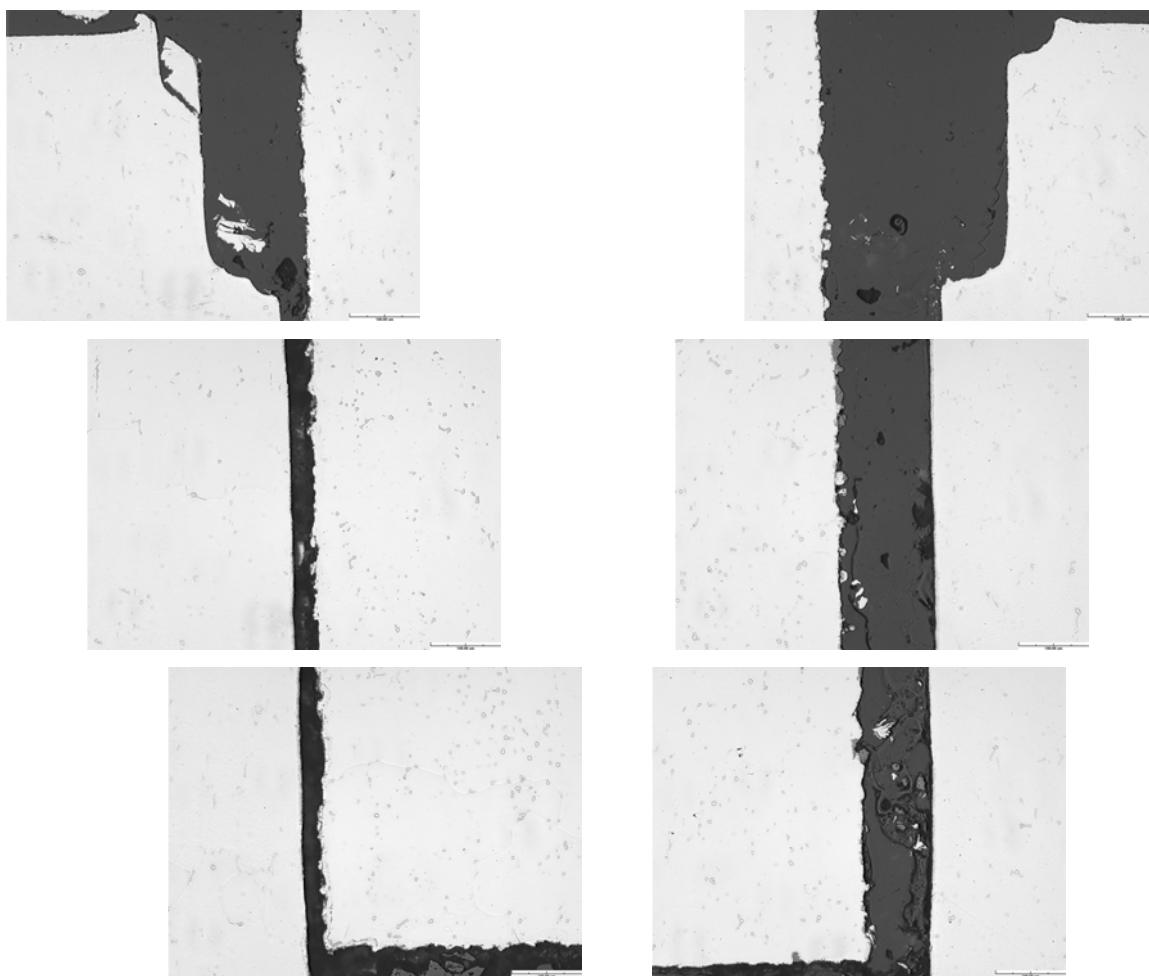


Figure A27 AARH003 – IN939 Pin in an IN738 Collar, 2000h at 700°C.

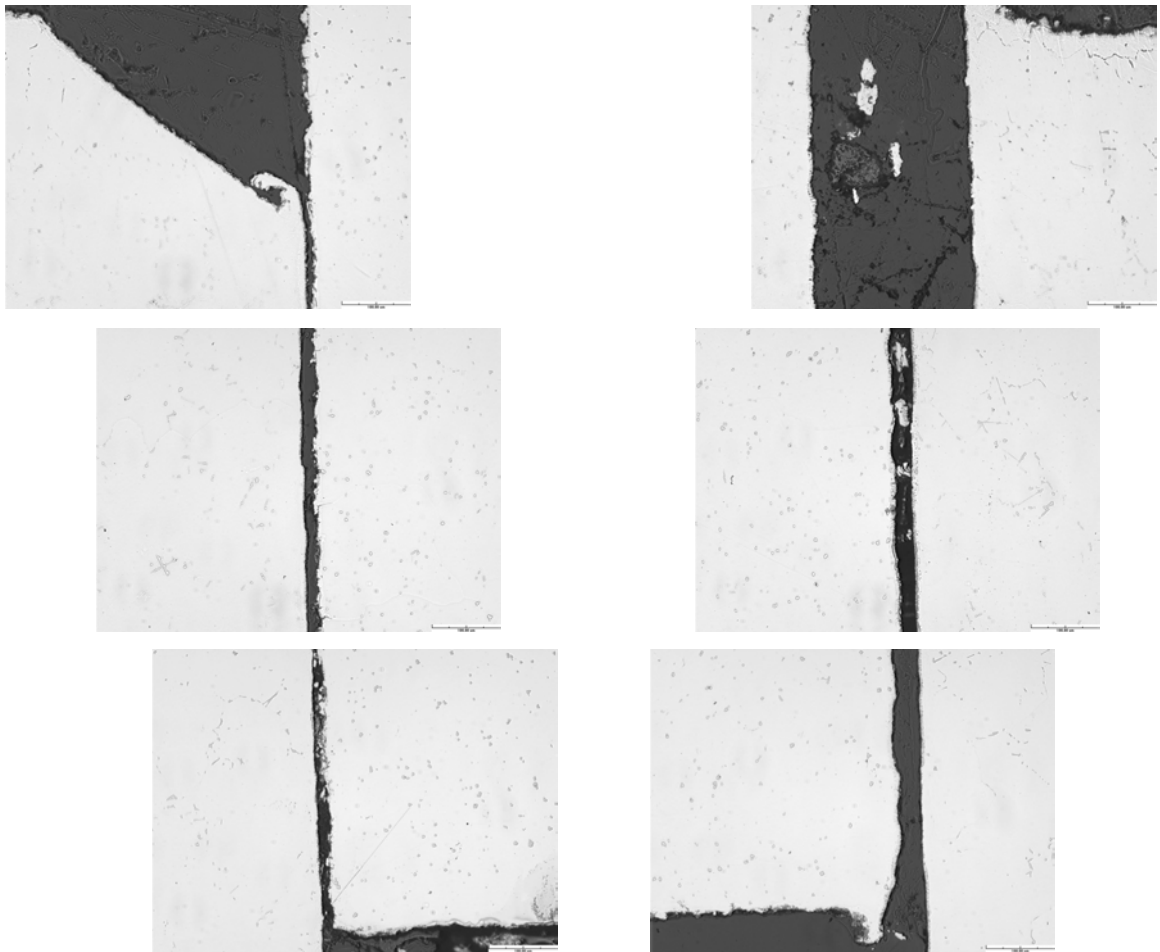


Figure A28 AARH008 – IN939 Pin in an IN738 Collar, 3000h at 700°C.

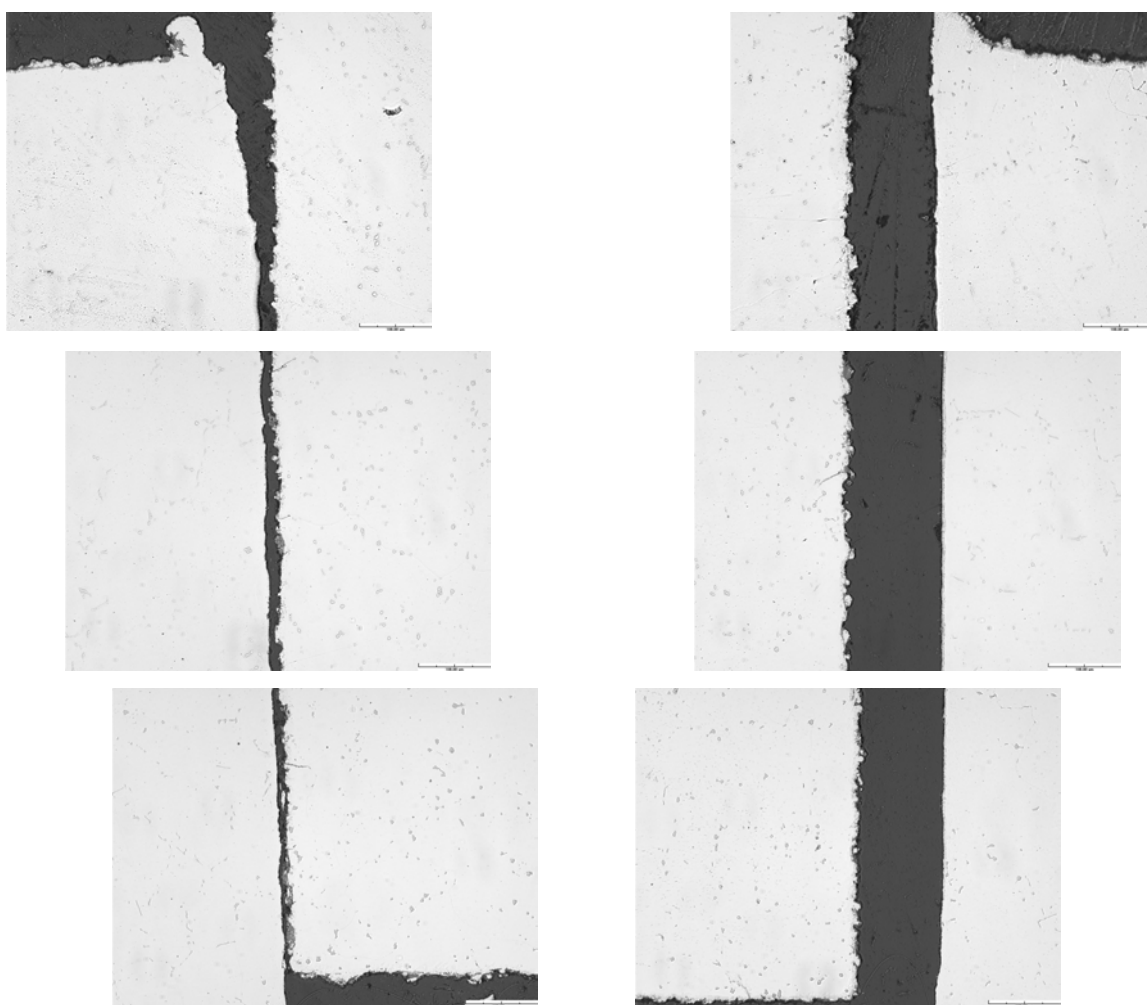


Figure A29 AARH007 – IN939 Pin in an IN738 Collar, 500h at 750°C.

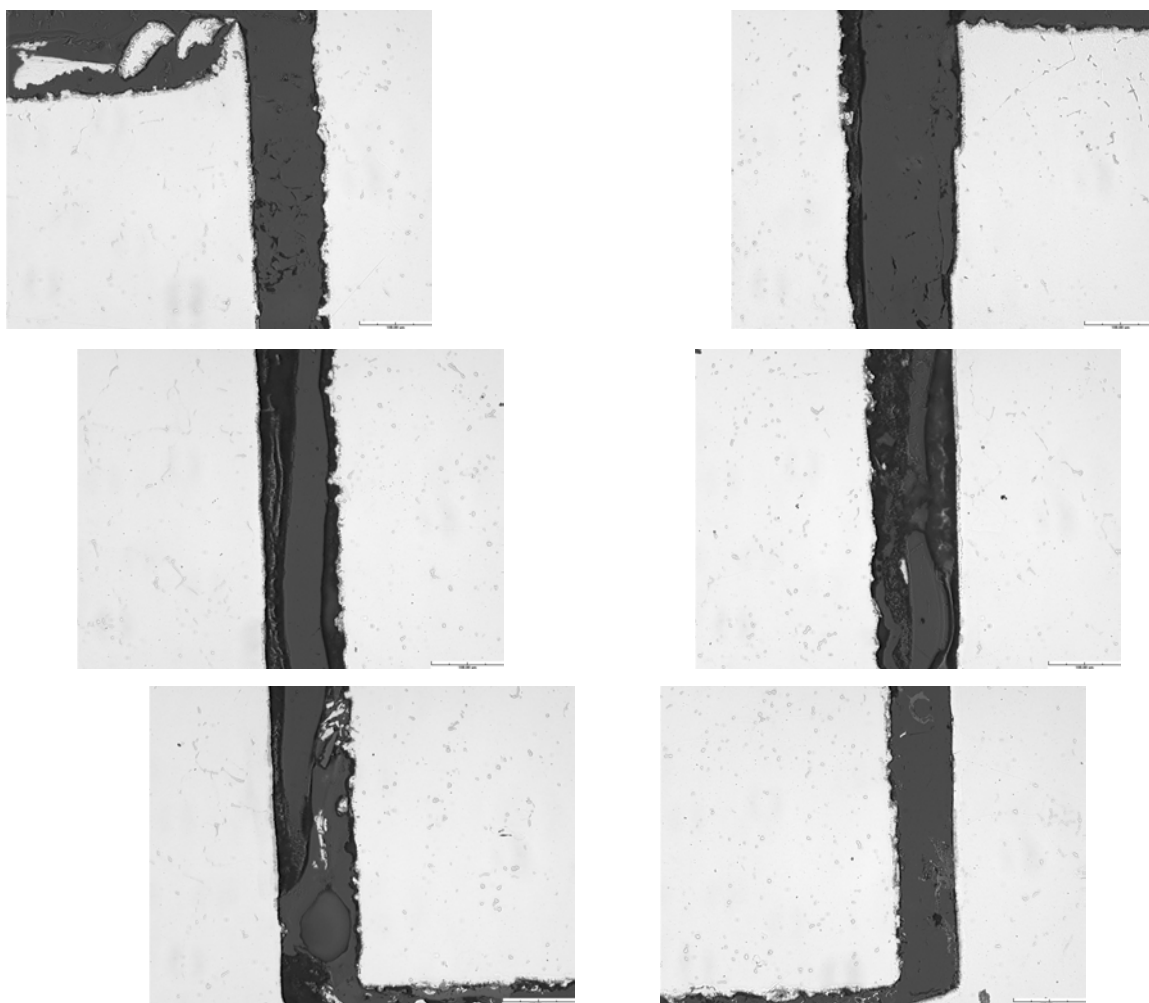


Figure A30 AARH005 – IN939 Pin in an IN738 Collar, 1000h at 750°C.

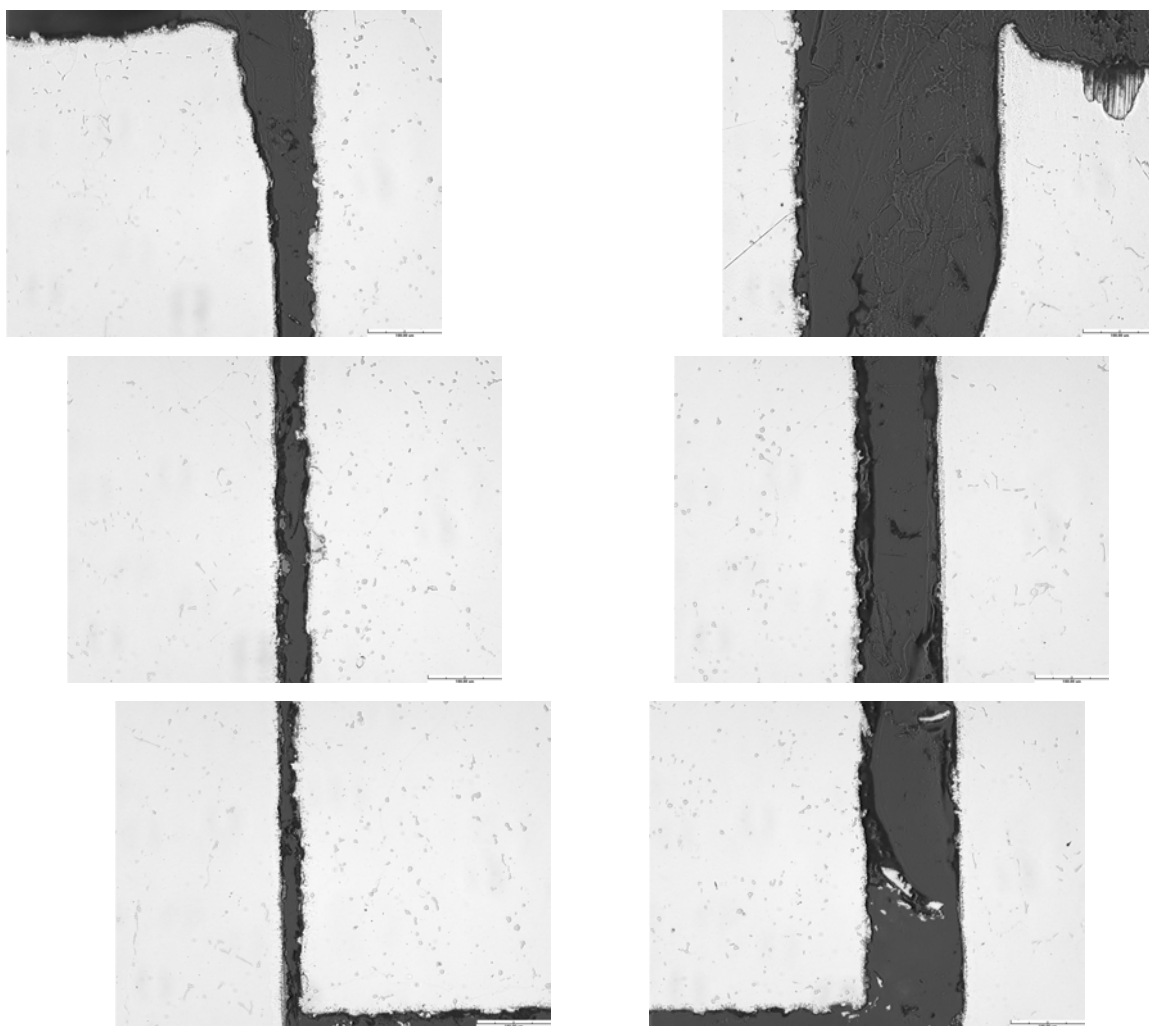


Figure A31 AARH006 – IN939 Pin in an IN738 Collar, 2000h at 750°C.

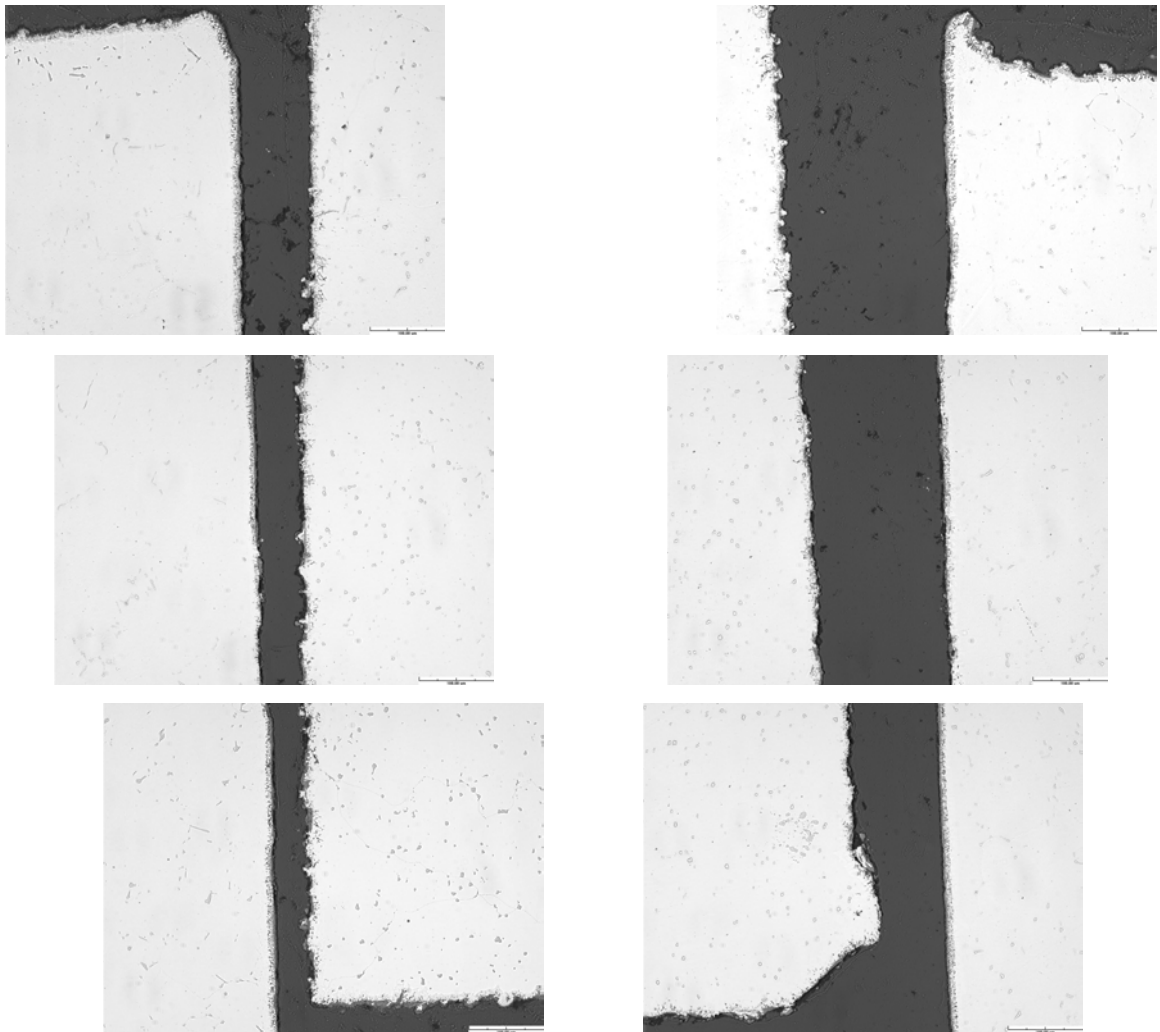


Figure A32 AARH004 – IN939 Pin in an IN738 Collar, 3000h at 750°C.

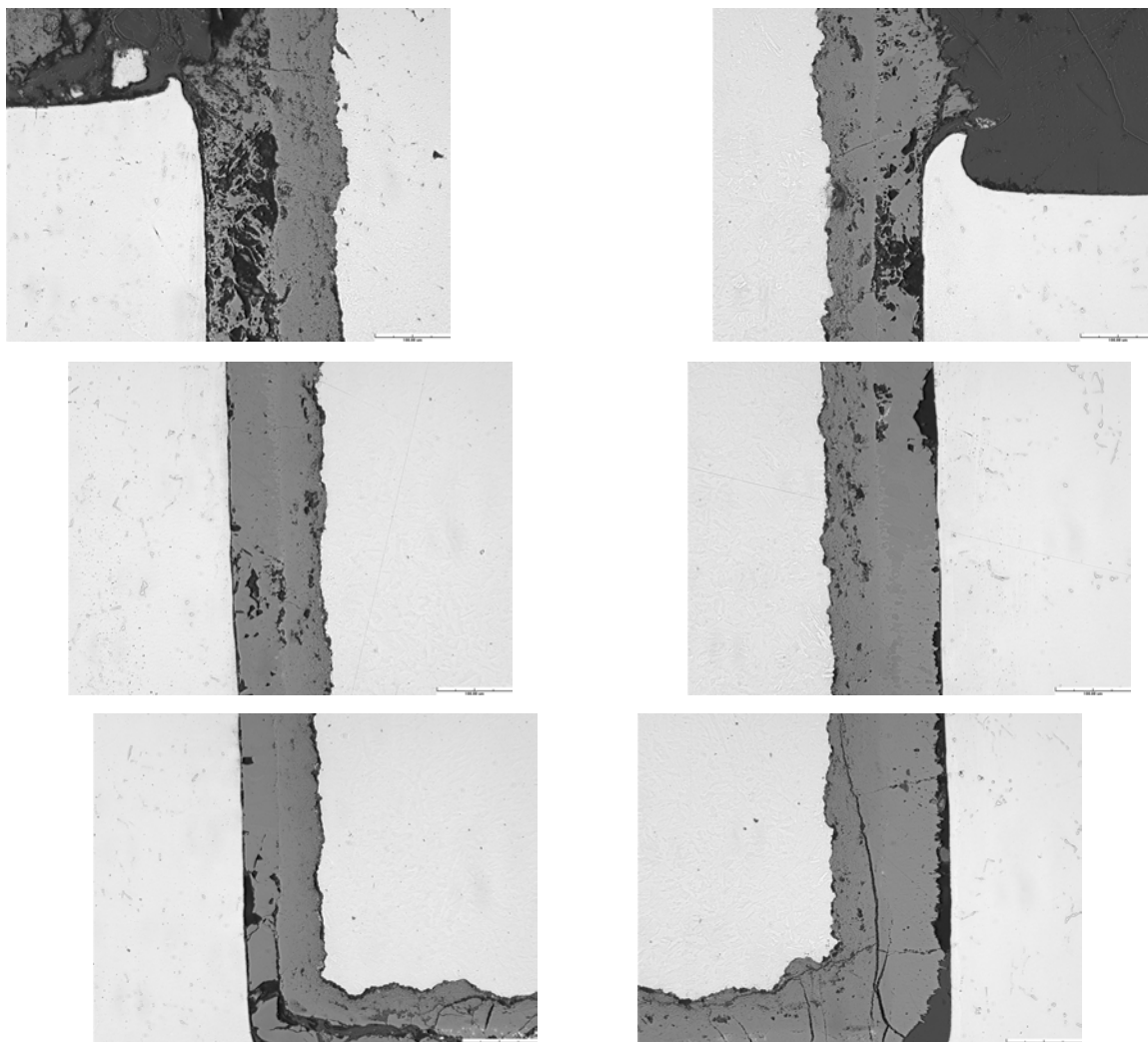


Figure A33 AARI001 – P92 Pin in an IN738 Collar, 500h at 650°C.

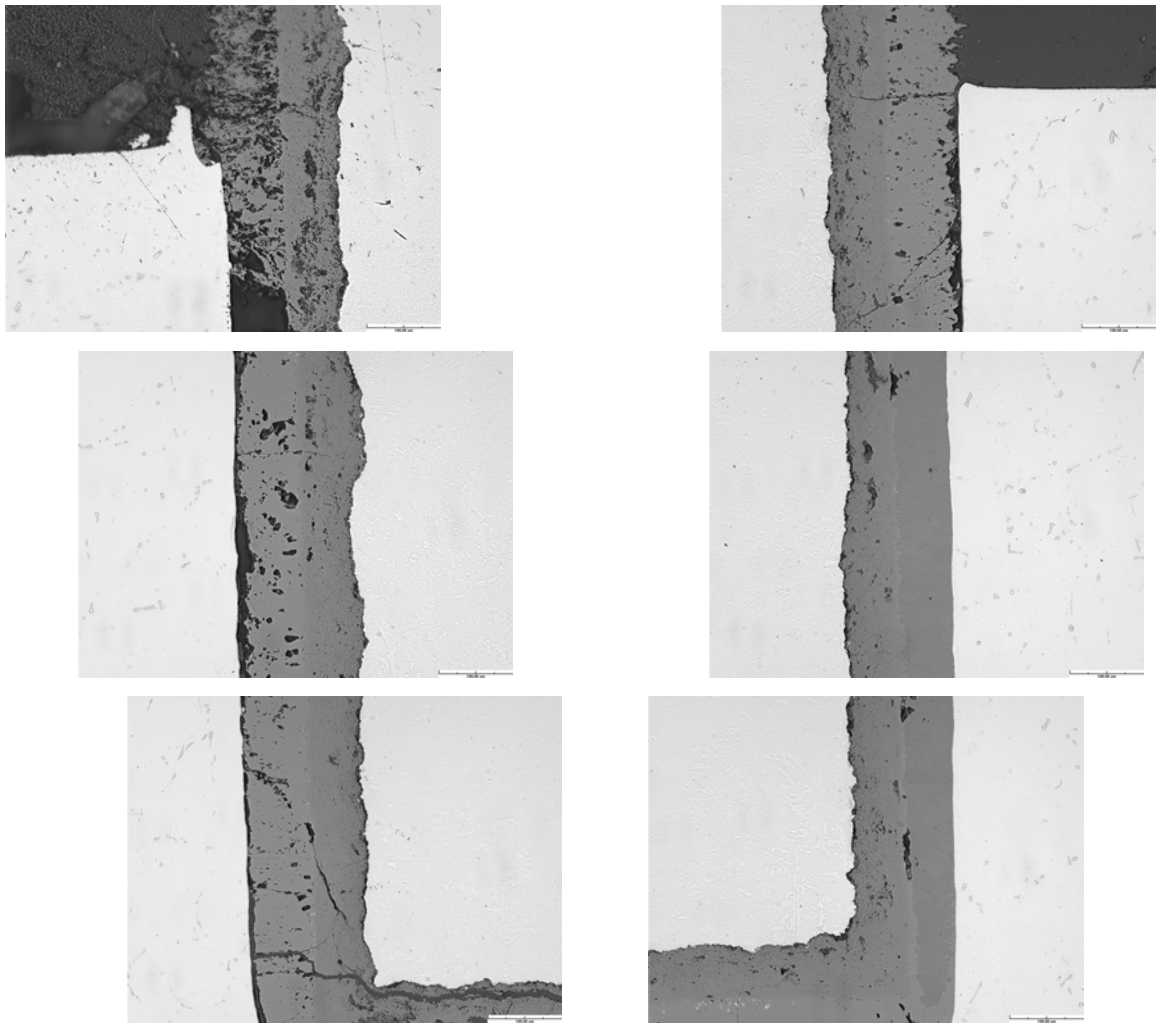


Figure A34 AARI002 – P92 Pin in an IN738 Collar, 1000h at 650°C.

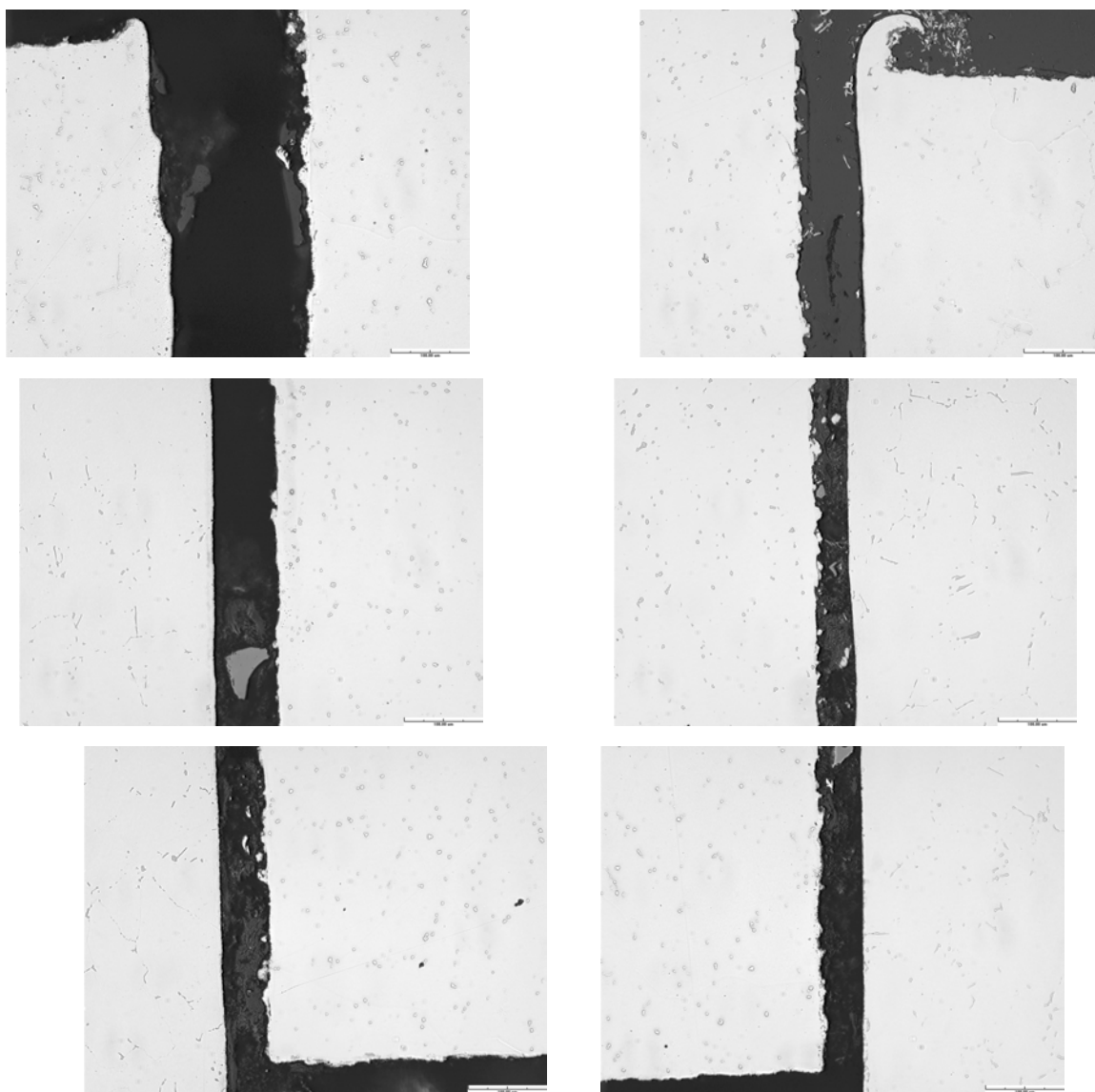


Figure A35 AARI003 – P92 Pin in an IN738 Collar, 2000h at 650°C.

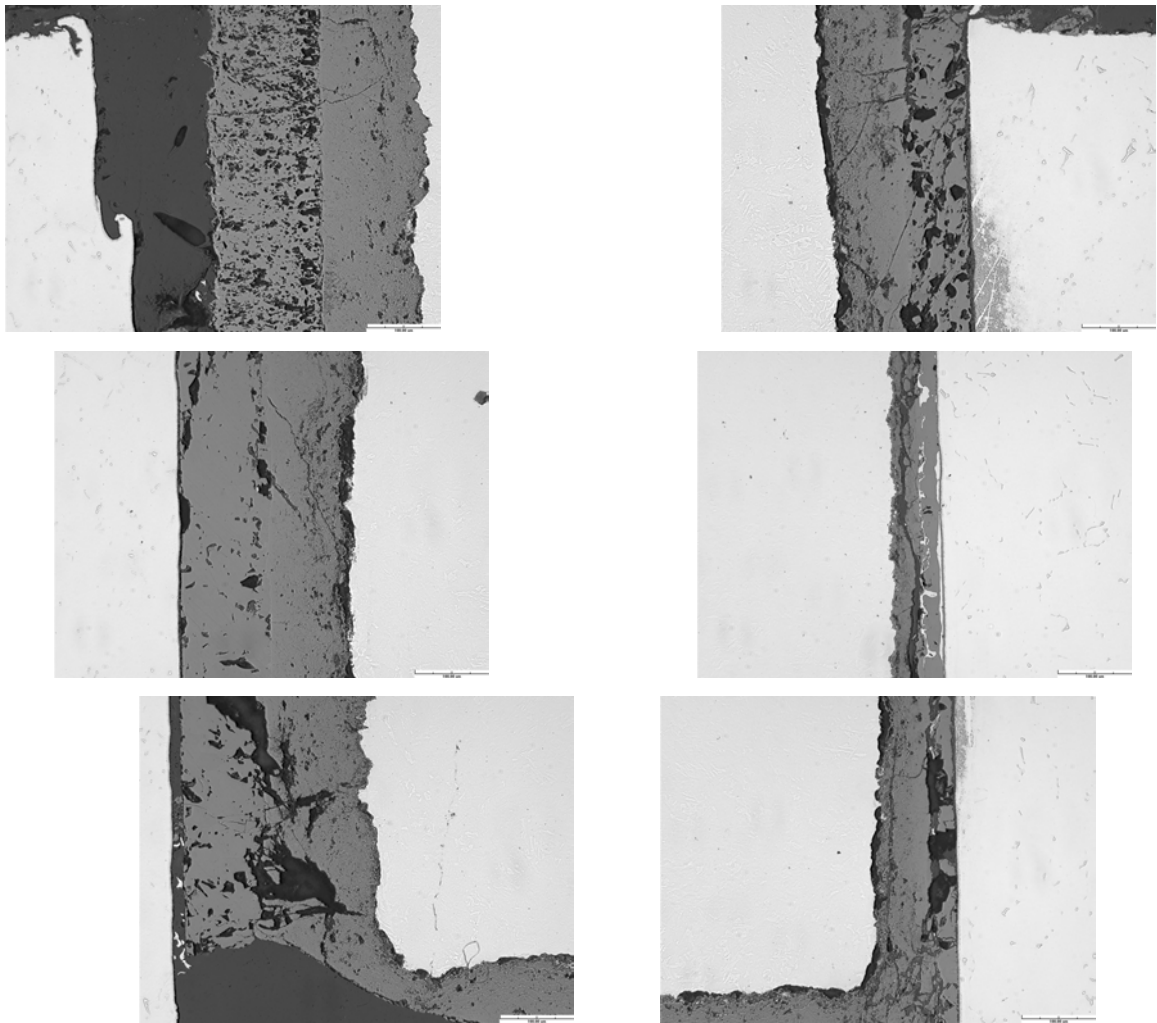


Figure A36 AARI004 – P92 Pin in an IN738 Collar, 3000h at 650°C.

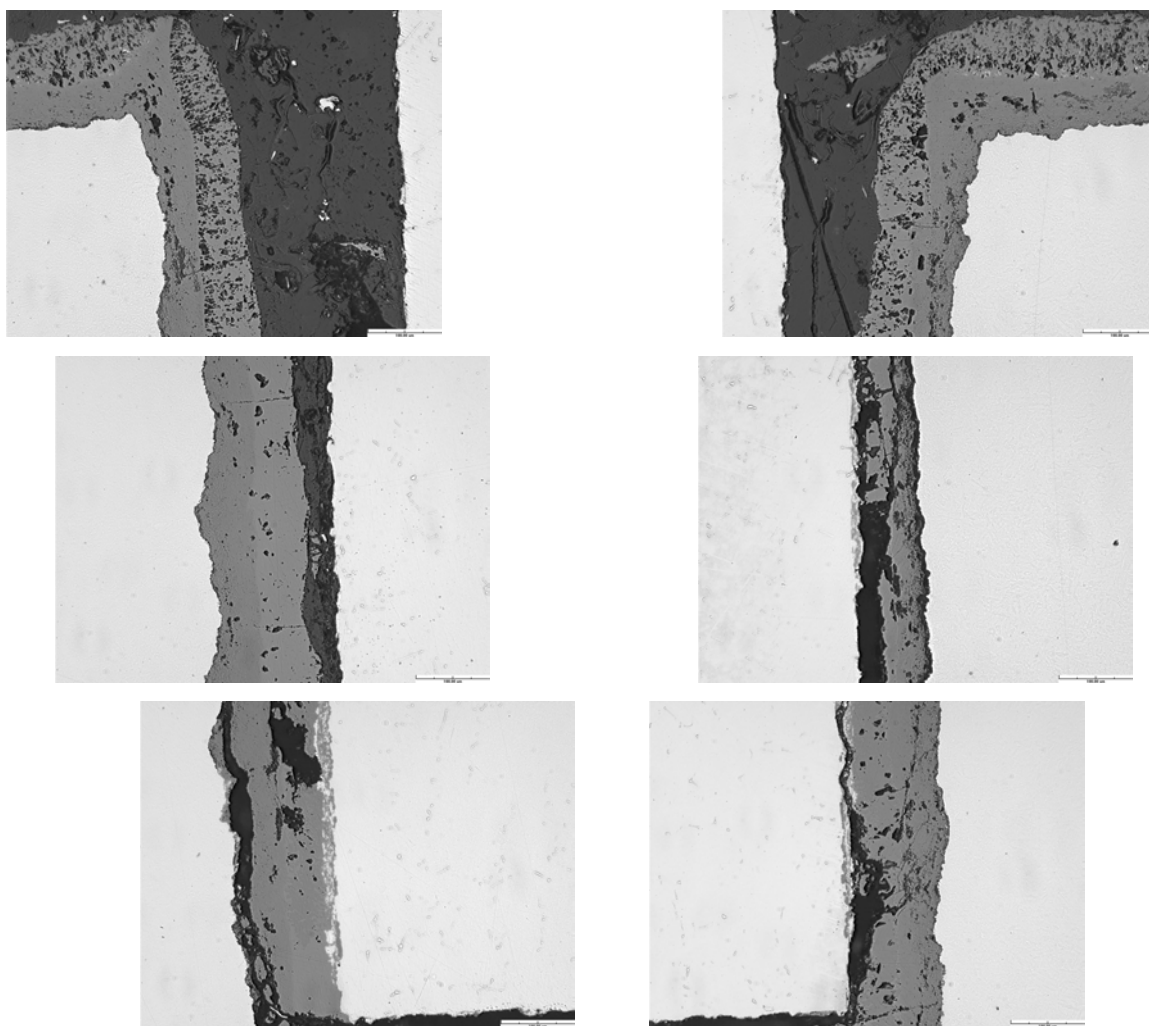


Figure A37 AARJ001 – IN738 Pin in a P92 Collar, 500h at 650°C.

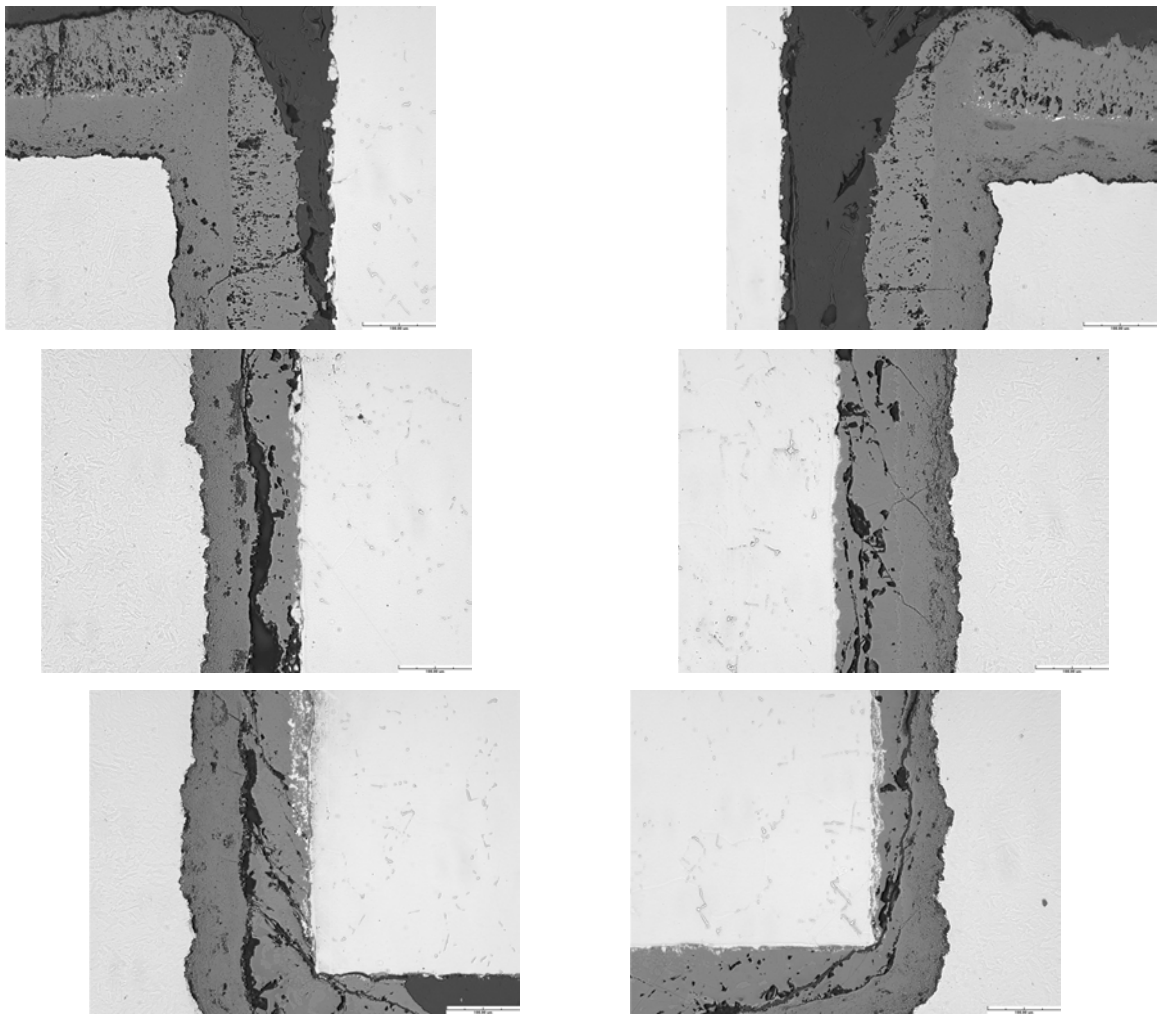


Figure A38 AARJ002 – IN738 Pin in a P92 Collar, 1000h at 650°C.

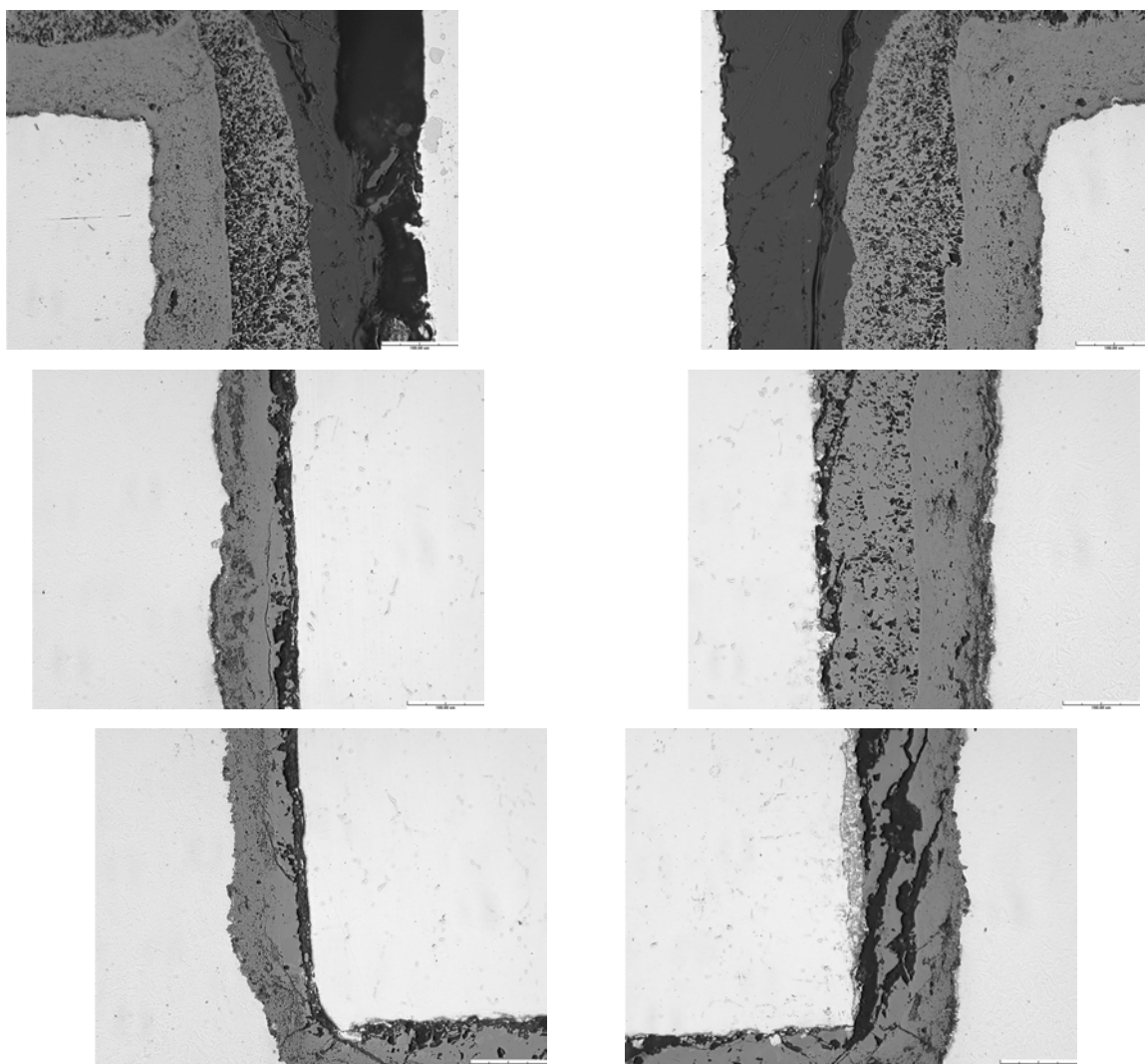


Figure A39 AARJ003 – IN738 Pin in a P92 Collar, 2000h at 650°C.

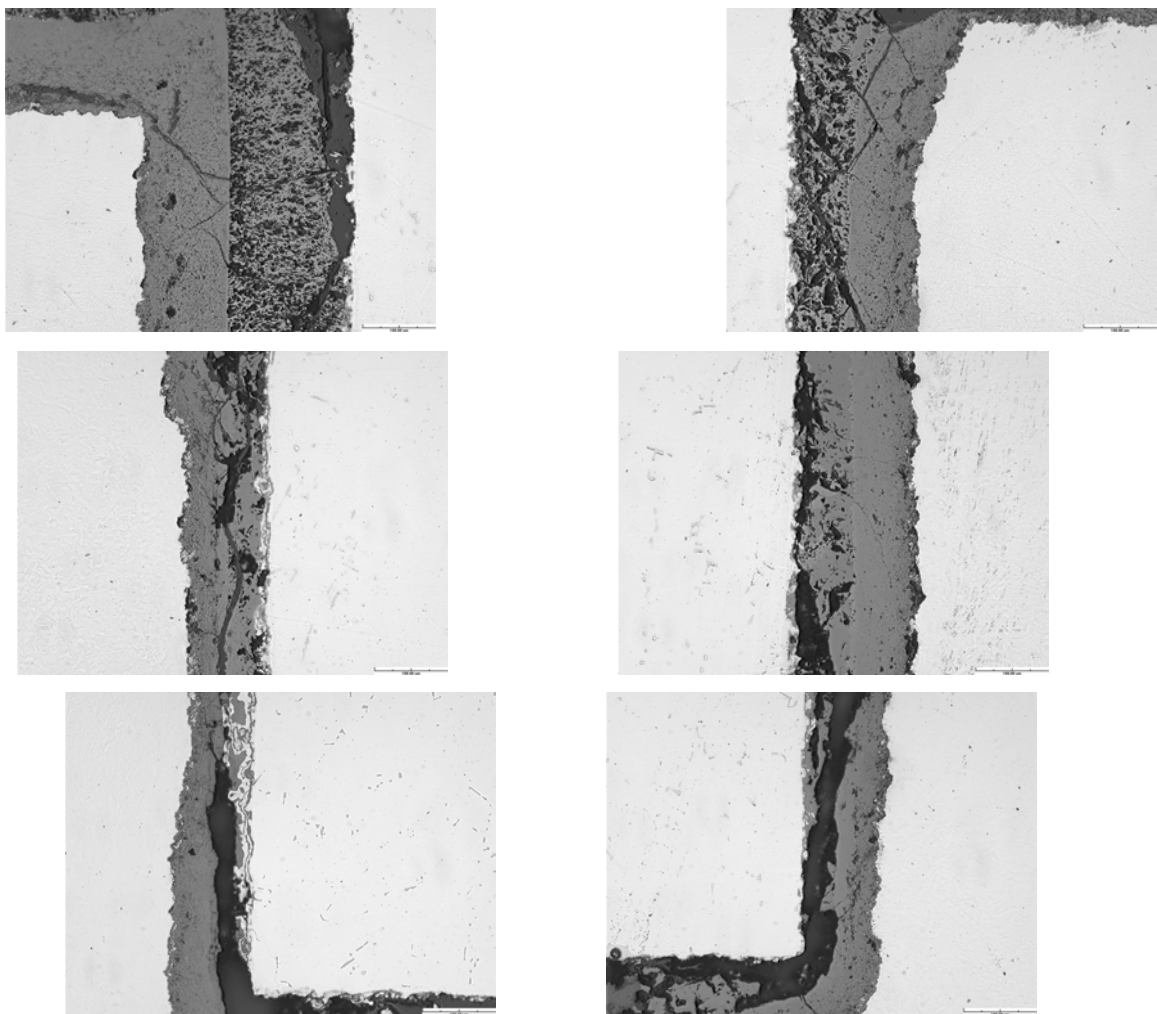


Figure A40 AARJ004 – IN738 Pin in a P92 Collar, 3000h at 650°C.

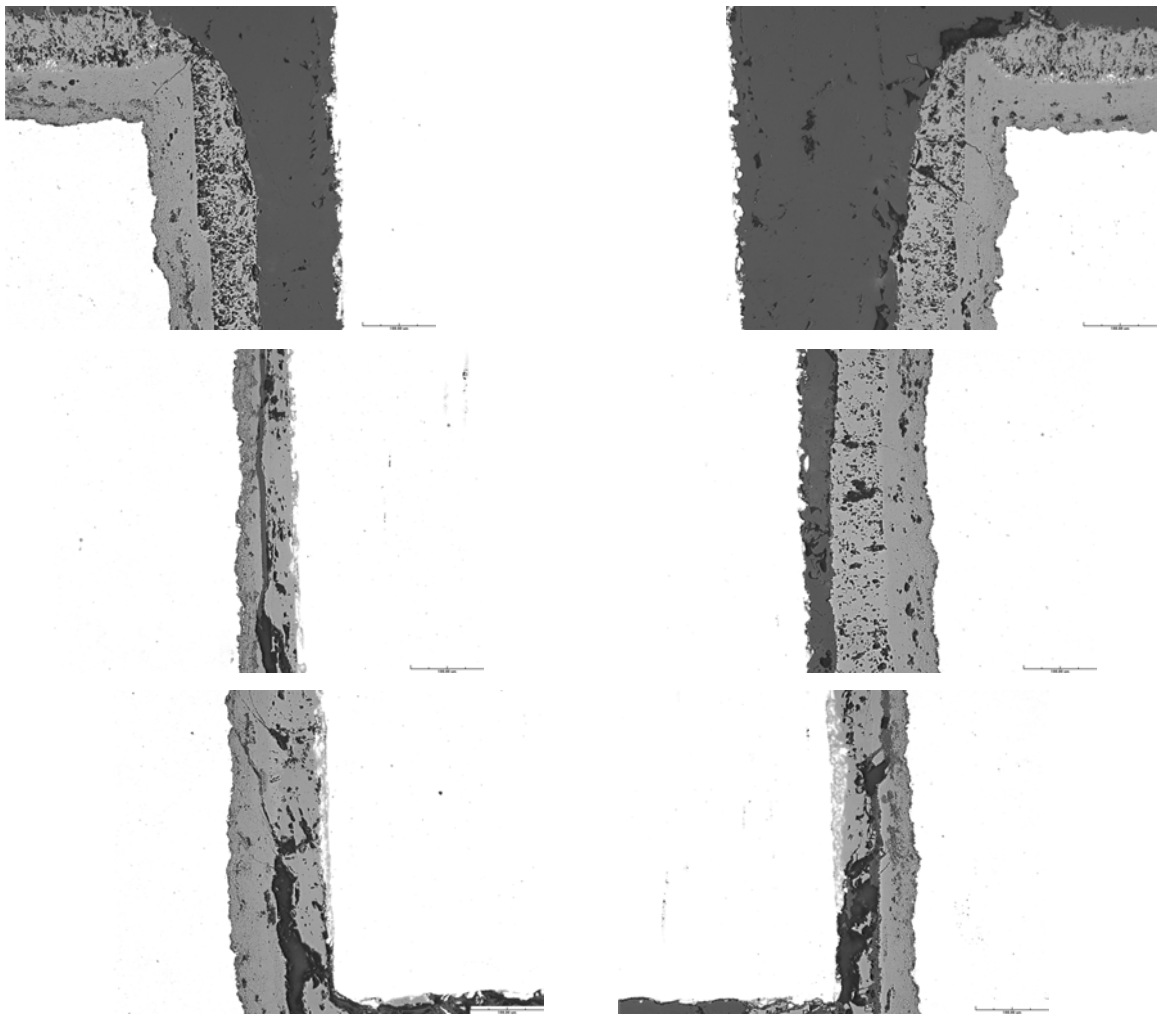


Figure A41 AARK001 – IN740 Pin in a P92 Collar, 500h at 650°C.

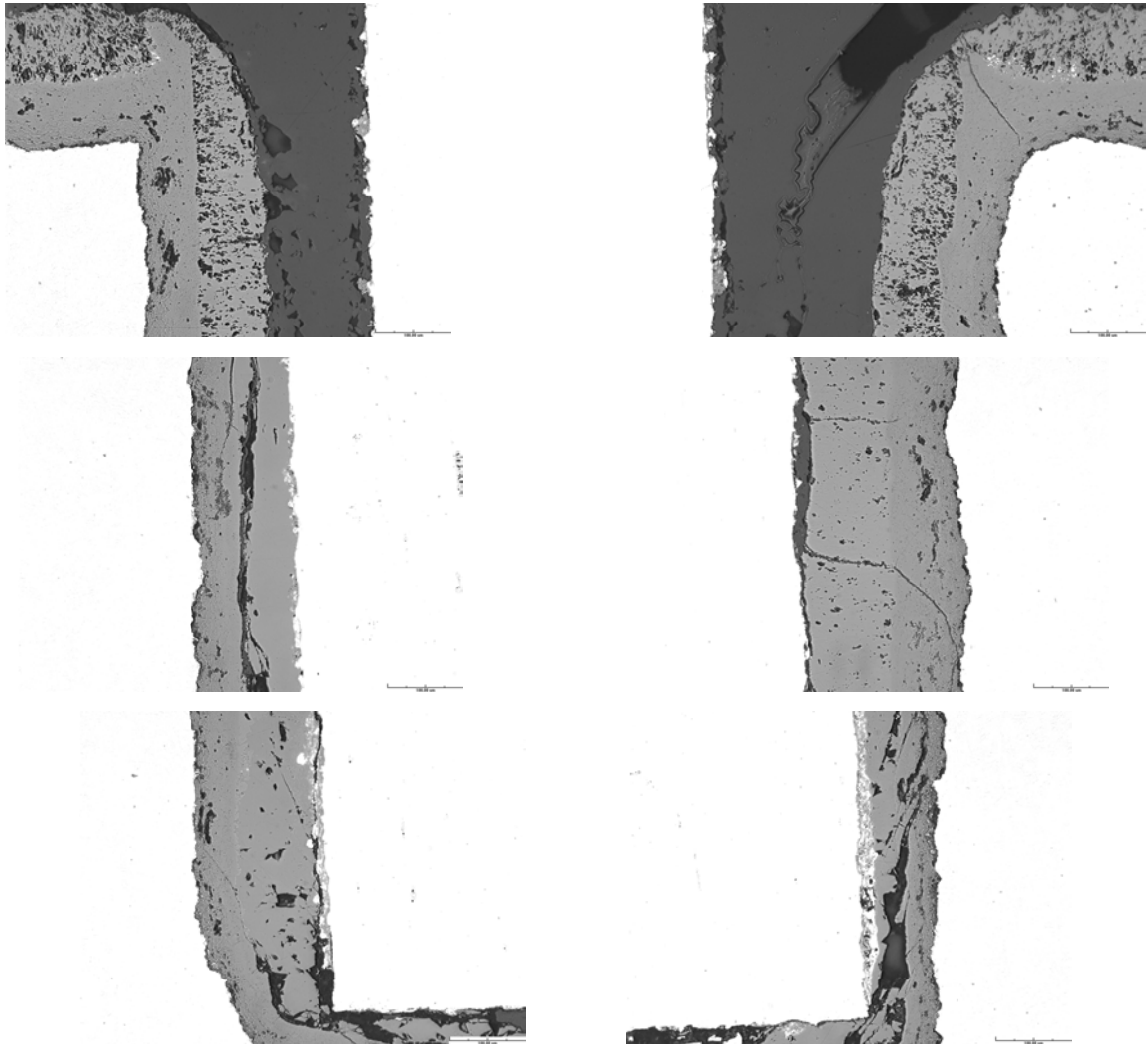


Figure A42 AARK002 – IN740 Pin in a P92 Collar, 1000h at 650°C.

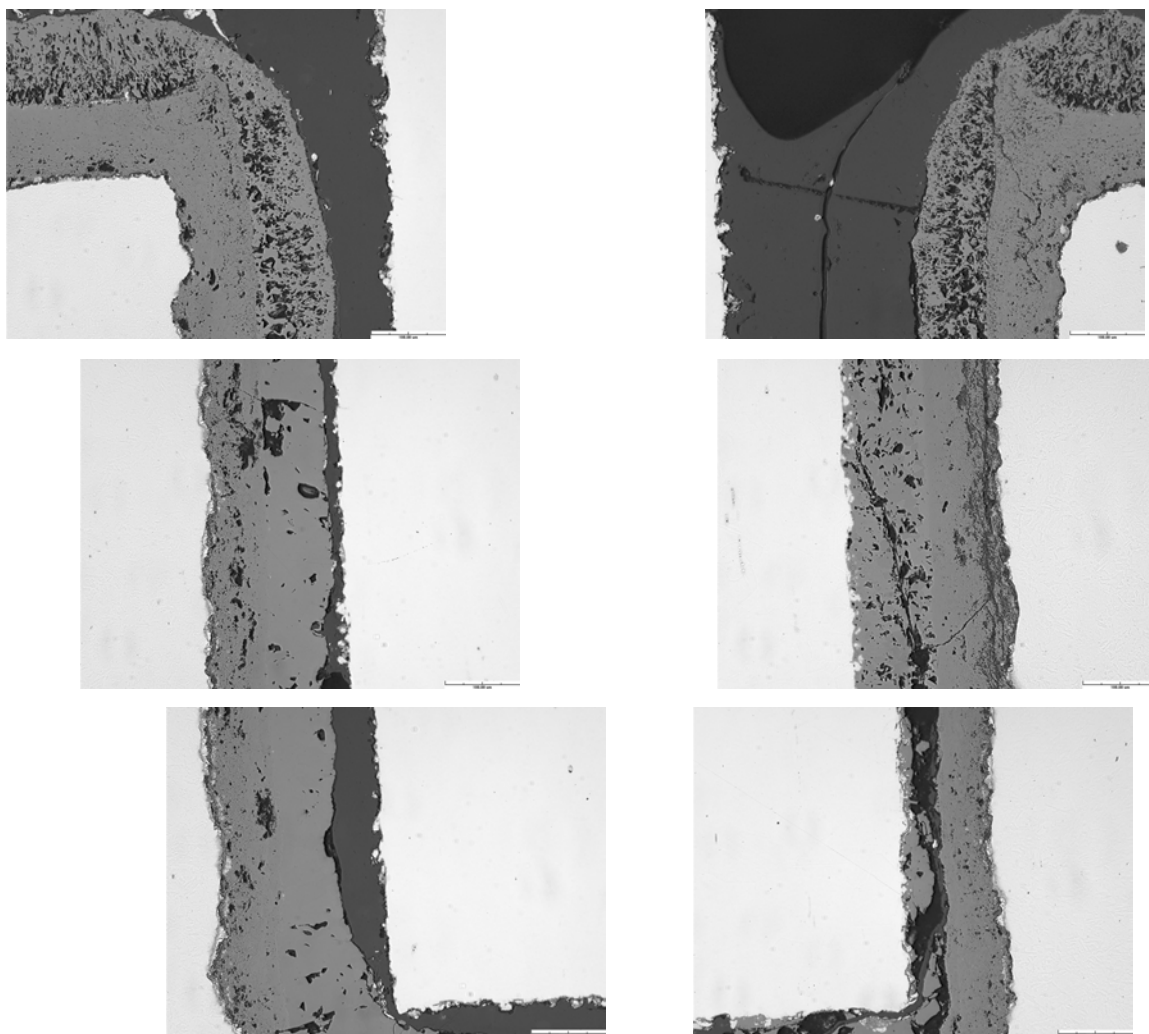


Figure A43 AARK003 – IN740 Pin in a P92 Collar, 2000h at 650°C.

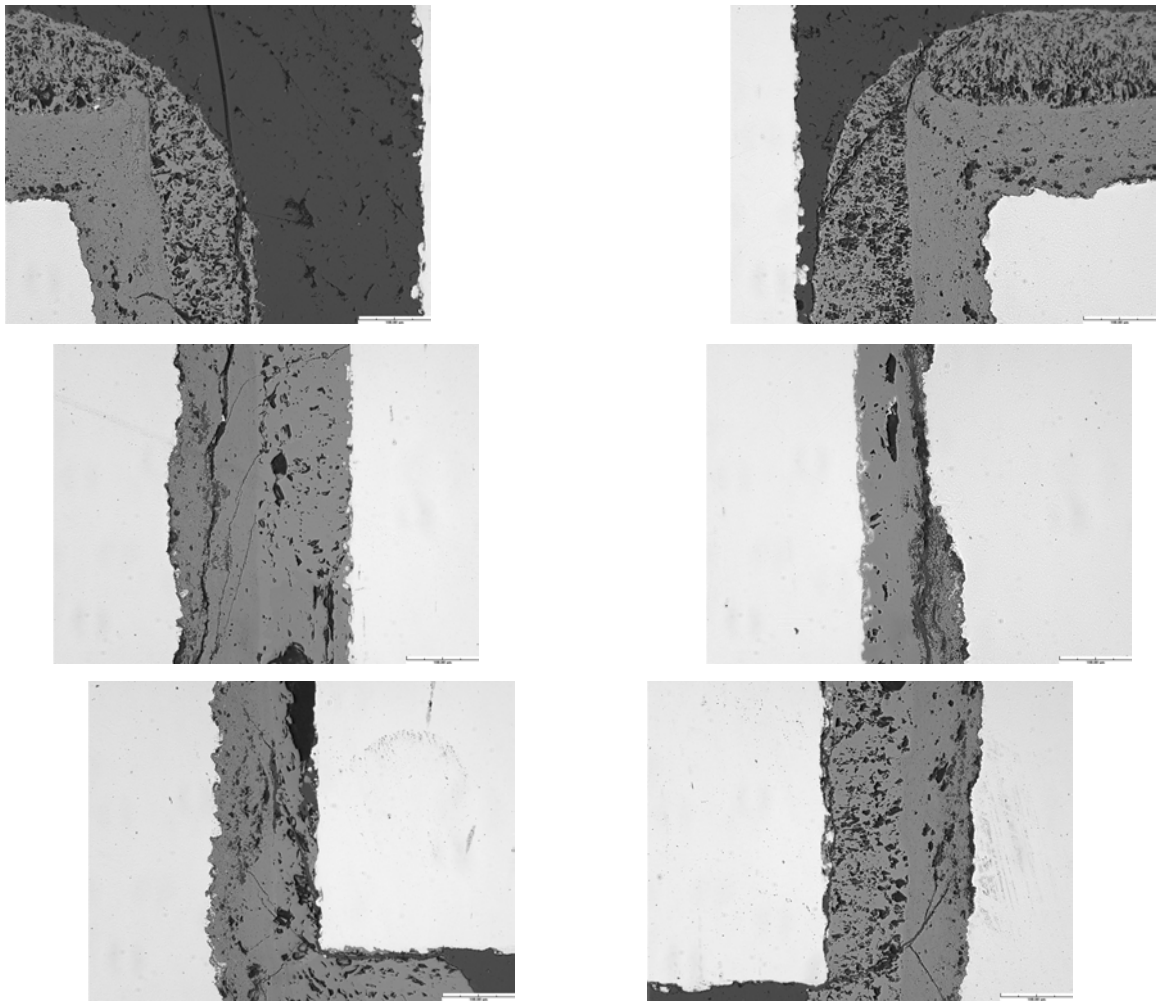


Figure A44 AARK004 – IN740 Pin in a P92 Collar, 3000 h at 650°C.

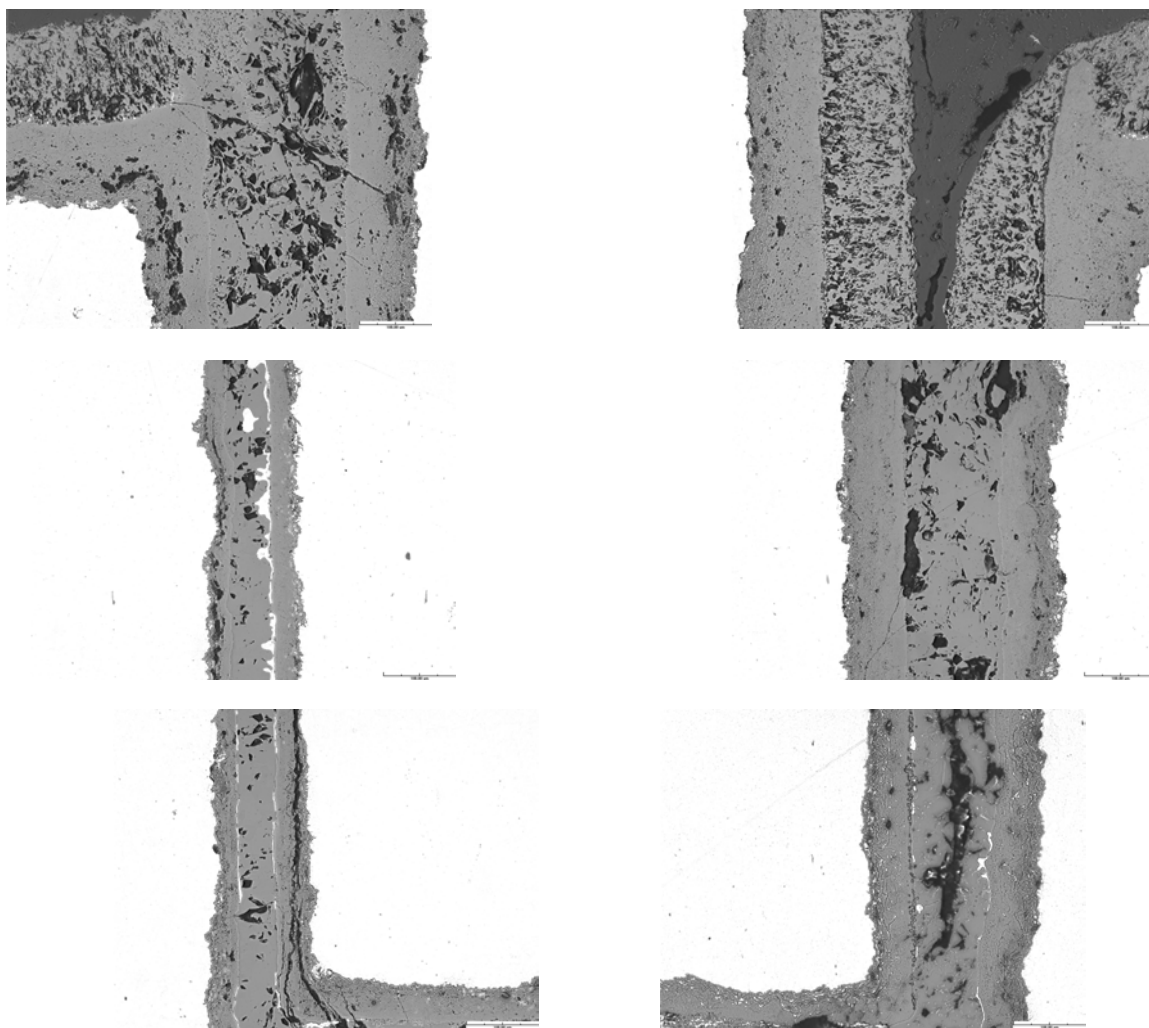


Figure A45 AARL001 – P92 Pin in a P92 Collar, 500 h at 650°C.

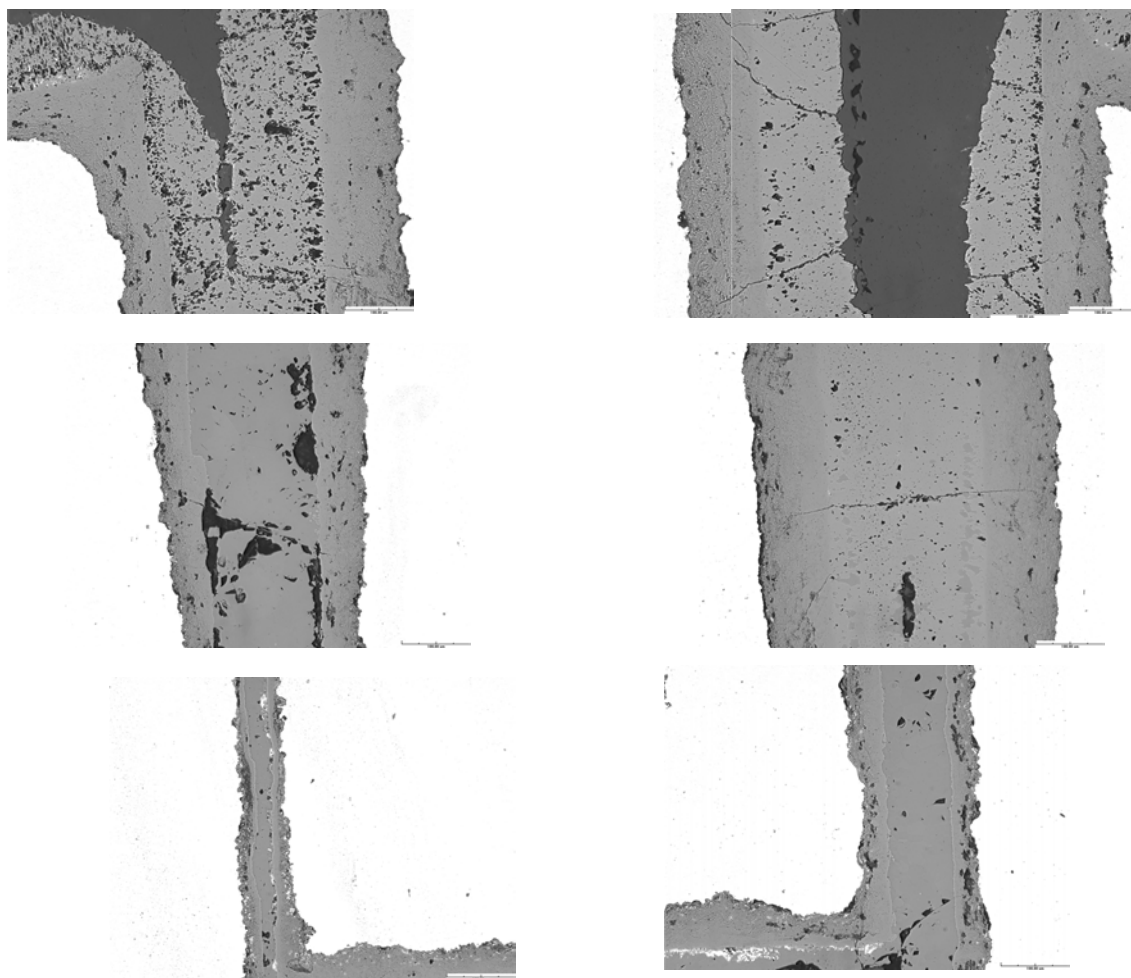


Figure A46 AARL002 – P92 Pin in a P92 Collar, 1000 h at 650°C.

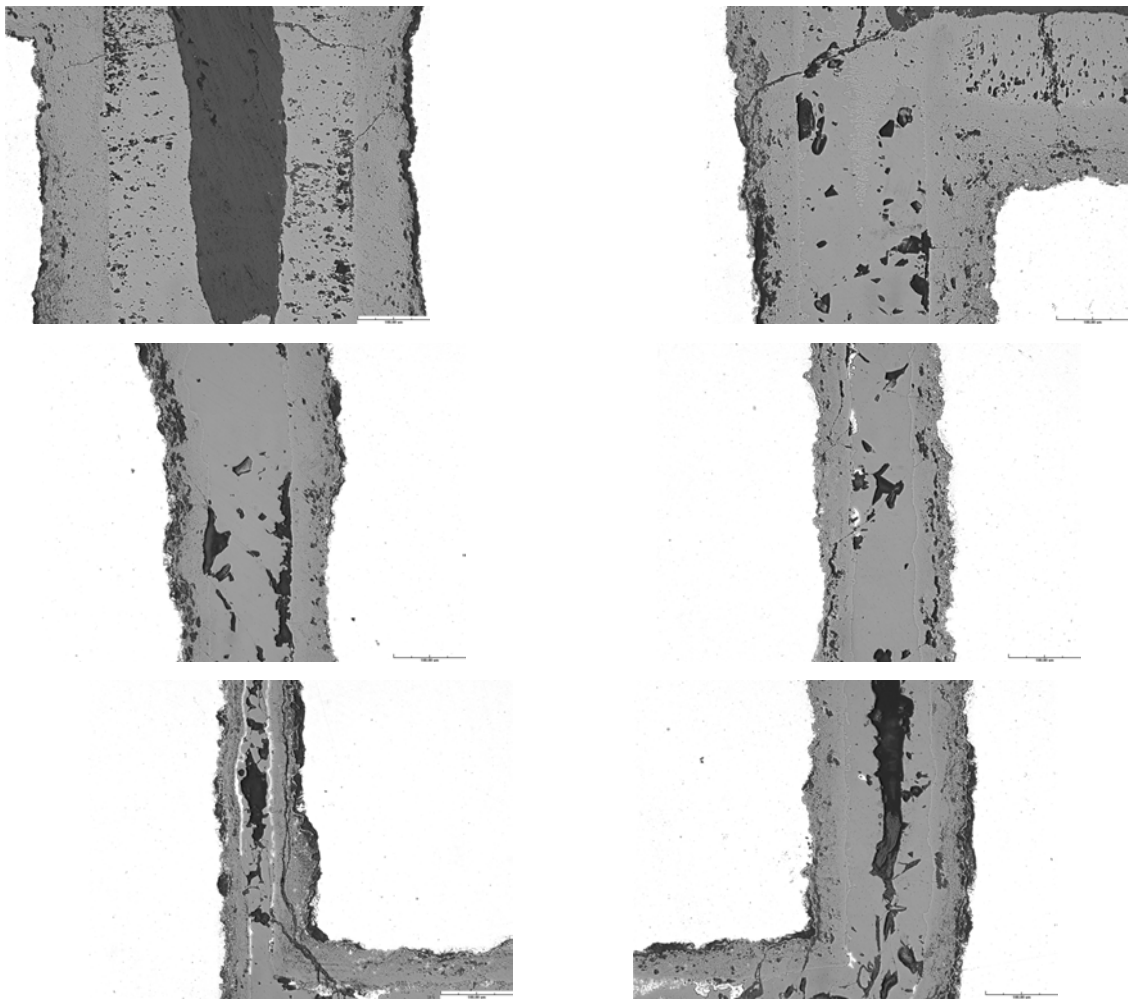


Figure A47 AARL003 – P92 Pin in a P92 Collar, 2000 h at 650°C.

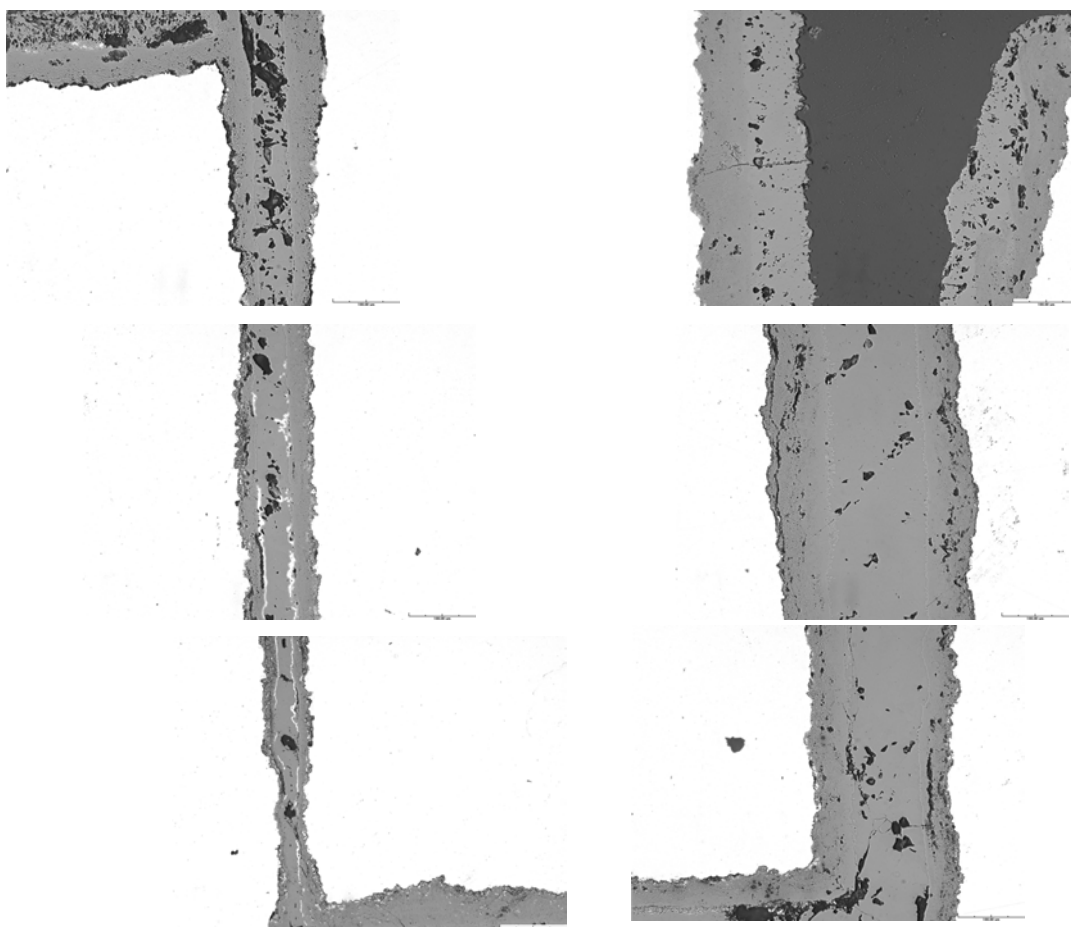


Figure A48 AARL004 – P92 Pin in a P92 Collar, 3000h at 650°C.