

# DEVELOPMENT OF A HIGH TEMPERATURE BOREHOLE FLUID SAMPLER

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## ABSTRACT

The objective of the high temperature fluid sampler development programme is to produce a downhole system capable of capturing a very high quality, representative sample of borehole fluid at a maximum temperature of 400°C and to deliver it to the surface for subsequent analysis. Minimal sample alteration and zero contamination are required.

The development of the high temperature fluid sampler for the NEDO Development and Production Technology for deep-seated geothermal resources research programme began in 1993 with the first prototype tool, rated for 300°C, scheduled for field testing during the summer of 1997. Following these tests the second prototype rated for 400°C will be designed, built and tested. The programme is currently expected to continue through to the year 2000.

The development programme has included current state of the art, concept and feasibility assessments. Extensive material corrosion and high temperature seal performance studies and subsystem design studies have been carried out. Several types of sampler have been identified and the controlled displacement sampler ( CDS ) method chosen as the one which offers the best potential for the highest quality sample and development at high temperature. A high temperature CDS sampling tool has been designed and constructed and is ready for downhole testing.

This paper presents the developments to date and outlines the sampler design.

## INTRODUCTION

In 1993 CSM Associates Limited (CSMA) was invited to participate in the NEDO geothermal research programme to develop the technology necessary to exploit the deep seated geothermal resource. CSMA's background in high temperature electronics, borehole instrumentation and extensive R&D experience were considered suitable to undertake the development of a very high performance borehole fluid sampler for use at temperatures of up to 400°C.

The sampler development programme was scheduled to take place over a period of some 7 years. The first year was a study into the current state of the art for fluid sampling, the techniques and methods used, the operational temperatures achieved and a recommendation for the new tool concept and its development. The second year established some basic material and seal properties at high temperatures and a first draft of the outline design was produced. Year three concentrated on the detailed design of a sampler rated for use at 300°C, including an extensive electronics system study, actuation system simulation and testing and the thermal system design. During the current year four, the sampler has been constructed and will have some preliminary downhole tests performed. Years five and six will see the sampler tested at 300°C and the 400°C sampler designed and constructed. Year seven will test the sampler at 400°C and assess what further developments are required.

## CONCEPT STUDY

The concept study included the following:

- Operational criteria and specification
- Risk assessment
- Technology review
- Materials review
- Design concepts
- Design recommendation

## Operational Criteria And Specification

The basic specification for the sampler was established to be as follows:

Temperature endurance	6 hours @ 400°C
Pressure rating	70 MPa @ 400°C
Tool diameter	82mm
Tool length	10m max
Sample volume	2 Litre
Deployment cable	Slickline
Fluid velocity	0-2m/s
Surface temperature	25 - 250°C
Surface pressure	0 - 10MPa

In addition to these an extensive list of analysis requirements from liquid and gas phases were given including geochemical and isotopic measurements.

### Risk Assessment

In the context of the sampler development, the interpretation of risk was the potential for a component or system to fail to perform to specification. Four categories were identified within which the risk of failure could be considered, namely:

- Geochemical
- Thermal
- Mechanical
- Operational

Within each category the sampler can be divided into mechanical and electronic systems.

The geochemical risks were considered to be limited to the mechanics in the form of contamination from corrosion products or sample alteration due to the method of capture and / or retention. Careful material selection and testing would overcome the first risk. An extensive study of sampling methods, their known problems and the geochemical behaviour of geothermal brines would be undertaken to minimise the second risk. Leakage into and out of the sample chamber were known to be potential problems.

The thermal risks include the mechanical issues of strength at 400°C, linear and differential expansion and stiffness of non-metallic parts. The seals pose the most demanding challenge. There are some (static) polymeric seals that CSMA has operated at temperatures up to 380°C and many field worthy seals for lower temperatures (up to 300°C). For very high temperatures it is difficult to avoid the use of metal seals but these are known to be difficult to operate reliably and the very high standard of cleanliness required does not lend itself to field operations. Electronically the risk is that the heat shield system (dewar) will not provide the required lifetime. As the heat leakage into the dewar is proportional to the temperature difference raised to the fourth power ( $\Delta T^4$ ) the electronics system would be operated at temperatures up to 200°C (the economics of motor and battery selection limited the final system temperature to 150°C).

The mechanical risks were confined to those associated with direct stress and were identified as being present in valves, seals, actuation mechanisms, material matching, sample extraction and manufacturing.

Operational risks were considered to be those associated with human interaction and although difficult to comprehensively define include those issues that could effect the ability to capture a high quality sample, eg

- ease of assembly and disassembly
- ease of cleaning the sample chamber
- ability to test and set the system functions
- ease of deployment
- ability to purge and seal the transfer system

## Technology Review

The technology review was an assessment of the state of the art in borehole fluid sampling and was conducted in three categories - a literature search, patent search and product search.

The literature search produced 82 relevant references based upon sampling devices, techniques and materials. The patent search produced 26 relevant patents from some 10 different countries, though some were duplicate filings of the same patent. The product search produced five relevant devices (moderately high performance) from a larger number of low specification products.

The combination of technical papers, patents and products provided a clear understanding of the current sampling technology and capability. Although some groups, notably in the USA - Los Alamos National Laboratories, Lawrence Berkeley Laboratories, Sandia National Laboratories and in the UK - Leutert (North Sea) Ltd, have engaged in some very high temperature sampling attempts none have achieved any reliable success at temperatures much above 300°C. It was imperative that as much as possible should be learnt from their experiences.

## Materials Review

The purpose of the review was to identify the materials that would be suitable from which to manufacture of the sampler. Two environments exist within the sampler, the dewar internal volume where temperatures at the opening could be as high as 300°C but with no contact with the borehole fluid and the sample chamber/tool structure where 400°C and high pressure, corrosive fluids would be present.

For the internal environment few problems were anticipated as there was an extensive range of alloys, plastics and ceramics which could meet all the expected design requirements. The principal problem was in the choice of materials for both the basic structure of the sampler and the sample chamber. Here two different levels of performance were required. For the structure the alloys used must be sufficiently corrosion resistant in order to avoid any corrosion damage to the close tolerance, fine finished sealing surfaces. For the sample chamber the same requirement exists except that the level of corrosion must be sufficiently low in order not to contaminate the sample itself.

For the internal materials little review effort was required. For the structural materials there were several alloys which appeared suitable, but for the sample chamber there was little or no data available at temperatures above 250°C or below 700°C on which to base an informed choice. A series of high temperature corrosion tests would be required.

## Design Concepts

Four basic methods of capturing a sample were identified. A pumped system where the sample is introduced to the sample chamber by a mechanical pump, a suction system where a piston is drawn down a cylinder by a mechanical arrangement, a flow-through system where both ends of the sample chamber are open allowing well fluid to pass through and which is closed at the required time trapping the sample fluid and a vacuum type where an empty volume is sealed and transported downhole to be opened at the required time allowing the well fluid to pass through a non-return valve.

The first two methods can be discounted due to mechanical complexity and unsuitability for development at 400°C. The vacuum system has the disadvantage that high temperature fluid entering a low pressure region will boil with subsequent alteration to the fluid chemistry. The flow-through system is the most simple and could be used at 400°C but cannot guarantee that the sample of fluid inside the chamber is representative of the borehole fluid at the depth of sampling. This is particularly true in non or low flowing zones, where the fluid inside the chamber could be a mixture from the borehole over some distance above the point of sampling.

As the concept study progressed a variation of the vacuum type of sampler became apparent, the controlled displacement sampler (CDS). In this system the sample chamber is separated by a moving piston whose position is controlled by a working fluid (water). When the sampler is opened, high pressure borehole fluid enters the sample chamber above the piston. The piston can only move slowly because the working fluid flow is restricted and can only leak away into an empty chamber at a controlled rate. As a result the pressure of the sample fluid is maintained close to that of the borehole and there is no boiling. The principal difficulty with a CDS system at 400°C is the need for dynamic sealing on the piston.

## Design Recommendation

Given the requirement of capturing as high a quality of sample as possible the recommended type of sampler was the controlled displacement type. This recommendation was supported by the fact that all the technology required for the CDS type would be required for the flow-through type and could thus be adopted as the fall back position if the CDS proved to be unsuitable.

## MATERIAL AND SEAL TESTING

### Materials Testing

A study of potential materials produced a list of alloys from three principle groups - nickel, cobalt and titanium. In addition stainless steel and bronze alloys would be included. The list of some of the materials tested is given in Table 1.

A corrosion test schedule was developed that would expose the selected metal specimens to a simulated geothermal (hydrothermal) environment. A reaction test vessel was designed and manufactured from a high strength stainless steel and gold lined to produce an inert enclosure. The vessel was designed to use a metal seal with a testing temperature of 400°C. However the preliminary tests to check the seal performance showed the metal seals to be unreliable even in laboratory conditions and so a custom made PTFE seal was substituted and the test temperature reduced to 325°C. This arrangement worked well.

The downhole hydrothermal environment was created by using deionised water and a mixture of mineral powders that were manufactured from selected mineral specimens. The minerals - pyrite, chalcopyrite and bornite with a CO<sub>2</sub>/C buffer would fix the pH, hydrogen sulphide and oxygen at demanding, but geologically feasible, values.

A total of six reaction vessels were used, one set as a buffer blank, containing no sulphide minerals but using thioacetamide to create a known initial concentration of hydrogen sulphide free from the uncertainty surrounding the performance of the complex mineralogical buffers and to test the possibility that the test results might have been misleading because of the acceleration of corrosion by transition metals released from the sulphide buffers. Sufficient fluid was included to ensure reaction pressures of approximately 20 MPa. The test vessels were filled and assembled in a dry CO<sub>2</sub> environment and sealed. They were then placed into an oven at 325°C for a period of 28 days prior to opening.

The results were determined by comparison of pre and post test weight, optical inspection, scanning electron microscope (SEM) inspection and polished section/SEM inspection.

Some predictable results were observed eg S316 stainless steel suffered severe pitting corrosion and the bronze alloy deep surface alteration and leaching of copper. A surprising result was the severe corrosion experienced by the titanium alloys. Later discussions with the manufacturers of titanium alloys suggested that previous performance expectations were based upon test temperatures of 200°C and that probable limits of 250°C apply to titanium in corrosive environments. Plates L1 & 2, P1 & 2 and V1 & 2 show examples of corroded specimens of titanium alloys Ti-grade 12, Ti-21S and stainless steel S316 at low and higher magnifications.

### Seal Tests

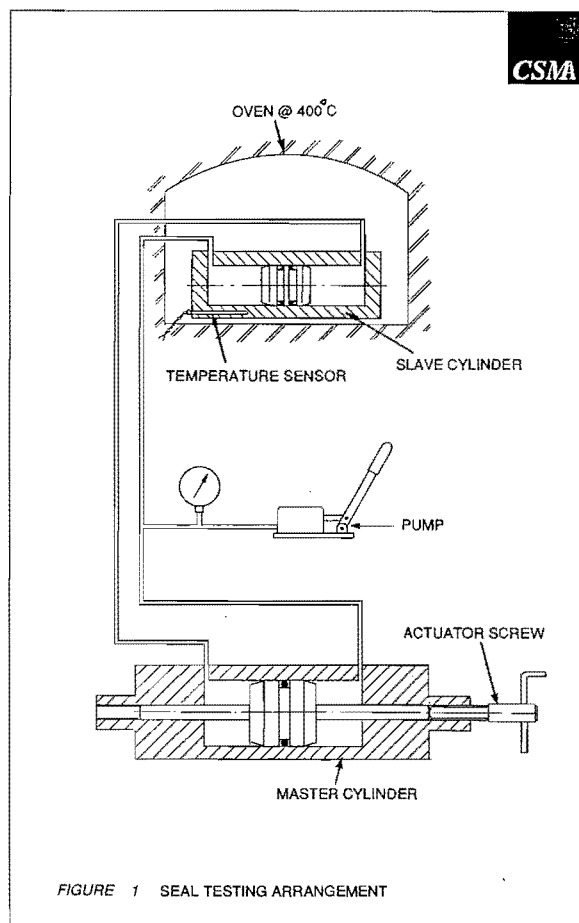
The CDS type of sampler requires the use of a dynamic (moving) seal on the piston that travels down the sample chamber. In addition it also requires seals that will operate reliably at 400°C.

The dynamic seal that was thought to offer some useful high temperature capability is of a filled PTFE material that is spring energised. CSMA has used a similar seal in static applications at temperatures as high as 380°C and so wished to evaluate its dynamic performance potential.

TABLE 1

MATERIAL
Nickel alloy 625
Nickel alloy C22
Nickel alloy 242
Nickel alloy B2
Nickel alloy C276
Nickel alloy S
Titanium alloy Ti 50 A
Titanium alloy Ti Grade 12
Titanium alloy Ti 21 S
Titanium alloy Ti 6Al 14V
Stainless steel S316
Cobalt alloy MP35N
Cobalt alloy MP159

The test rig for the dynamic high temperature tests consisted of a slave and master cylinder arrangement. The slave cylinder contained a piston fitted with two seals for testing. The piston could be moved by displacing the piston in the master cylinder. As Figure 1 shows the slave is placed into the furnace and remotely connected to the master. Once the slave was up to the required temperature the seals were moved for 30 strokes of the slave cylinder and then pressurised differentially to 50 MPa to confirm and test the sealing integrity. This test closely reflects the requirements of the CDS seals.



The results showed that the PTFE seals worked well at temperatures up to 300°C but at 358°C suffered extrusion and failed. Clearly the absolute operational limit was above 300°C but this was probably the safe limit.

As the corrosion tests had shown the metal seals did not perform reliably. A series of tests were carried out to determine what thickness of coating, when applied to the metal seal, would achieve reliability. To do this three seals were coated with 10, 25 and 75µm of gold respectively and fitted onto one of the reaction vessels with an adapted lid that enabled a hydraulic pump to be connected. The intention was to raise the pressure to 100 MPa at room temperature and hold this for a seven day period. The 25 and 75µm coated seals worked well but the 10µm coated seal would not maintain a pressure above 30 MPa. For economic reasons the 25µm coating was the recommended thickness.

#### SAMPLE CHAMBER DESIGN (300°C)

The material and seal trials confirmed what could reasonably be expected to work at 300°C and so the detailed design of the sampler was commenced. The main intention throughout the design phase was to achieve 400°C rated systems where reasonably possible. This would ensure maximum development experience and minimise the amount of redevelopment necessary when the final 400°C sampler is designed.

To this end the concept for the sample chamber assembly would be a sample chamber and working fluid housing inside a heat

shield, all located inside the main pressure housing. In this way the temperature to which the internal assembly would rise would be controlled by the incoming hot sample fluid and the heat capacity of the mechanical assembly. There would also be the advantage that the heat shield would keep the sample hot and eliminate the volumetric contraction that causes the internal pressure to drop and risk the inward leakage of additional well fluid as the tool is extracted from the borehole.

One of the main developments during the design phase was the non-metallic inlet valve seal. This was developed to ensure reliable sealing of what will be a liquid containing fine solid particles. Large particles will be removed by passing the sample fluid through a fine, 10µm, filter. The trials of the seal included liquid with 10µm rock particles in, pressure ranges from 35-140 MPa for periods of some 15 hours and temperatures up to 470°C.

The other two developments were the sample extraction point bursting discs and the sampler actuation system which uses a magnetic coupling mechanism across an internal pressure housing to operate the opening latch system.

Due to an impending patent application it is not possible (at the time of writing) to add any more detail to the above.

#### ELECTRONICS SYSTEM

The electronics system is being developed on two levels. The first is a comprehensive microprocessor based system which will have the capability to monitor and record the borehole and electronics temperatures, system and actuator battery voltages and currents and accept actuation command data, all with respect to time. The system would be self monitoring using a "watchdog" circuit to ensure that one bad data point will not cause a system failure. The watchdog circuit would have its own, real time, clock and in the event of a microprocessor system failure would be capable of resetting and starting the processor inserting the actuation command data with respect to real time.

The second level of electronics development is a simpler system for development use. This uses 9 pre-set delay periods which are mechanically selectable. After the chosen period has expired the actuation motor is switched on until it reaches a limit switch when it stops. There is no monitoring or fail safe system.

The simple system is being used for the 300°C sampler and it is expected to use the comprehensive system for the 400°C sampler. Both systems will be dewatered and rated for use at temperatures up to 150°C.

## TESTING

The 300°C sampler will be subjected to a series of tests at CSMA's borehole test site to evaluate the systems functionality performance. Samples at depths of 2.6 Km and temperatures of 100°C will be taken. Some tests will include runs down the borehole but without opening the sampler. This will confirm that the inlet bursting disc system and actuation system do not leak or operate unintentionally. After the UK based tests the sampler will be shipped to Japan for testing at 300°C in geothermal boreholes. A similar testing programme to that already carried out will be performed.

## FUTURE WORK

After the downhole tests at 100° and 300°C the sampler design will be reviewed. Any problems with the design will be addressed and development work carried out as necessary. Following this the 400°C sampler will be designed and constructed. Once the testing at 400°C is completed it is possible that further development work will include developing additional sensors on the tool to monitor the sample temperature, pressure, conductivity and pH. The development of a multi-chamber sampler, to enable time lapsed or multiple samples from one run into the borehole, may be considered.

## ACKNOWLEDGEMENTS

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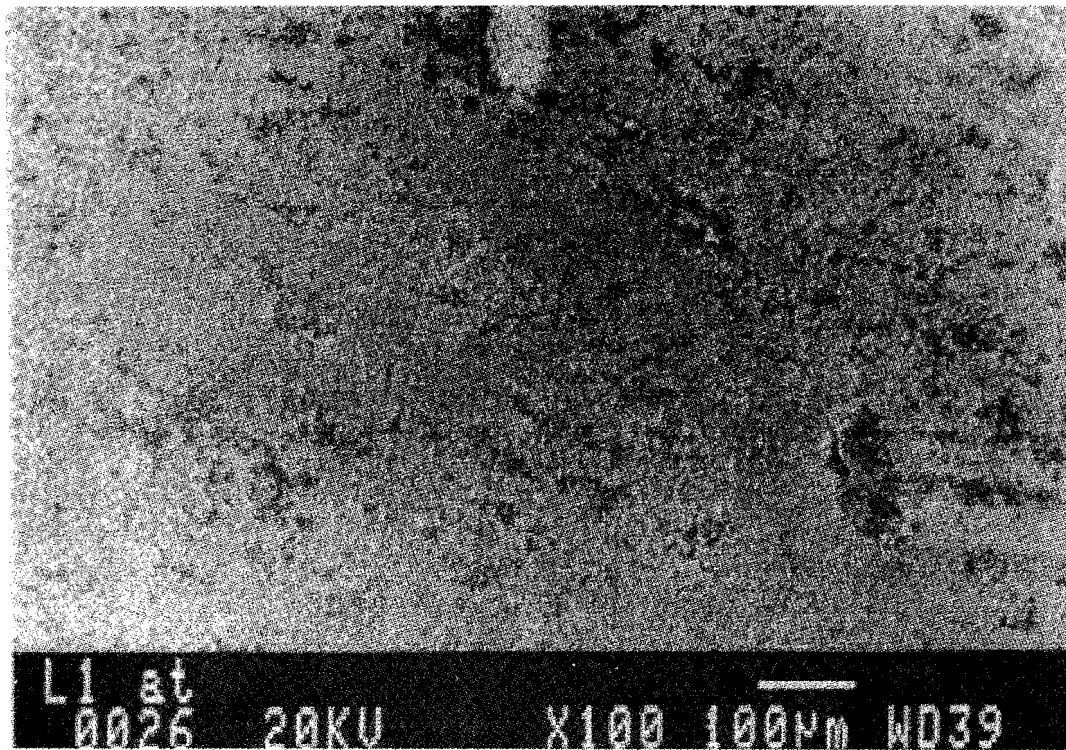


Plate L1. Titanium Ti Grade 12. After the test

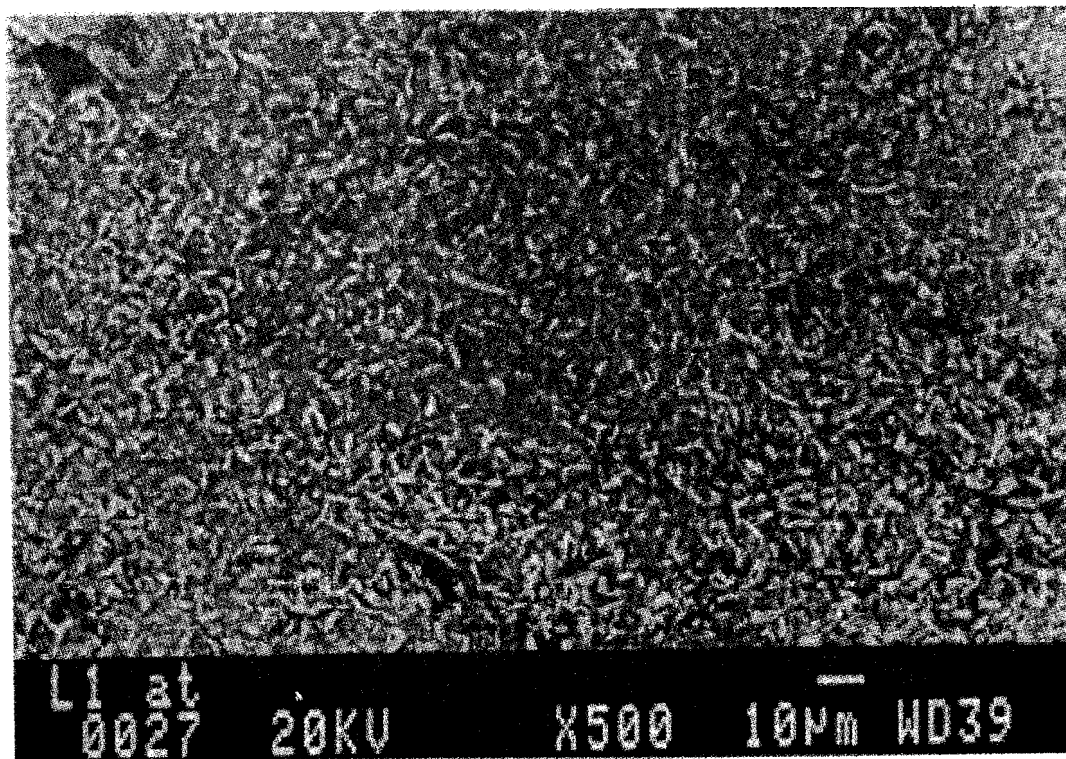
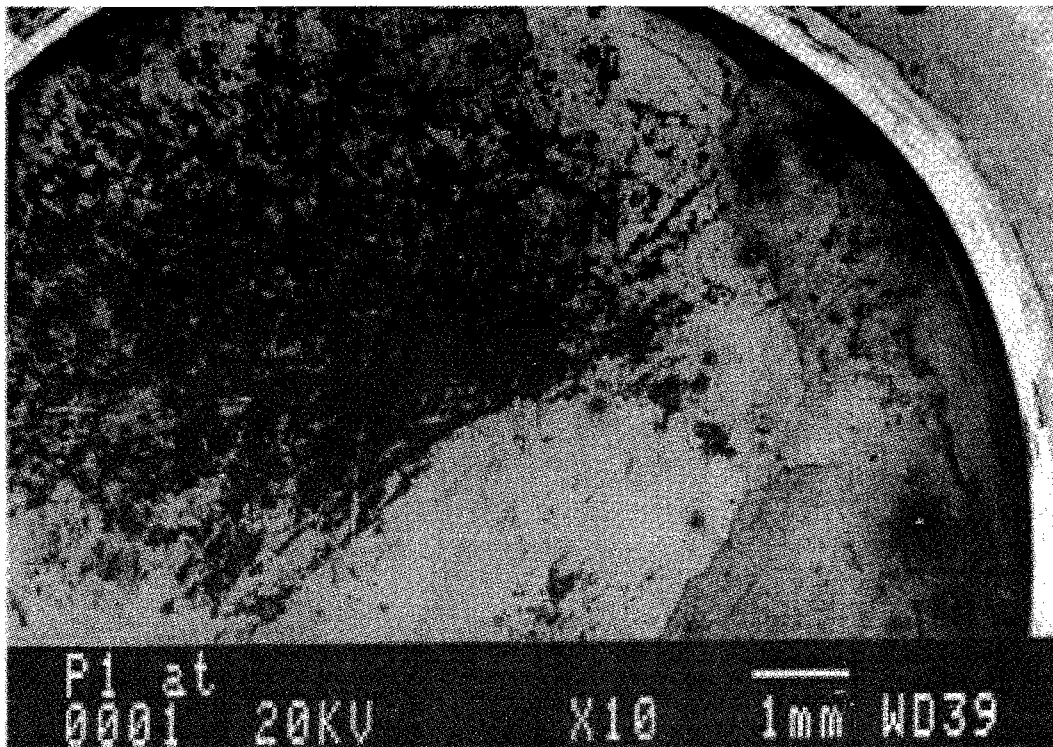
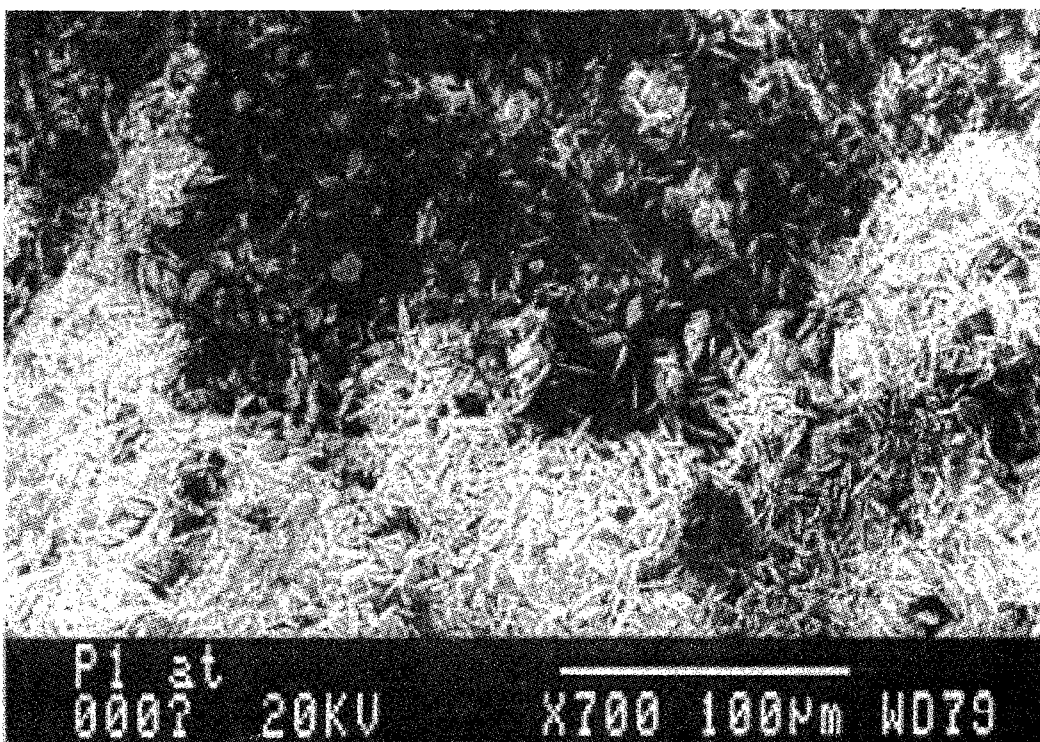


Plate L2. Titanium Grade 12. Corrosion coating on the surface is clearly visible.





*Plate P1.* Titanium Ti 21S. At low magnification, the poor test surface finish is already apparent.



*Plate P2.* Titanium Ti 21S. The dark areas are surface deposits from the reaction fluid. The light areas are corroded metal.



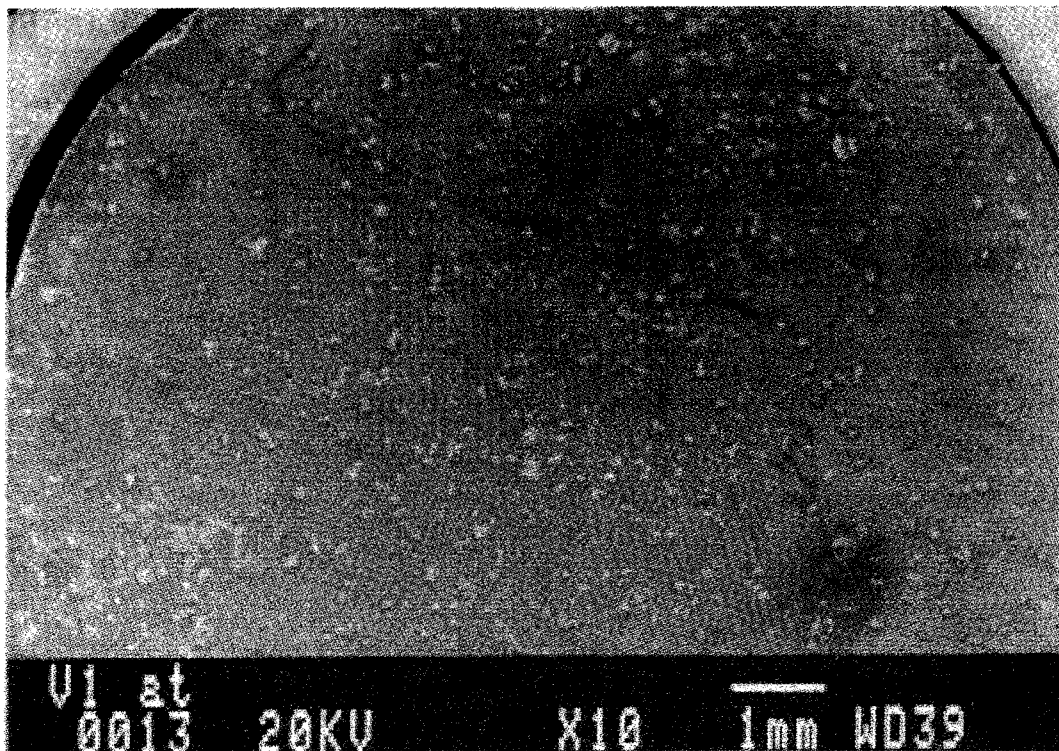


Plate V1. Stainless Steel S316. Pitted zones on the post test surface.

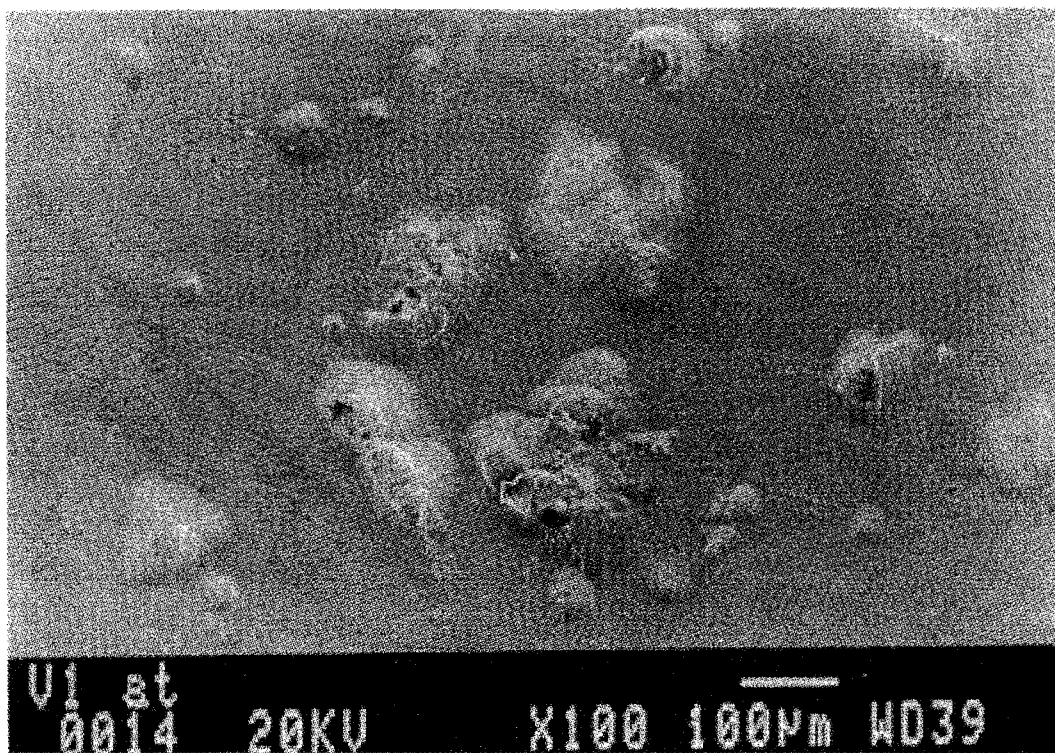


Plate V2. Stainless Steel S316. The corroded areas at closer magnification.