Examples of the use of small specimens in irradiation testing of fusion structural materials

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Petten, 1 October 1999

K5071/99.28349/I

Paper to be presented at European Working Group "Hot Laboratories and Remote Handling" Plenary Meeting, 13-15 October 1999 at European Institute for Transuranium Elements - Karlsruhe, Germany

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12 page(s) MMI/GLT/NO i28349	approved : B. van der Schaaf

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1 Introduction

The tendency to use small size specimens in nuclear materials research has started many years ago. The driving force for the use of small specimens originates from:

1. restrictions on specimen dimensions dictated by irradiation facilities, for example effective irradiation volume in beam experiments, thermal and neutron gradients in reactor experiments, irradiation space and efficiency;
2. reduction of waste, because of the increasing costs involved;
3. limitations on material availability and on dimensions of product forms.

Certain materials properties do not allow an unlimited decrease in size. Issues like lack of constraint or specimen sizes that approach the grain size of the material do not allow an unlimited reduction in dimensions. The nuclear materials research, where the relative effects of radiation on materials properties is assessed, allows a certain reduction of specimen size because the properties will be measured on the same specimen geometry before and after irradiation. For absolute measurements there are more stringent limits in miniaturisation.

Reduction of specimen dimensions provides for efficient use of material volume and irradiation space. As an example the volume of a standard (10x10x55 mm) Charpy impact specimen is 16 times the volume of a miniature KLST type sample (3x4x27 mm). Considering the number of about 15 specimens for the determination of a Charpy transition curve, the efficiency is clearly demonstrated.

Over the years various types of small size specimens were used at NRG to investigate a wide range of mechanical properties. Typical small size specimen types ranging from tensile, creep, (low cycle) fatigue specimens, Charpy impact samples and Compact Tension (CT) specimens are used.

With these specimens the following experiments are performed:

- tensile tests;
- creep tests;
- low cycle fatigue (LCF) experiments;
- crack propagation and fracture toughness experiments;
- impact tests.

As a consequence, the testing techniques as well as the remote handling capability for testing small size specimens have to be considered.

This paper gives some examples of the use of small size specimens and the experimental facilities for testing these specimens.

2 Experimental

2.1 Material

Various materials have been investigated in the frame of the Fusion Technology Programme, ranging from austenitic stainless steels to Vanadium alloys and Reduced Activation Ferritic Martensitic (RAFM) Steels. Of these materials, a variety of small size samples have been manufactured.

For Low Cycle Fatigue (LCF) experiments on welded materials with limited thickness, samples are used in accordance with figure 1a.

The maximum plate thickness of the available Vanadium alloy was ¼", so that for tensile tests and Charpy experiments small size samples had to be used as shown in figures 1b and 1c.

The austenitic stainless steels were available in various product forms of which the maximum plate thickness was 40 mm. Yet, the need to use small size samples primarily arose from the limitations of the irradiation capsules and the irradiation conditions. The conditions to realise a relatively high neutron dose of 10 dpa in a short period meant that HFR positions had to be used with the highest dose rate, and the highest γ-heating. To achieve homogeneous temperature and neutron conditions the sample dimensions had to be as small as possible.

For the elasto-plastic fracture mechanic experiments (J-tests), 2.5-mm CT specimens are used as given in figure 1e. An additional limitation for the austenitic stainless steels is the high radioactivity of the irradiated material. This limitation is less relevant for the RAFM materials, so that thicker specimens can be used like the one in figure 1d.

2.2 Specimens

The various types of samples and their dimensions are given in figure 1.

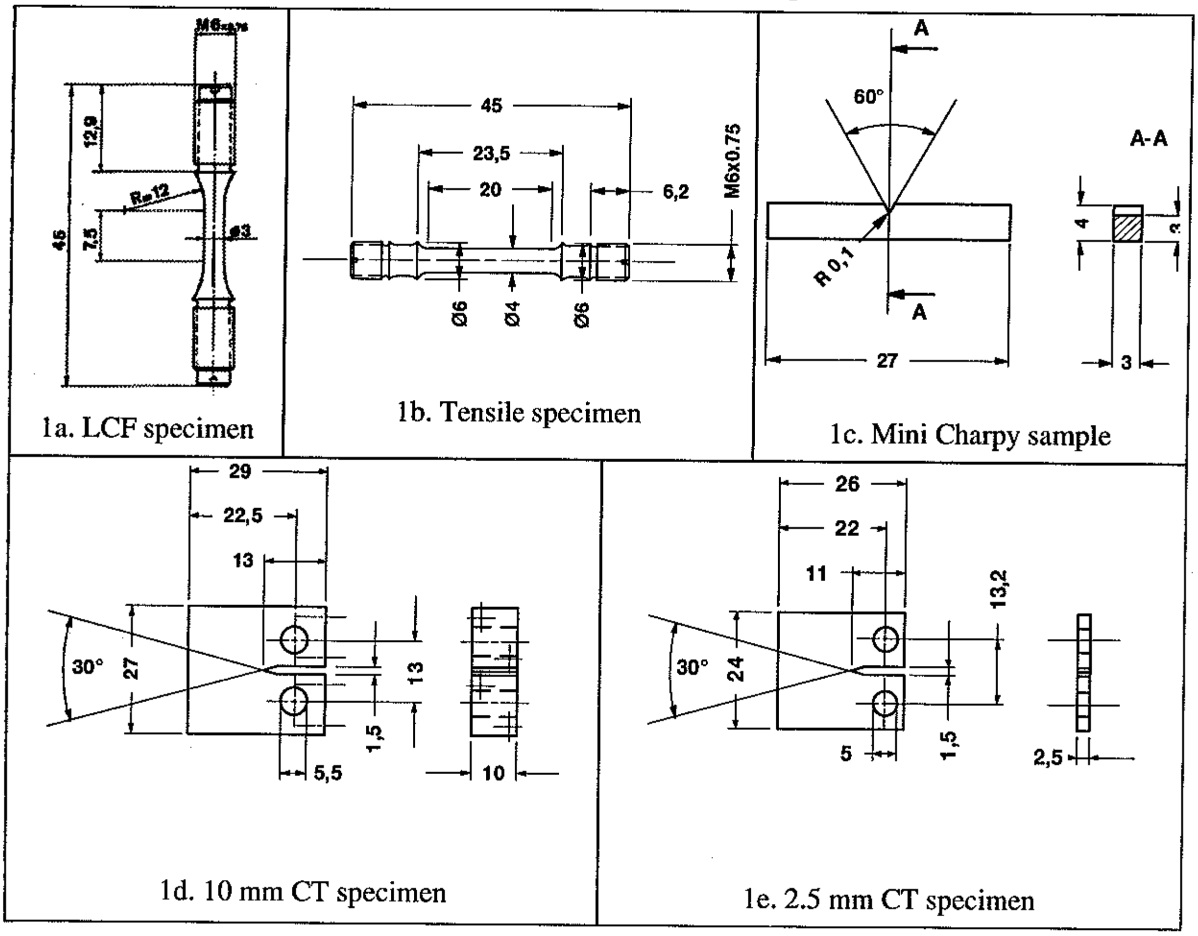


figure 1 Review of some small size specimens

All specimens are taken in the T-L orientation with respect to the rolling direction of the plate. The orientation code is given in figure 2.

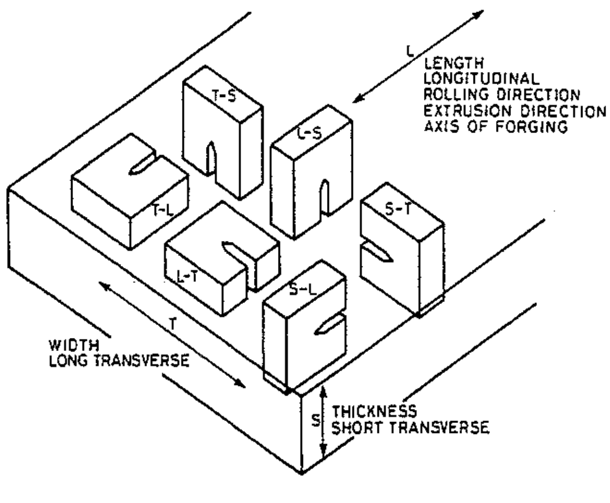


figure 2 Schematic of crack plane orientation code for rectangular sections

2.3 Irradiation experiments

Depending on the type of irradiation experiment (temperature and dose) and the type of specimens involved, a specific irradiation capsule is designed. In the given examples three types of capsules are used, namely the so-called ILAS, MINOSSE and CHARIOT capsule. Each capsule is specifically designed to accommodate the specific specimen type.

The ILAS (**I**rradiation of **L**ow **A**ctivation **S**pecimens) capsule contains 56 tensile specimens, which are irradiated at 325°C at a target dose of 6 dpa. (see figure 3)

The MINOSSE (**MIN**iaturised c**O**mpact Ten**S**ion Sp**E**cimens Irradiation) capsule permits the loading of thirty 2.5-mm CT specimens, which are placed in two stacks. The irradiation temperature is 325°C and the target dose is 10 dpa.

The irradiation temperature of the CHARIOT (**CHAR**py and compact tens**IO**n specimens irradia**T**ion) capsule is 300°C with a target dose of 2.5 dpa and contains 48-sub size Charpy samples and thirteen 10-mm thick CT samples.

The temperature of the specimens is controlled by a vertical displacement unit and by individual gas mixtures regulating the heat transfer between the specimens and coolant. The temperature distribution inside the specimen holder is measured by means of type K thermocouples.

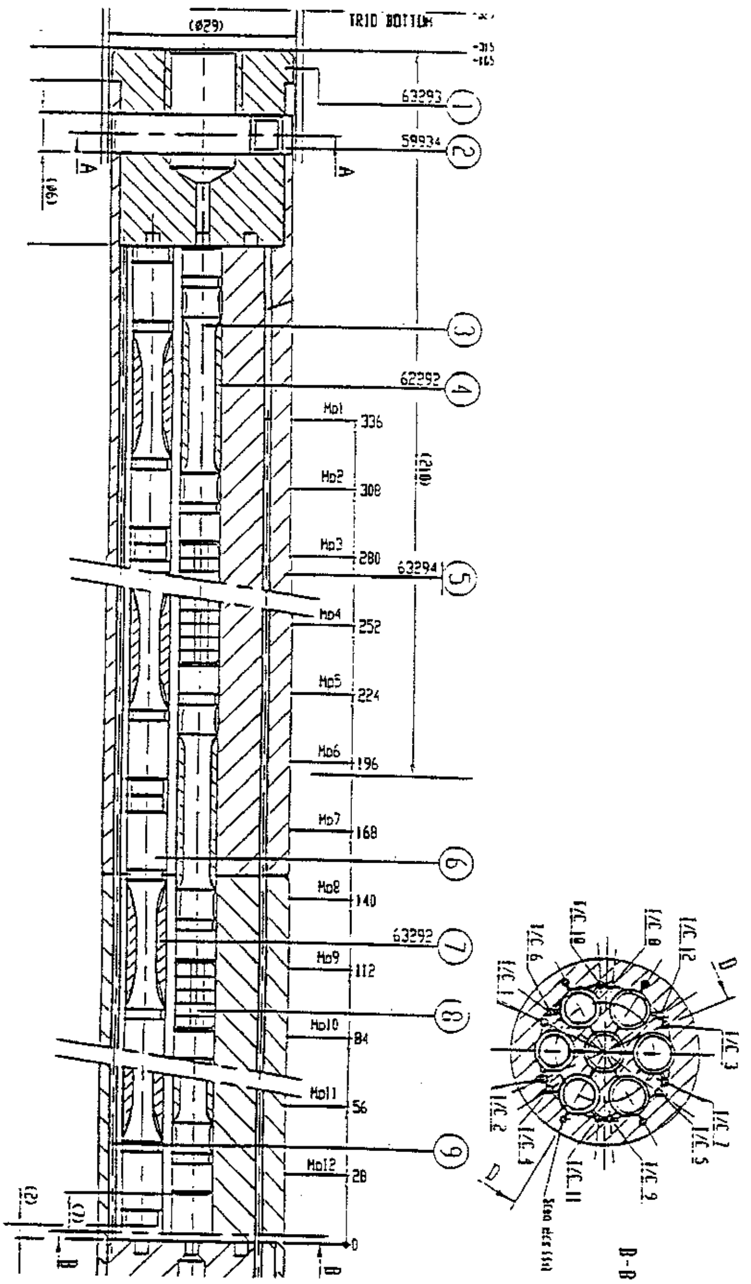


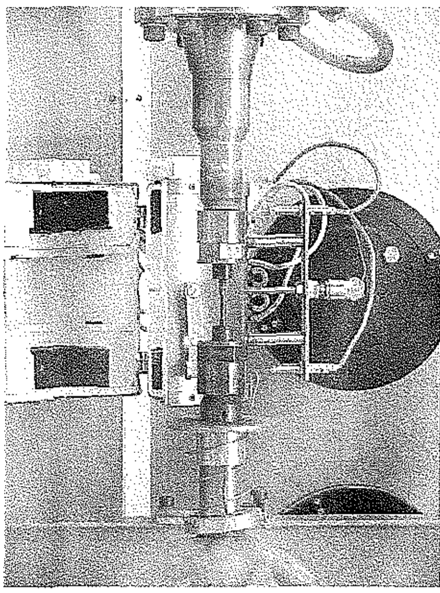
figure 3 Schematic of the ILAS capsule

2.4 Experimental procedure

All mechanical testing experiments are performed in the Hot Cells Laboratories. Tests for the fusion technology program are performed at temperatures from RT up to 425°C in an air environment, with the exception of the Charpy impact tests, which can be performed in the range of −180 to +300°C. The cryogenic temperatures are achieved by using liquid nitrogen.

Heating up/cooling down and stabilising of the temperature can take up to one and a half-hour. A computer records the specific test parameters (e.g. tensile curves) during the tests. Broken specimens can be measured optically after testing to determine the characteristic properties, like total elongation, reduction of area, or lateral expansion of the Charpy impact specimens, whereas fracture surfaces can be analysed to measure the real crack lengths. A more detailed description of the procedures is given in the next chapter.

An impression of the facilities for tensile testing and for fracture mechanics is given in figures 4 and 5 respectively.



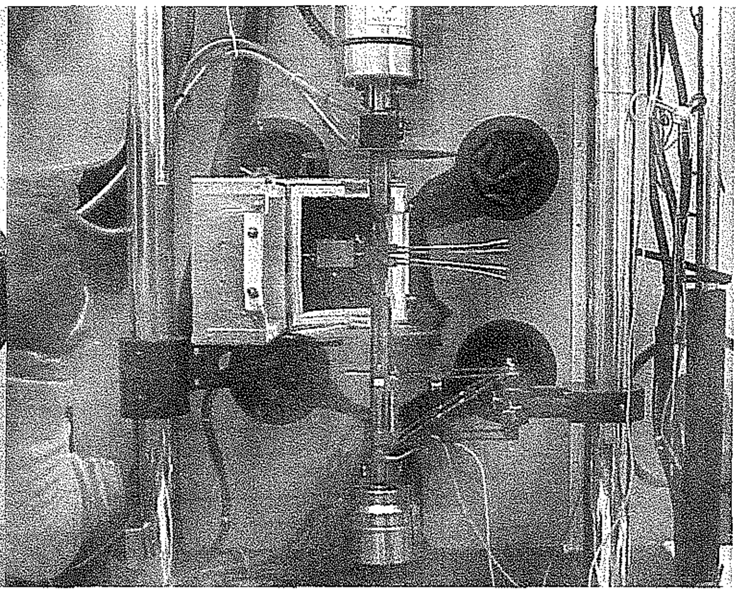


figure 4 Tensile testing assembly   figure 5 Set up for fracture resistance experiments

2.5 Remote handling small size samples

To load small size samples in a testing machine under remote controlled conditions sometimes turned out to be a challenging problem. Proportional reduction of the dimensions often results in special adaptations for instrumentation of the specimen as well as in special gripping devices.

Performing tensile tests at high temperature using a direct strain-measuring device is difficult. One of the reasons is that high temperature strain gauges are not available for these purposes and therefore electronic transducer systems are used, which are placed outside the furnace. Consequently, special expensive adaptations of the specimens have to be designed. However, load-actuator displacement diagrams are adequate when large amounts of tests are performed in the frame of scoping material properties.

Miniaturisation of CT-specimens introduces experimental limitations, such as direct measurement of load-line displacement at elevated temperature, which could not be used for testing the 2.5-mm thick CT specimens from the MINOSSE irradiation experiment. Due to the lack of displacement instrumentation it was not possible to determine crack length from unloading compliance measurements. Therefore, an electrical potential drop technique is used to obtain the crack length data for calculations of the J-parameter. The positions of the current leads in the specimen and the dimensions of the leads were also a compromise between testing requirements and remote handling techniques.

Sometimes, automated testing systems are developed to perform large series of tests, like Charpy impact testing. Inherently, testing systems for small size samples are taking less hot cell volume. However, certain effort is required to control the temperature of the small specimens during testing in an adequate way.

3 Examples of results

3.1 Tensile test

The tensile experiments are performed on an INSTRON 1362 electro-mechanical testing machine, which is installed in a medium activity hot cell. The testing assembly includes a 10 kN load cell and a three zone furnace supplied by Severn Furnace Limited (SFL), UK. The furnace is supplied with an Eurotherm 900 EPC series controller. The strain rate is $5x10^{-4}.s^{-1}$, obtained from a constant actuator movement.

Strength data at specific plastic strain levels, such as 0.2% yield strength as well as the ultimate tensile strength and uniform elongation are calculated from the load-displacement curves. Other tensile properties, like the total elongation and reduction of area are measured on the specimen after the test.

Two typical curves for an unirradiated and irradiated V-4Cr-4Ti alloy tested at 325°C are presented in figure 6.

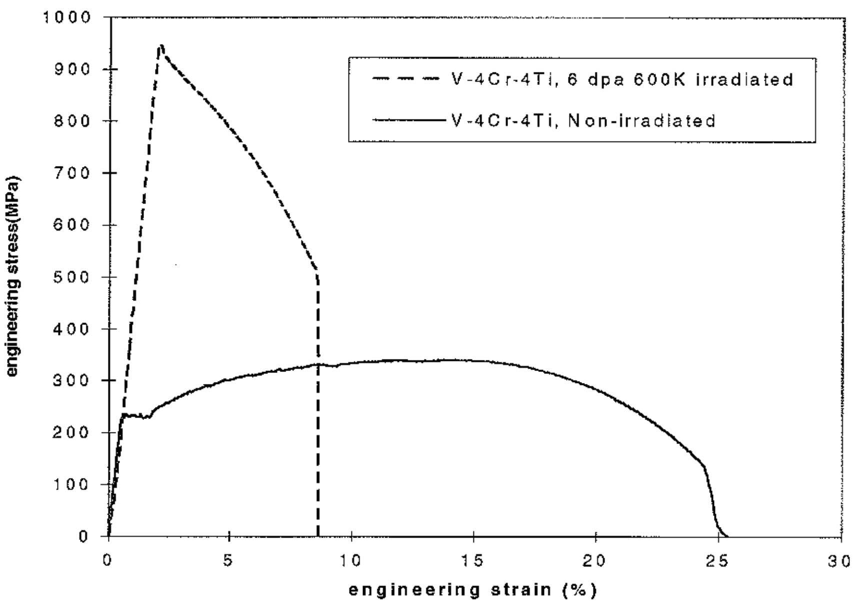


figure 6 Typical tensile test curve of an unirradiated and a 325°C, 6 dpa irradiated Vanadium alloy specimen tested at 325°C

3.2 Fracture resistance experiment

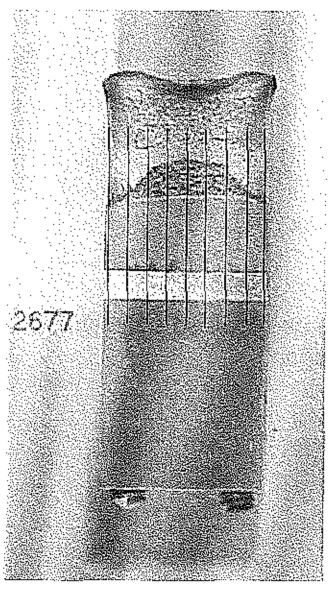
The post-irradiation fracture resistance experiments are performed on a 100 kN INSTRON servo-hydraulic testing machine, installed in a hot cell. The test procedure is based on the recommendations as described in the European Structural Integrity Society (ESIS) P2-91D procedure of the European Group on Fracture (EGF).

To accommodate the small size CT specimens, the capacity of the load cell has been adjusted to 20 kN. The load-line displacement is measured by means of a strain averaging system with two 20 mm transducers. The measurement devices are automatically reset at the beginning of each fracture mechanics experiment. A separate direct current potential drop (DCPD) system is used for crack monitoring. The computer controls this crack data acquisition in real time.

Elevated temperature tests are performed in a two-zone furnace with thermocouples located in both clevises. The specimens are tested in air environment after a temperature stabilisation period of about 30 minutes.

The plain-sided specimens are fatigue pre-cracked at room temperature after irradiation, in order to avoid effects of the irradiation environment on the crack tip. Automatic stepped down loading at regular crack length intervals is applied for the fatigue pre-cracking.

The final DCPD crack length measurement is adjusted with the final crack length as optically measured on both fracture surfaces (weighted 9-points averaging method). In figure 7 an example is given of a CT 10-mm fracture surface, whereas in figure 8 the result of a fracture resistance experiment is presented.



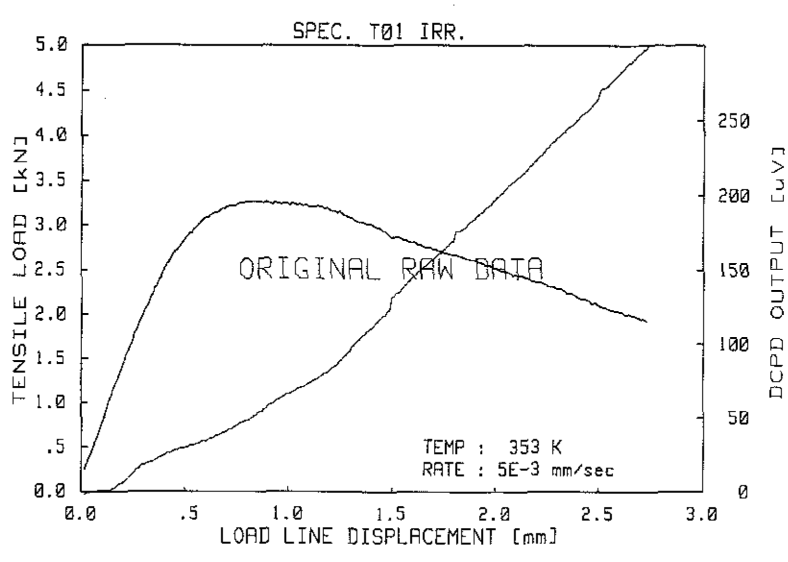


figure 7 Fracture surface analysis

figure 8 Load-displacement vs tensile load and dc-output curves as recorded during a fracture resistance test

3.3 Charpy impact test

Impact testing is performed on an instrumented and automated miniaturised PW5 system, manufactured by INSTRON-Wolpert. The potential energy is 50 J and the impact speed is 3.85 m/s. The system includes a heating/cooling device with which temperatures can be reach from –180 to 300°C, and an automatic specimen transfer module. The transfer module allows automatic positioning of the specimen in the heating/cooling device as well as automatic positioning on the anvil after conditioning.

The testing practice is according to the ISO-CD 14556 (Draft) standard. The testing temperatures range from –150 to +150°C.

The results are presented in terms of impact energy, lateral expansion and load-displacement of the impact. The impact energies are used to plot transition curves from which the ductile-to-brittle transition temperature (DBTT), and to determine the upper shelf energy (USE) for the material.

An example of the DBTT curve for an RAFM alloy is given in figure 9.

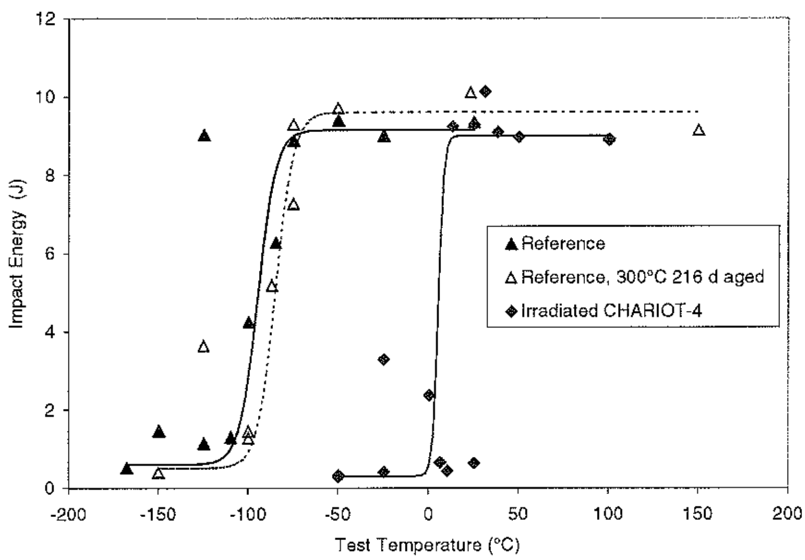


figure 9 Example of Charpy impact curves of a RAFM plate material in the as-received condition, unirradiated aged condition, and a 300°C, 2.5 dpa irradiated condition