



RECENT DEVELOPMENTS AND PRESENT STATUS  
OF SHIELDED  
MECHANICAL AND PHYSICAL TESTING FACILITIES  
AT THE NETHERLANDS ENERGY RESEARCH FOUNDATION,  
ECN - PETTEN

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P R E L I M I N A R Y

C O P Y

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ABSTRACT

The present hot cell facilities for mechanical testing was initiated some 30 years ago. From that time onward the facilities expanded in connection with ECN's extensive mechanical testing programmes and physical experiments on irradiated materials. Recently, this resulted in the implemented facilities for physical measurements and welding experiments.

Further, the existing creep testing facility has been improved and a fully instrumented Charpy impact testing machine has been installed in a reconstructed lead cell.

These upgrades are intended to support all phases of the experimental work and have improved the capabilities of the testing laboratory.

This paper describes some of the improvements and emphasizes on the recent developments of the shielded facilities.

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## 1. INTRODUCTION

The ECN testing laboratory carries out research programmes for national and foreign customers.

In the last two decades mechanical testing on irradiated materials has been carried out with emphasis on advanced testing techniques such as low cycle fatigue, creep, and fracture mechanics. Recently, the determination of physical properties and the weldability of irradiated materials has become of interest.

As a consequence, new experimental facilities for the determination of physical properties have been set up. With these facilities physical measurements, like thermal conductivity, thermal diffusivity, and density can be performed. For the determination of some physical properties an advanced laser system is used, with which also welding experiments on irradiated materials can be performed.

A facility to perform annealing and heat treatments on irradiated materials is also available. For this purpose a high temperature vacuum furnace will be installed in one of the hot cells.

The laboratory has close relation with the Joint Research Centre of the European Communities. Recently, a joint facility, the European Reference Laboratory has been established to serve the European Networks on Structural Integrity and Materials Aging [1,2].

The last survey with respect to the hot cell facilities for mechanical testing was given for this forum in 1988 during a meeting in Jülich, Germany [3], whereas an extensive description of the creep testing facility was given in Brasimone, Italy [4].

## 2. AVAILABLE TESTING FACILITIES (Present Status)

### 2.1. General Summary

At present the available mechanical and physical testing facilities for irradiated materials at ECN allow for the following experiments and measurements:

- determination of mechanical properties like
  - creep;
  - tensile;
  - low cycle fatigue (LCF);
  - fatigue crack propagation (FCP);
  - fracture mechanics (FTT);
  - charpy impact;
- determination of physical properties like
  - thermal diffusivity;
  - thermal expansion;
  - density/swelling measurements;
- reweldability and reconstruction of irradiated samples;
- in-cell specimen measuring device;
- annealing experiments on irradiated materials.

The shielded medium activity facilities available for this purpose consists of:

- three cells, each equipped with electro mechanical testing systems to perform the tensile and low cycle fatigue experiments;
- two cells, instrumented with the laser system for physical testing and welding experiments, including a high-vacuum/high-temperature chamber;
- a creep testing facility with nine creep testing machines;
- three cells for fatigue crack propagation and fracture toughness testing experiments and charpy testing;
- a high-activity cell in which the specimen measuring device and annealing furnace is installed.

Next to these facilities laboratories are available for research of un-irradiated materials.

In the following sections of this paper an impression of the recent developments in the hot cell laboratory is given with emphasis on the latest technical evaluations for mechanical testing and the facility for physical measurements.

### 3. MECHANICAL TESTING (Recent Developments)

#### 3.1. Creep Testing Facility

After every three years of operation a maintenance and servicing period is carried out for the creep testing facility. The main objective is to decontaminate and to overhaul in-cell equipment. At the same time components can be replaced and repaired, whereas newly developed techniques can also be built in. In this period all in-cell active materials are taken out and the facility is decontaminated [4].

During the last overhaul, specific improvements are introduced. Improvement was reached by introducing a movable door on one of the faces of the cell to provide a better accessibility. The dimensions of the door allows personnel to enter the cell using a complete pressure suit. In figures 1 the former and the present situation respectively is given of the creep testing cell.

A second facility consisting of three creep testing machines was decommissioned and reconstructed again to accomodate a small instrumented Charpy impact tester.

The design and construction of this new facility, as well as the technical requirements were specified by the operators. Next to these requirements specifications according to testing standards had to be met as well.

#### 3.2. Charpy Testing Facility

An INSTRON-Wolpert Charpy impact testing machine has been installed in a medium-activity cell. In conjunction with this system a specimen positioning and conditioning module manufactured by Severn Furnace Ltd (SFL) has been designed to accomodate miniature notched specimens.

Positioning of the specimen in the test area is done accurately, without the operator having to use manipulators or tongs.

The conditioning system is an integrated heating, cooling system to condition specimens to the required temperature between  $-180^{\circ}\text{C}$  and  $+500^{\circ}\text{C}$ .

Heating is achieved by cartridge heaters inside the furnace, whereas cooling is done by liquid nitrogen, supplied from a vessel.

The conditioning system consists of a Eurotherm 902 temperature controller, which controls both the heating elements as well as the nitrogen cooling. The nitrogen cooling is activated through a 24 V dc solenoid valve and triggered via an output inside the controller. From the cooling system dry nitrogen can be bypassed to minimize the chance of condensation in the conditioning system when operating at cryogenic temperatures. The system is supplied with 4 type K thermocouples. Only two are used, one for temperature control and one for the alarm unit. The other two are provided as spare in case of breakdown. A faulty thermocouple can simply be unplugged and replaced by plugging the connector into an unused thermocouple connection.

The sequence for transfer and temperature control is achieved by a PLC. The PLC is interfaced with three stepper motors via suitable drive hardware. The system layout is given in figure 2.

The control console is situated outside the cell and ensures that the specimen is transferred from the conditioning module to the hammer within less than 3 seconds. Extensive alarms are provided together with logic connection to the control console. This is to ensure that the specimen temperature is within the set limits, that the transfer is done in the set period, and that the hammer swing has been cleared.

The Charpy impact test consists of breaking by one blow from a swinging pendulum, under specific conditions, a test piece notched in the middle and supported at each end. With the described system 3x4x27 mm specimens can be tested. The testing machine is additionally instrumented to determine force or energy-displacement curves. Direct analog reading of the impact energy from a dial gauge is also possible.

Force measurement is usually achieved by using two active strain gauges, attached to the standard tup to form a loadcell. Displacement measurement is normally determined from force-time measurement. The force-time relationship measured on the tup is proportional to the acceleration characteristic.

Recording of the dynamic signals is achieved digitally. For this purpose a TULIP DT80 PC, including an INSTRON-Wolpert software package is used to measure and to collect the load signals. After processing both the force-energy/displacement curves as well as the maximum energy values the relevant data are calculated and presented on screen. The relevant data can also be printed and stored on disk.

An example of a recording of the force-displacement and energy-displacement curves is given in figure 3.

#### 4. WELDING EXPERIMENTS AND PHYSICAL PROPERTIES

##### 4.1. Reweldability and reconstruction of irradiated samples

During the life of a nuclear power plant certain components have to be replaced. Replacement of irradiated components requires that irradiated materials have to be welded. Welding of irradiated austenitic stainless steels might cause high temperature induced cracks due to the presence of helium in the irradiated steel. The weldability of irradiated structural steels is being studied by using laser welding techniques followed by metallographic and mechanical examination.

For this purpose a laser system has been installed in a hot cell, which is shown schematically in figure 4. The present set up allows for welding of flat specimens and modifications are planned to weld cylindrical specimens and tubes as well.

Precautions are made to prevent contamination of the cell by volatile and aerosole welding products. In figure 5 a weldment of a 1 mm thick irradiated and unirradiated plate is shown.

Part of the facility concerns an industrial Nd:YAG laser with the following specifications.

Laser system	: LASAG KLS-311/321
Laser medium / wavelength	: Nd:YAG / 1.064 $\mu\text{m}$
Pulse duration	: 0.1 - 20 ms
Pulse energy (max)	: 3 - 70 J (0.2-10 ms); 30 J (20 ms)
Peak pulse power	: 6 - 10 kW
Maximum average laser power	: 260 W
Beam diameter	: 6 - 12 mm (unfocused)
Applicable peak power density	: $\leq 50 \text{ GW/m}^2$
Applicable peak energy density	: $\leq 100 \text{ MJ/m}^2$

The laser beam can be connected to several experimental set ups for different applications such as:

- the measurement of thermal diffusivity (4.2), through an optical fiber to an in-cell set up;
- thermal shock testing (4.3), directly to a vacuum chamber;
- welding experiments, through an optical fiber to an in-cell welding equipment.

##### 4.2. Thermal diffusivity

The determination of the thermal conductivity coefficient at high temperatures is difficult to perform and requires large material samples. For special materials this can be troublesome. To overcome this drawback the laser-flash method is used to determine this parameter. Since the density and specific heat of materials are well known the diffusivity coefficient can be used for the indirect determination of the thermal conductivity coefficient.

With the laser-flash method the plane side of a sample receives a short energy pulse, generated by a pulsed-laser system or a flashlight. From the increase in temperature at the backside of the sample the diffusivity coefficient can be calculated. In figures 6 and 7 the principle of the laser-flash method to determine the thermal diffusivity property and the sample holder is shown respectively.



This method has some major advantages e.g.:

- it is a short term measurement;
- direct measurement of the coefficient;
- it is applicable:
  - in a wide temperature range;
  - for materials with diverse physical properties;
  - for small size samples.

For the ECN facilities inside the Hot Cell Laboratory a TEKTRONIX TDS-420 digital oscilloscope is used for data-acquisition and data-processing and analysis is performed by DADISP (DSP software) on a TULIP 486 computer. For temperatures up to 750°C a Type K open thermocouple is used for measuring the temperature transient.

#### 4.3. Thermal expansion

An experimental high vacuum set up has been built up for thermal shock testing of candidate protection materials for plasma facing components in fusion devices. Thermal shocks are generated by the LASAG laser.

For proper quantification of energy deposition the measurement of the optical absorption is possible either direct by in-situ calorimetry or indirect with a separate set up for reflectivity measurements using a small Nd:YAG laser. The material response to pulsed energy deposition can be quantified by measuring the surface temperature. A customized fast pyrometer (KLEIBER 270B), with integrated CCD camera, is available with a measuring range of 525-2525°C or 575-3225°C, depending on the material spectral emissivity. This emissivity can be preset between 0.1 and 1.0 when known, or measured by preheating the specimen in the measuring range. The measuring spot size is about 0.8 mm in diameter and the response time is better than 10  $\mu$ s (full scale).

A Quadrupole Mass Spectrometer is integrated in the system. Species originating from the area of laser pulse impact can be detected in a mass range of 1-200 m/e with a maximum scanrate of 2 Hz.

#### 4.4. Density/Swelling Measurements

Materials irradiated with neutrons show dimensional changes due to lattice defects or gaseous transmutation products or both. Measurement of dimensional changes can be performed by several means, like measurement of the specimen volume or measurement of actual dimensions, the latter giving the advantage to discriminate geometry or anisotropy effects.

The specimen volume can be obtained by immersion in a liquid and measure the reduction of specimen weight. An in-cell facility is available using a METTLER AT 264 micro-balance, with a range of 22 g and an accuracy of 10  $\mu$ g. The immersion can be in either demineralized water or ethylalcohol, depending on the surface condition of the specimen. As the density of the liquid is a main factor in the measurement, its temperature can be measured to within 0.2°C accuracy.

With the present set up the determination of the material density was demonstrated with an accuracy of 0.015 g/cc, for metallic specimens of a few cc.

The complications of liquid immersion may be avoided by a more direct volume measurement using a helium pycnometer. The possibilities of such equipment for application in a hot cell environment are investigated.

## 5. MISCELLANEOUS

### 5.1. Specimen Measuring Device

For the measurements of crack length and elongation after fracture of tested samples a measuring device has been designed and installed in one of the high-activity cells. This instrument consists of a X-Y table which is fully computer controlled. Both compact tension (CT) as well as cylindrical specimens can be measured.

The average crack length of the CT samples is determined using the 9-point measuring method. The average elongation of cylindrical specimens is measured at two faces, namely at 0 and 90°. From these values the averaged is calculated.

Measurements and calculations are done with the software programme MEAS, developed by ECN-Facilities Unit, which also controls the X-Y table.

After a series of measurements the data are stored and a data report can be printed out. Another software package is used to extract data from the MEAS data base for the final data analysis.

Connected to this measuring device is a camera set up which can display the sample parts on a high resolution colour screen, including crosswires. In combination with a periscope the parts are magnified, and the right reference points can be fixed.

The images can be stored in two ways, namely:

1. As a picture. On the unit a video printer is connected, which can produce directly a colour picture from the camera signal. An example is given in figure 8.
2. As a digital data base. A video digitizer interface transforms the camera signal into digital data, after which it is stored and can be shown on any PC at any moment.

### 5.2. Annealing Furnace

A LINN High Therm GmbH furnace is available for temporary installation in one of the high-activity cells to perform annealing experiments on irradiated materials. The gastight metal muffle allows operation under gas-atmospheres as well as under low-pressures ( $10^{-5}$  mbar). The furnace chamber is made from a heat-resistant alloy. The door is watercooled and provided with a door plug with radiation plates and an inlet and outlet for protective gas. The door locking system has been adjusted for remote handling and based on the principle of threaded bolts and springs to intercept explosions.

The heating is achieved from four sides using Kanthal heating elements embedded into the fiber muffle. Temperature control is done with a Eurotherm 902P programmable controller, whereas the temperature measurement is done by Type K thermocouples.

In summary the specifications are:

Chamber inner dimensions: 180x240x110 mm (WidthxDepthxHeight);

Chamber outer dimensions: 450x640x530 mm (WxDxH);

Service temperature : max. 500°C under vacuum;

: max. 1050°C under protective gas;

Temperature accuracy : within 5°C;

H<sub>2</sub>O/min. : 1.0 liter;

Vacuum :  $10^{-5}$  mbar;

Heating volume : approximately 7 liters

tion.

6. REFERENCES

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7. FIGURES

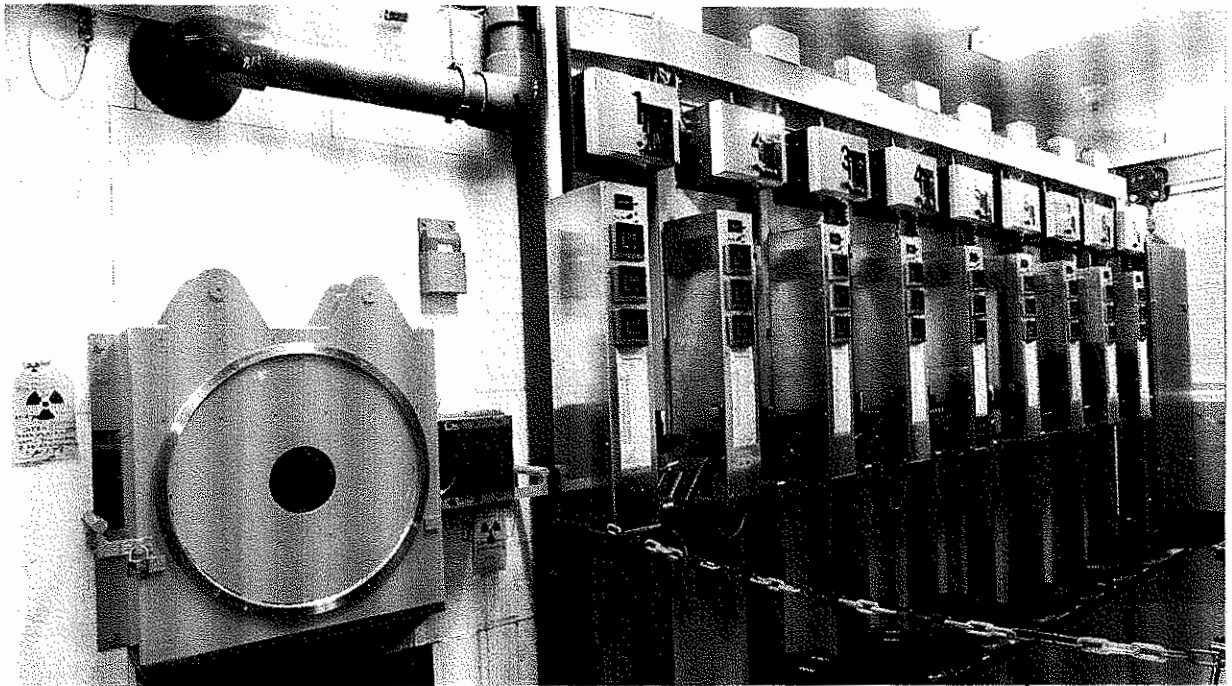
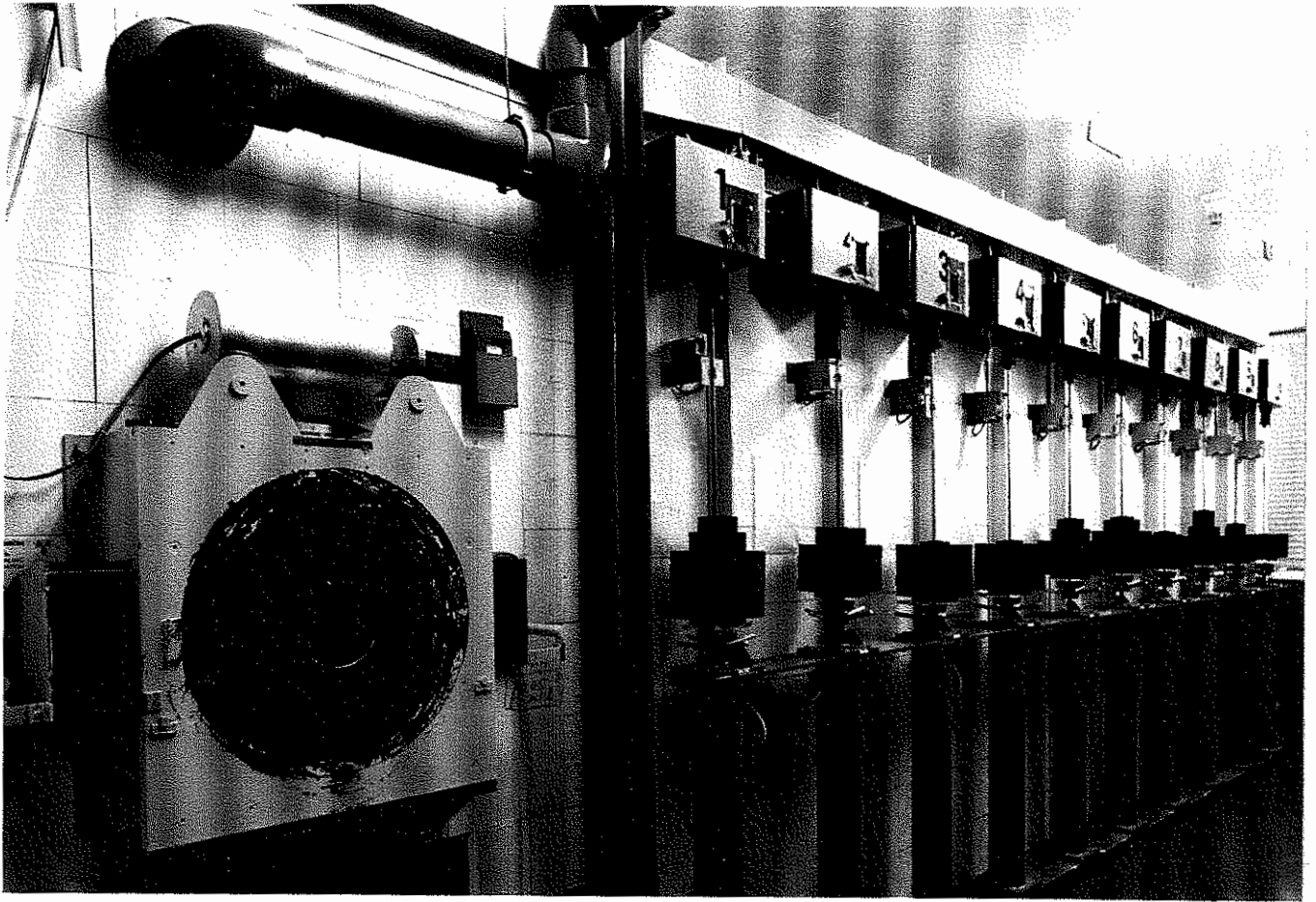


Figure 1 Former situation (above) and present (below) situation of creep testing facility

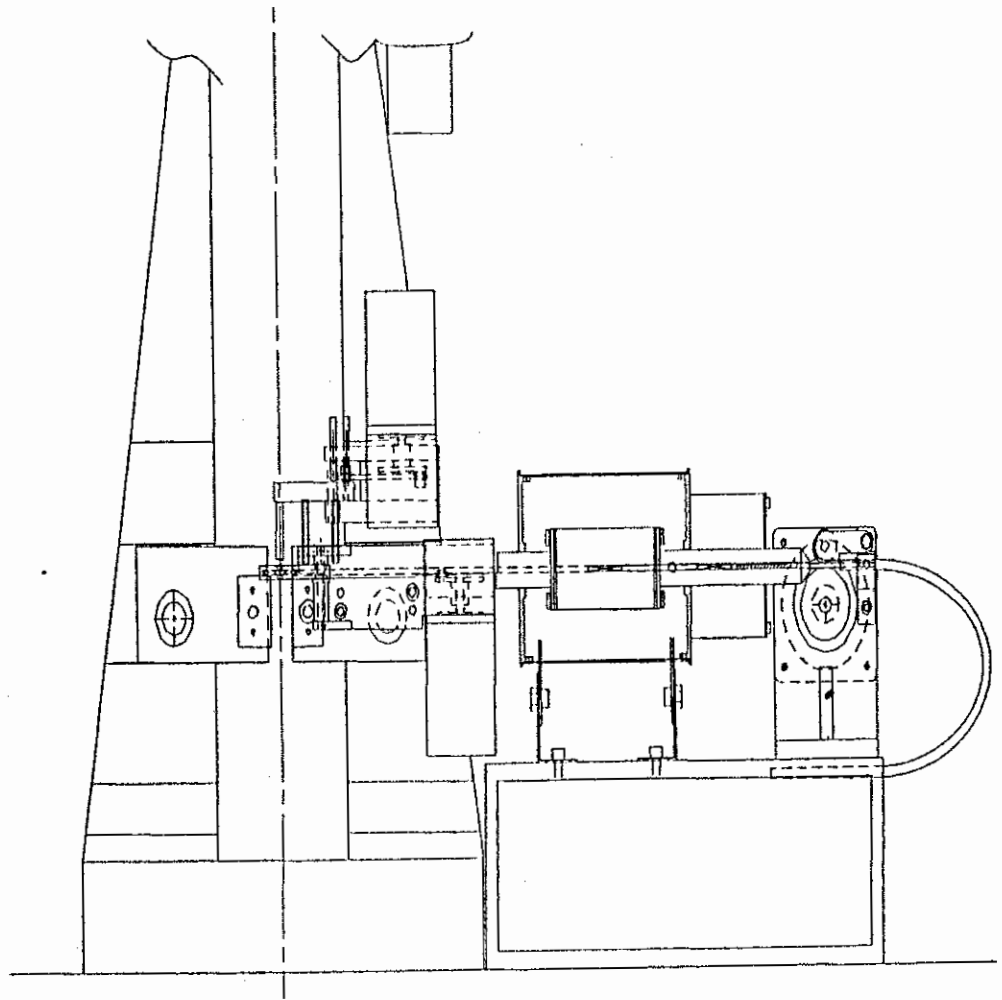


Figure 2 System layout of instrumented Charpy impact tester

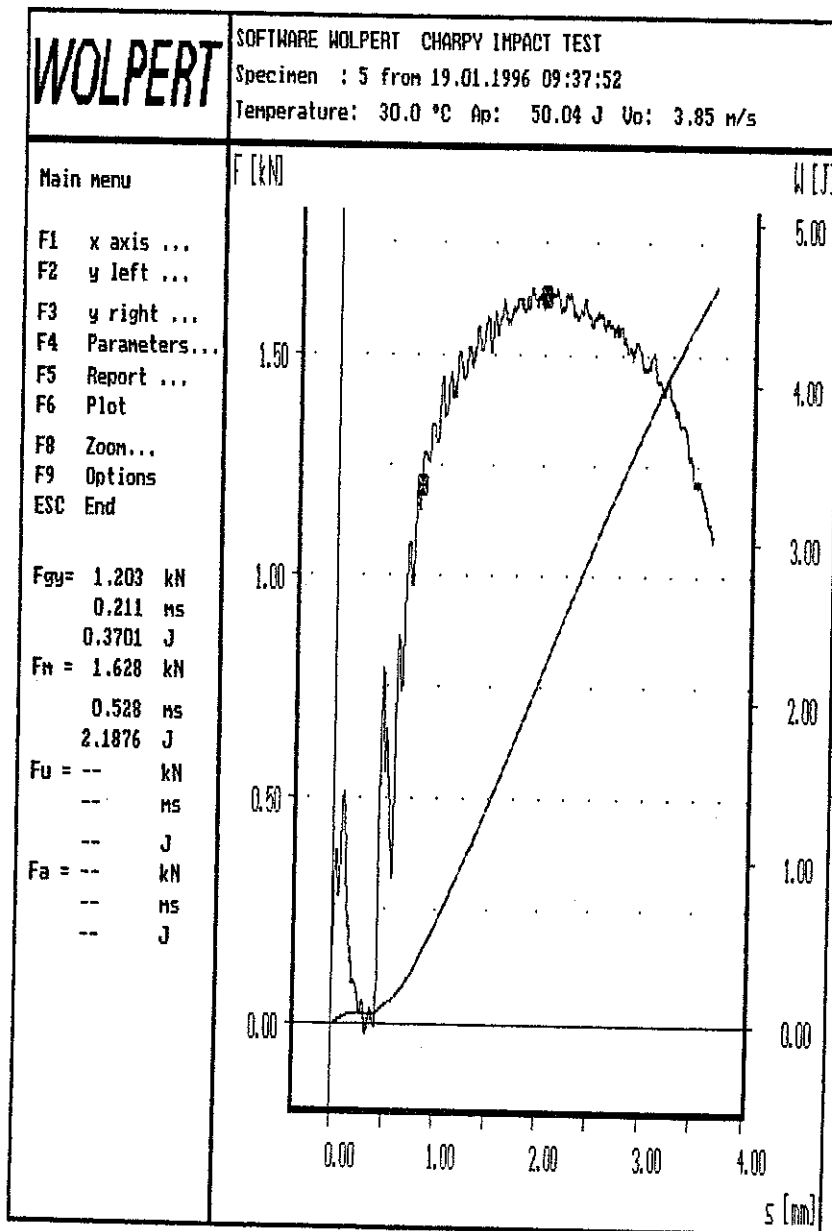


Figure 3 Example of a recording of force-displacement and energy-displacement curves

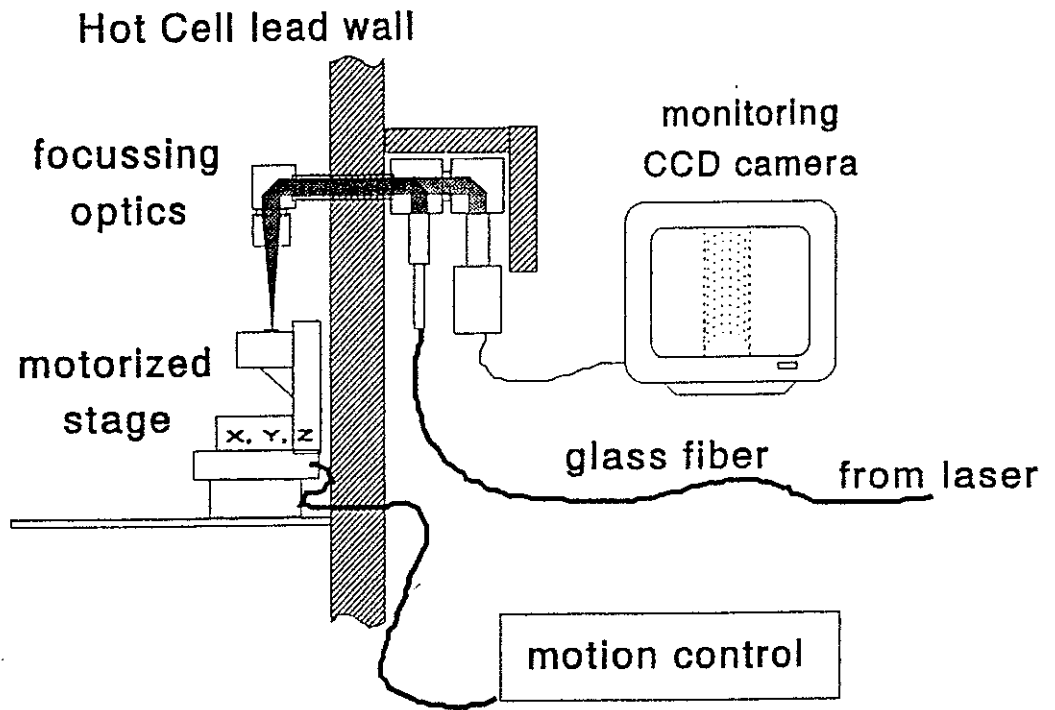


Figure 4 Layout of shielded laser facility

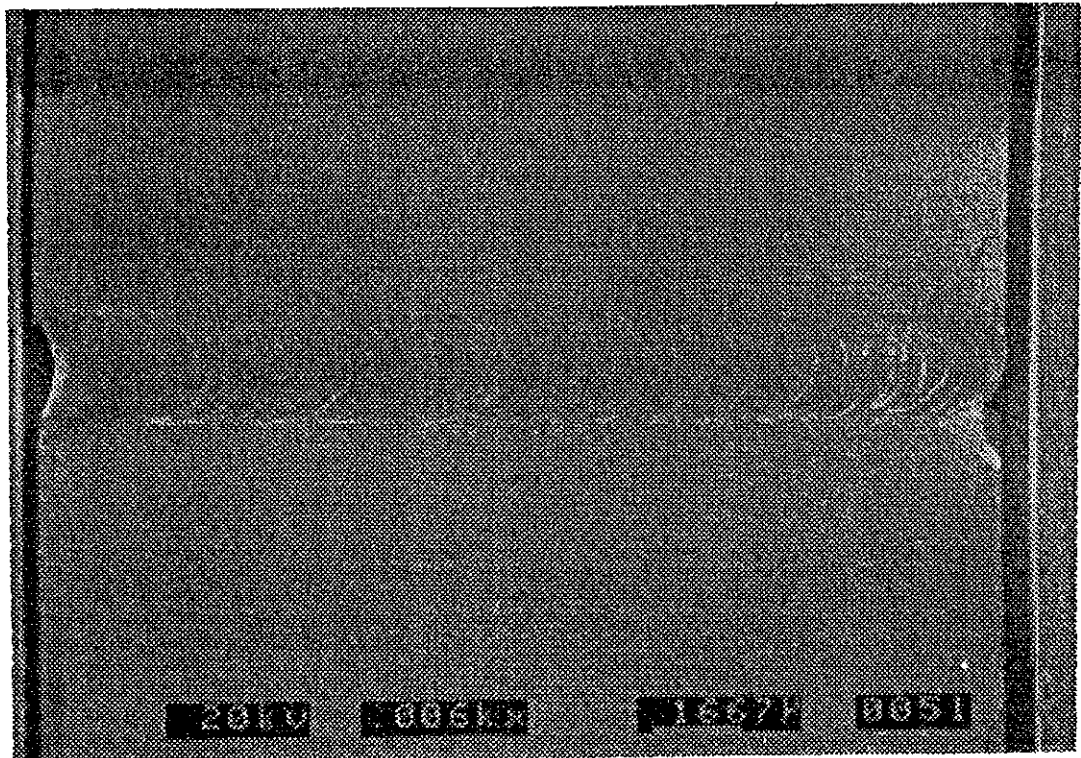


Figure 5 Laser weldment of irradiated (5dpa) and unirradiated SS 316LN, 1 mm thick

## Thermal Diffusivity by Laser-Flash Method

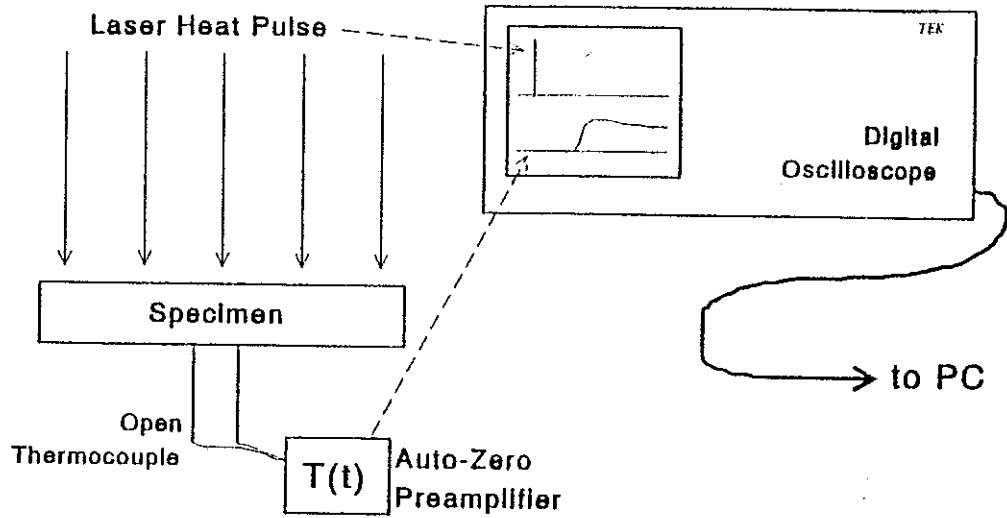


Figure 6 Principle of laser flash method to determine the thermal diffusivity property

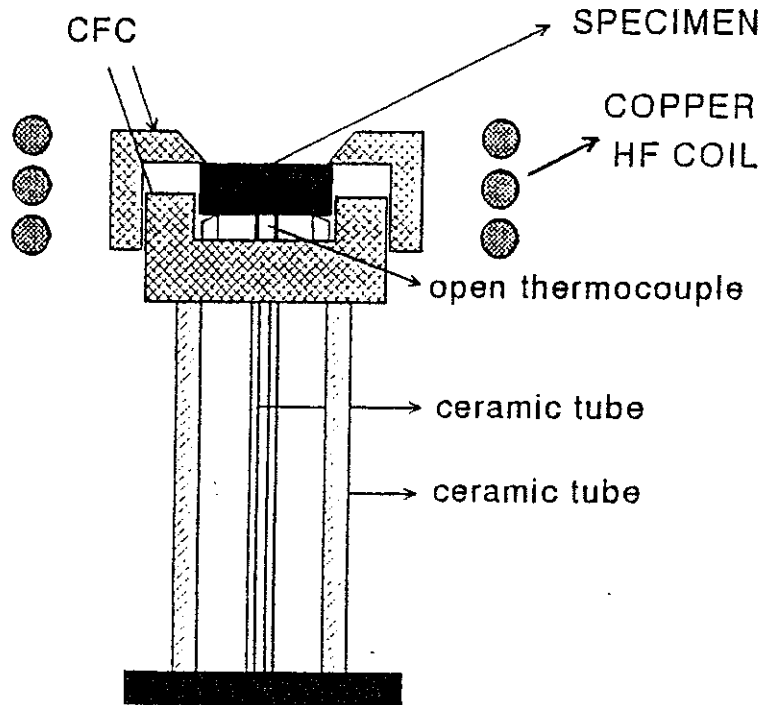


Figure 7 Sample holder for thermal diffusivity measurements



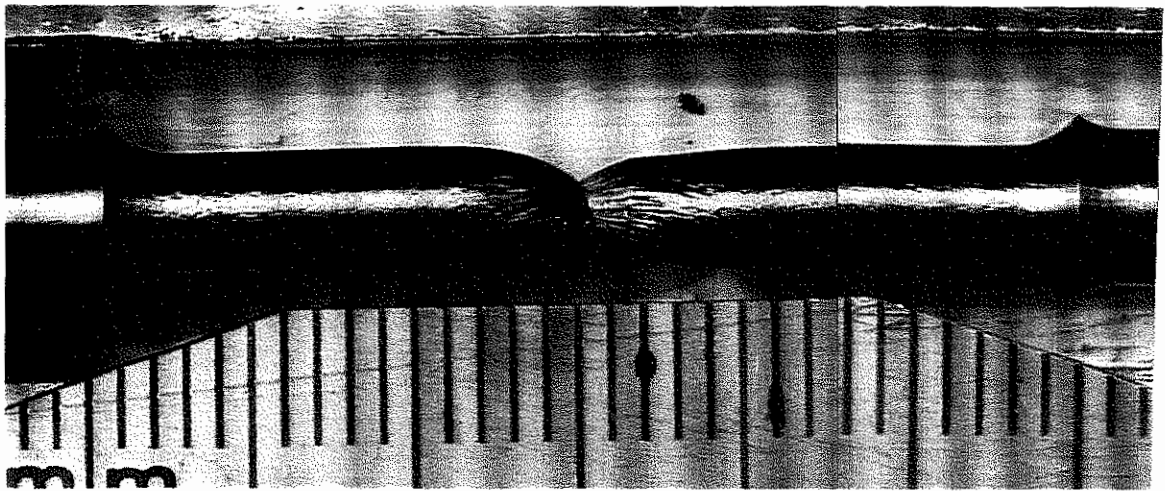
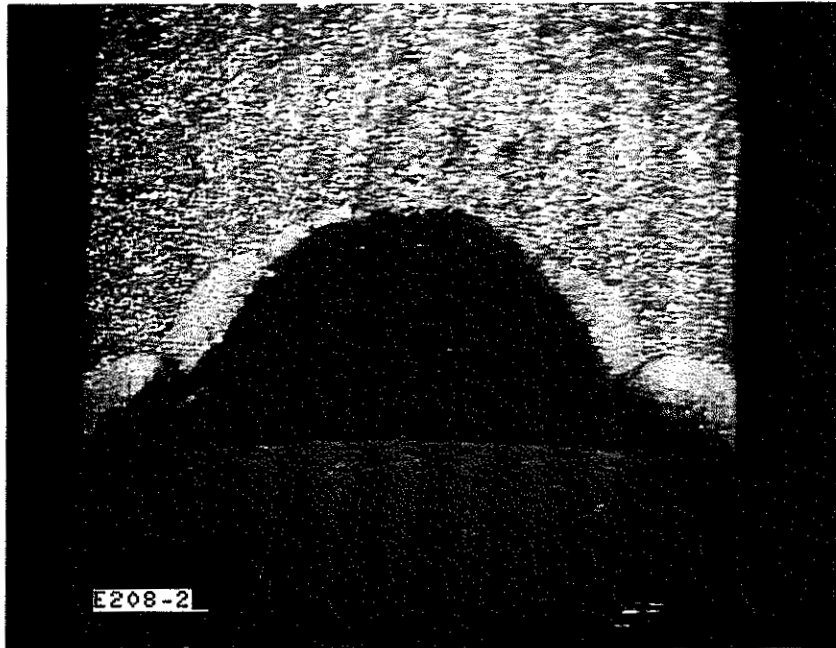


Figure 8 Above: Fracture surface of a compact tension specimen after fracture toughness testing;  
Below: Picture of tensile specimen to calculate total elongation and reduction of area;  
Both pictures produced by video printer.