

# Techniques for mechanical tests of small size specimens in the hot cells of FSUE SSC RIAR to study structural materials for fusion reactors.

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## Introduction

At present, small size specimens are widely used to study the postirradiation behavior of ferritic-martensitic materials for fusion reactors [1-5]. First of all, it is due to the limited irradiation volume, in which experimental conditions (temperature, damage dose and neutron spectrum) can be identically provided. Thus, the small size specimens allow us:

- to increase the number of irradiated samples of different materials;
- to minimize the uncertainty of the specimen irradiation parameters (temperature and irradiation damage) within one and the same irradiation volume;
- to compare the postirradiation characteristics of different materials and reveal the role of certain factors (effect of alloying elements, microstructure features, heat treatment etc) in the material radiation damage.

During several years the promising ferritic-martensitic steels have been irradiated in the BOR-60 reactor as candidate materials for the fusion reactor first wall. One of the PIE stages is examination of the mechanical properties using small size specimens. For this purpose FZK IMF II (Karlsruhe, Germany) has developed and delivered to FSUE SSC RIAR two facilities for mechanical tests. The first facility is an instrumented remote impact testing machine Zwick 5113 - HKE that is intended for the Charpy tests of the KLST specimens in the temperature range from  $-190^{\circ}\text{C}$  to  $+680^{\circ}\text{C}$ . The second facility is based on INSTRON 1362-DOLI and intended for tensile and low-cycle fatigue (LCF) tests. It is equipped with the "MAYTEC" HTO-08 furnace and high-temperature PMA-12/V7-1 extensometer that allows the instrumented tests in the temperature range from  $+20^{\circ}\text{C}$  to  $+800^{\circ}\text{C}$ . This equipment is mounted in the hot cells of the Material Science Complex of FSUE SSC RIAR and has been operated successfully during the last two years.

## Impact bending testing.

The KLST-type specimens (27x4x3mm) with a 1mm V-notch are used for the impact bending testing.

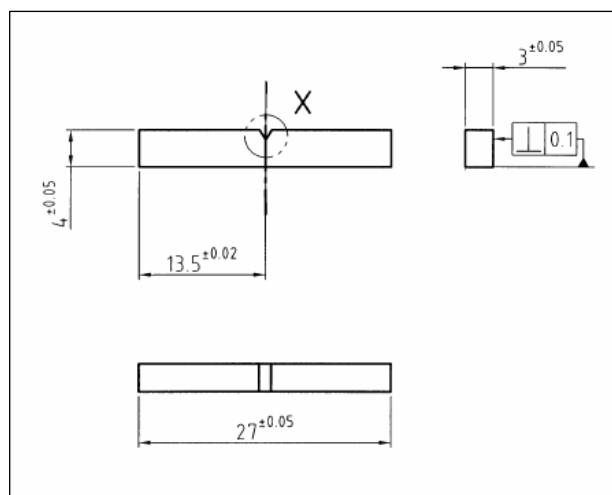


Fig.1. Miniaturized KLST-Charpy specimen (all dimensions in mm).

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The used impact testing machine Zwick 5113-HKE has the following characteristics:

- distance between supports for the specimen is 22 mm;
- 15J pendulum impact hammer with an instrumented tup  $\varnothing 2\text{mm}$ ;
- device for the specimen cooling/warming in the temperature range from  $-190$  to  $+680^\circ\text{C}$ ;
- automatic system to move the specimen out of the magazine into the thermostat and to fix the specimen on the supports.

Figure 2 presents the general view of the impact testing machine and specimen control unit

The specialized AUTOTEST and ImpactWin software and central processor are used to set the testing parameters, to control the tests and to process the data acquisition and treatment.

For each test the “force-time” curve was recorded and the impact energy value (specimen fracture energy) was determined by integration. The test protocol contains the test temperature that is determined with the account of the change of real specimen temperature during its moving from the furnace to the supports and pendulum hammer falling. The testing technique corresponds to ISO-CD14556:2000(E).

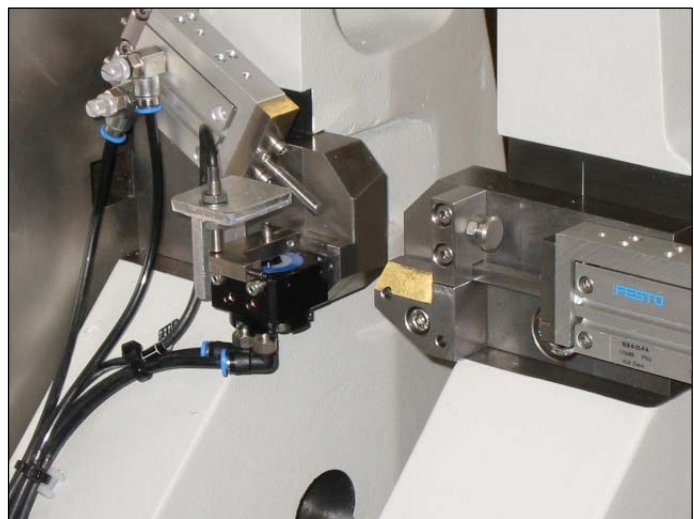
The diagram of fracture energy vs. test temperature is then plotted, and the ductile-brittle transition temperature (DBTT) is determined at which the fracture energy is a half of Charpy upper shelf energy. Figures 3 and 4 present the typical fracture diagrams of irradiated specimens in the ductile-brittle transition region as well as fracture nature. The dependence of impact energy vs. testing temperature is presented in Fig.5.

After the testing the photos of specimen fragments are made using the instrumented remote microscope TIMM-250C located in the hot cell. The obtained photos are processed and used for determination of a portion of ductile and brittle fracture components.

Table 1 presents the impact toughness testing results of promising materials for the fusion reactor first wall [6]. The data related to the characteristics of irradiated materials are obtained using equipment located in the VK-39 hot cell of the FSUE SSC RIAR Materials Science Complex.

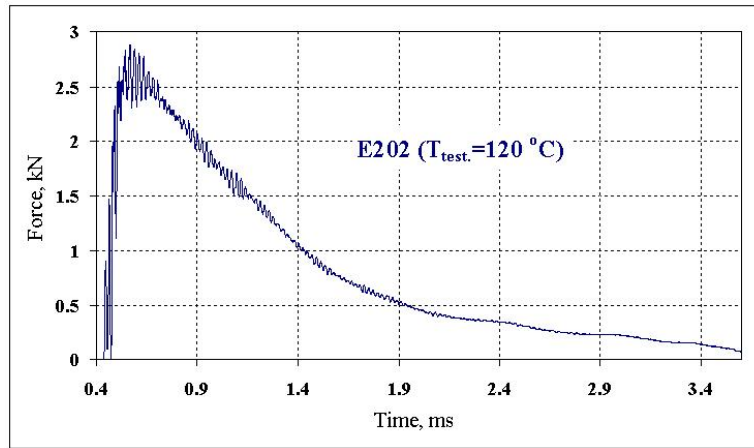


a)

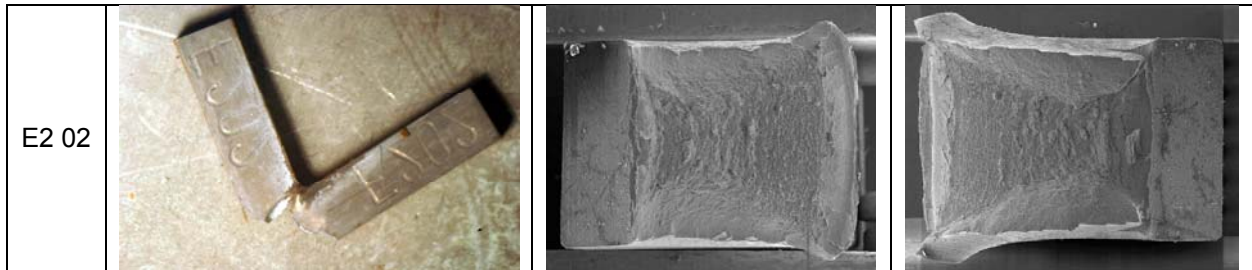


b)

Fig.2. General view of the impact bending testing machine (a) and unit to control the specimen location on the supports (b)

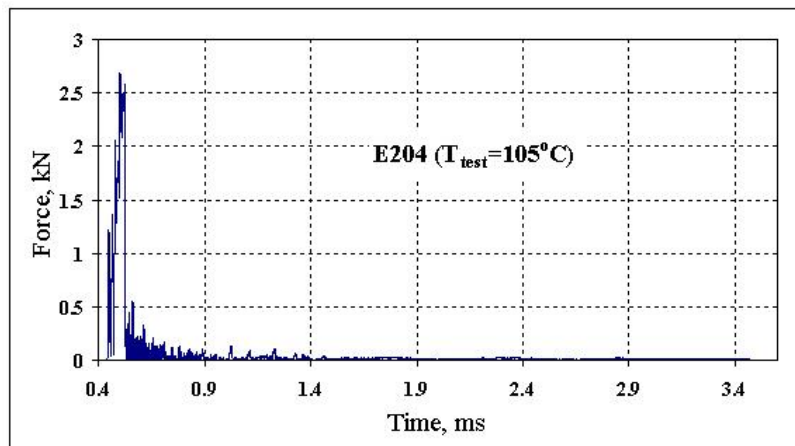


a)

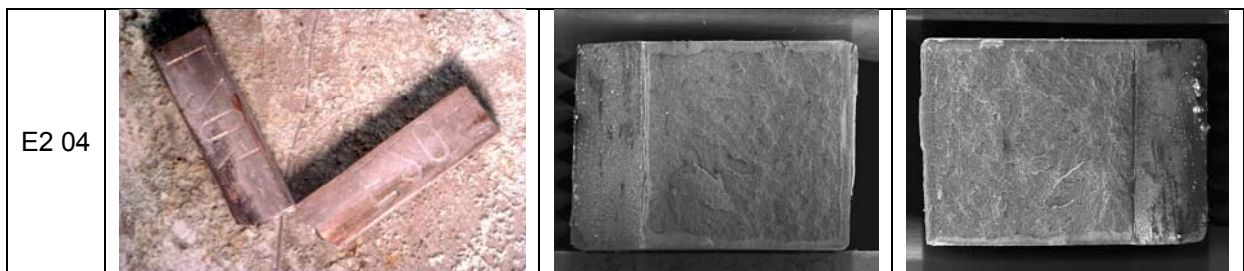


b)

Fig.3. The force-time curve at  $T_{test} = 120^{\circ}\text{C}$  (a), general view of the tested specimen and fracture appearance (b).



a)



b)

Fig.4. The force-time curve at  $T_{test} = 105^{\circ}\text{C}$  (a), general view of the tested specimen and fracture appearance (b).

Fig.5.: The energy vs. temperature diagram for irradiated ferritic-martensitic steel.

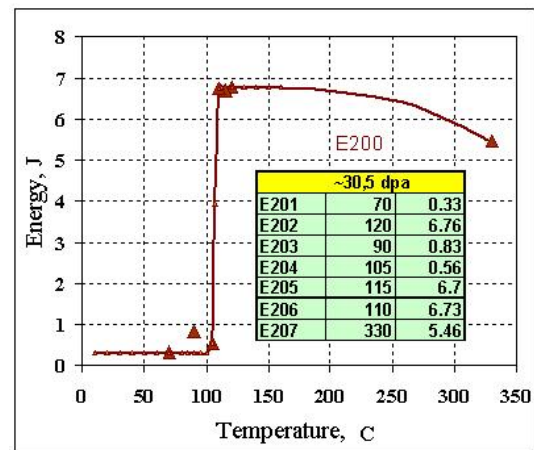


Table 1: Characteristics of impact toughness of candidate materials for the fusion reactor first wall [6].

Material	$T_{irr.}$ , °C	Dose, dpa	USE unirr., J	USE, irr., J	DBTT unirr., °C	DBTT, irr., °C	$\Delta$ DBTT, °C
EUROFER 1	332	31.8	9,8	7,0	-81	137	218
EUROFER 2	332	31.8	9,84	6,76	-90	107	197
F82H	332	32.3	9,4	5,0	-72	148	220
OPT IVc	332	32.3	9,12	5,84	-105	48	153
ODS-EUROFER	332	31.8	2,54	1,5	135	382	247

### Low-cycle fatigue testing.

Small size specimens with a cylindrical gauge length and thread heads are used for the strain controlled low cycle fatigue testing. The specimen is presented in Fig.6. There are several steps in preparing specimen for the testing. They are:

- additional cleaning in the ultrasonic bath;
- installing of special washers on the specimen heads;
- adjusting and check of the distance between the washers and specimen edges.

Some additional devices and instruments were developed to perform the above operations remotely in the hot cell.

The tests are performed at a special remote machine. The frame of the electro-mechanical testing machine Instron 1362 with a rigidity of 450kN/mm is used. This machine was modernized and equipped with the following devices:

- three-zone furnace HTO-08 of "MAYTEC" production and control unit to maintain the set temperature and register it during the testing;
- high-temperature extensometer PMA-12/V7-1 with ceramic knife-rods to perform strain-controlled experiments in the temperature range from +20°C to +800°C.;
- out-cell digital controller EDC 120 of "Doli" production;
- central processor with the «Messphysik» software to set the testing conditions and collect and process the testing data;

The Instron 1362-DOLI machine mounted in the K-12 hot cell is presented in Fig.7.

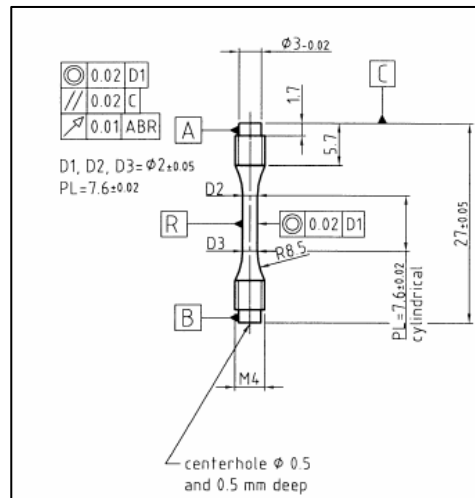
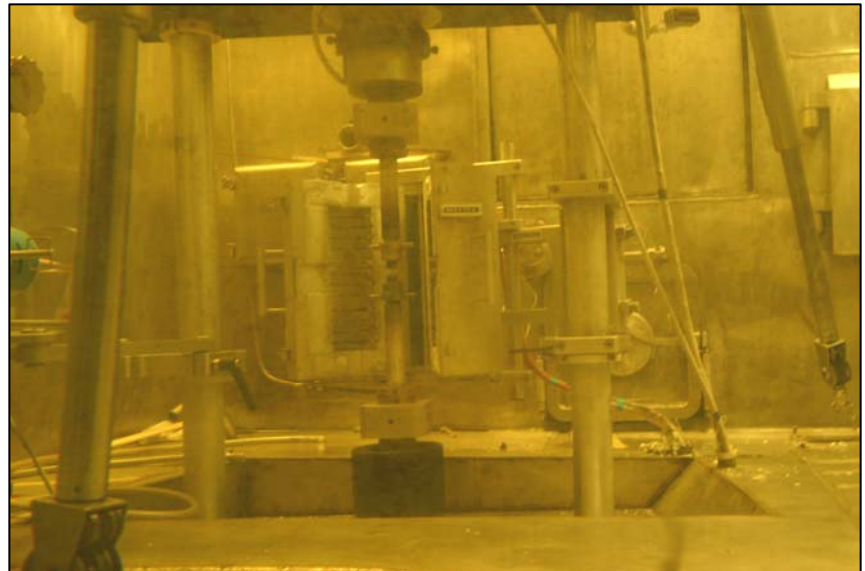


Fig.6. Specimen for the low-cycle fatigue testing.



a)



b)

Fig.7. General view of the low-cycle fatigue testing machine before (a) and after (b) its installation in the hot cell.

The specimen is located in such a way that the head prominences coincide with calibrated slots of the upper and lower grips of the testing machine. Such location results in the direct load transfer to the specimen under compression. As for the tension semi-cycle, the load transfer is made through the head washers. It is important to avoid any gap in the threaded couplings of the whole load train to obtain a symmetric cycle of the elasto-plastic strain. For this purpose a special procedure was developed for the specimen location and its preliminary loading in the elastic area under the continuous force control.

The next step in the testing preparation is the PMA-12/V7-1 extensometer installing. This operation is also performed under the continuous force control so as to avoid any damage of the ceramic knife-rod. The position of rods on the specimen working part is controlled by a mobile digital TV camera located in the hot cell. Fig 8 shows a view of the irradiated specimen and extensometer rods on it.

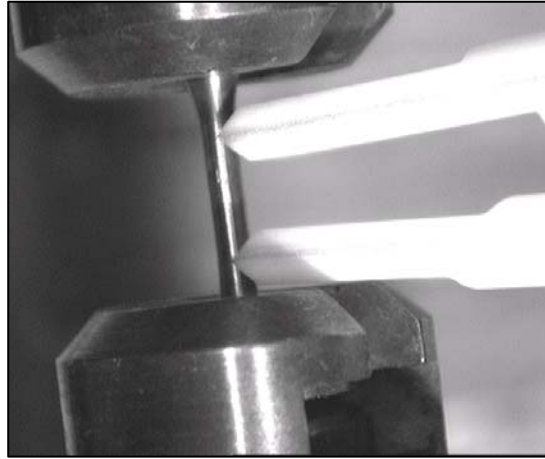


Fig. 8. Specimen with extensometer probes on it.

The testing control and data acquisition and treatment are performed by the central processor with the «Messphysik»ASTM E606 software. This software provides for different types of testing by varying the force, strain and other cycling parameters as the wave form and frequency of cycling. As for our experiments, a rigid cyclic elasto-plastic strain of the material is used. Here the strain range remains constant but the corresponding force changes with the increasing number of strain cycles. The average strain rate makes up  $3 \times 10^{-3}$  1/s. All tests were performed at 330°C. To compensate stresses occurring during the specimen heating up to the test temperature, the heating is performed at a controlled load value of 50N. Figure 9 presents the typical picture of the hysteresis loops change observed during the test of irradiated ferritic-martensitic steel specimens. All examined irradiated ferritic-martensitic steel specimens can be referred to the cyclically unstable softening materials. These materials typically possess an initial softening with further decrease of its intensity and loop stabilization. Figure 10 presents the diagram “peak stress– number of cycles” that is used to calculate the number of cycles  $N_d$  at which a defect (fatigue crack) occurs. According to the ASTM Standard E606, the maximal number of cycles  $N_f$  prior to failure is determined as the number of cycles at which the load decreases by 50% from the maximal value. After the testing, the specimen was cooled to room temperature at a constantly controlled load value of 50N. Then the specimen was ruptured into halves by tension. Each fragment was photographed by means of TIMM-250C microscope. Then the fragments were examined at the remote scanning electron microscope Philips XL-30 installed in the hot cell of Material Science Department. Figure 11 presents the examination results of the irradiated specimen subjected to the low-cycle fatigue testing and tension up to rupture.

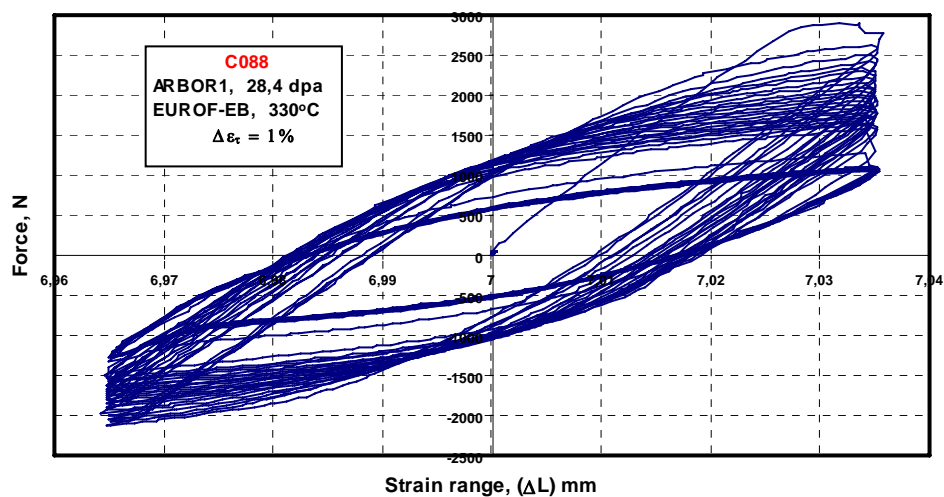


Fig. 9. Hysteresis loops observed during the irradiated specimen testing.

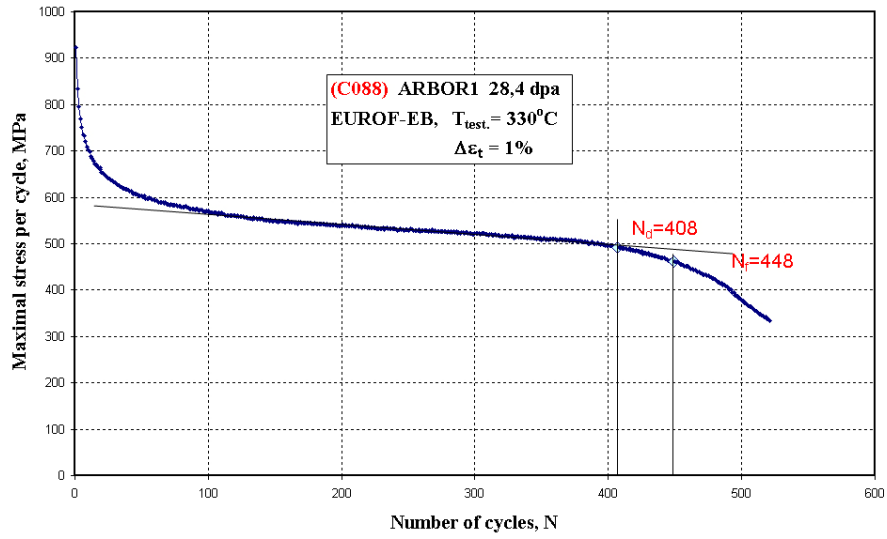


Fig.10. Maximal stress per a cycle versus the number of cycles during the irradiated specimen testing.  
 $N_d$  – the number of cycles to defects formation;  
 $N_f$  – the number of cycles to failure (50% stress drop)

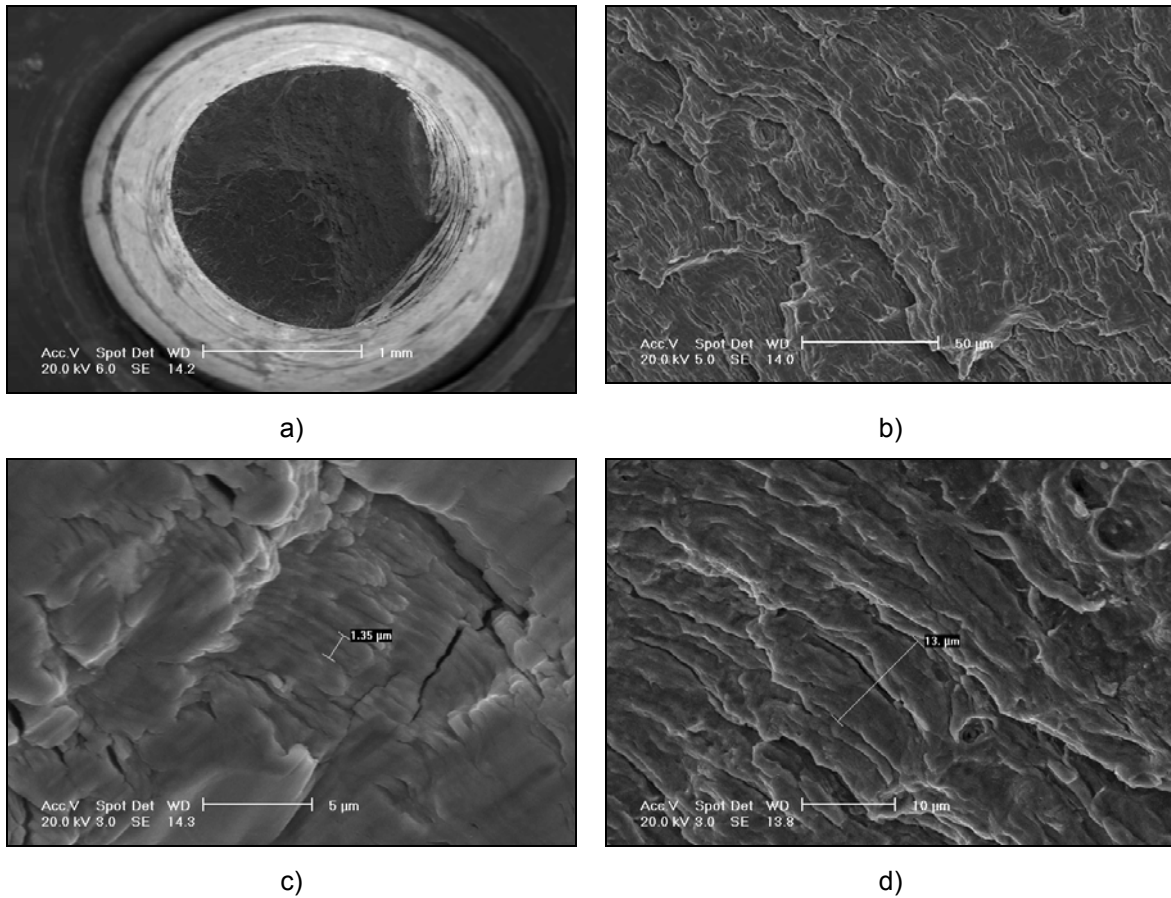


Fig.11. View of the irradiated specimen fracture surface after the low cycle fatigue testing (a) and fracture appearance in a fatigue crack generation region: near the specimen surface (b), in the middle area of the fatigue crack propagation (c) and in the area of defect close (d).

## Conclusion.

The paper presents the techniques for impact bending and low cycle fatigue testing of irradiated small size specimens developed by FZK IMF II together with FSUE SSC RIAR and implemented in the Material Science Department of FSUE SSC RIAR. The implementation of the above techniques in the hot cells required additional devices and equipment as well as try out of special procedures when preparing and performing the irradiated specimen tests. The examinations of ferritic-martensitic steel specimens show the efficiency of the above techniques to obtain data on the material properties after irradiation under the conditions resulting in low-temperature radiation embrittlement that is typical for the materials of this class. The obtained results were presented at the International Conference on the ICFRM-12 Fusion Reactor Materials [6].

## References

1. R.Kasada, H.Ono and A.Kimura, Small specimen test technique for evaluating fracture toughness of blanket structural materials, Fusion Engineering and Desing, 81(2006) pp 981-986.
2. G.E.Lucas, G.R.Odette, H.Matsui, Amoslang , P.Spatig and J.Rensman, The role of small specimen test technology, in fusion Materials development, ICFRM-12, Santa Barbara, USA, 2005, abstracts, session07A, p111.
3. W.R. Corwin, A.M.Hougland. Effect of specimen size and material condition on the charpy impact properties of 9Cr-1Mo-V-Nb steel, The Use of Small-Scale Specimens for Testing Irradiated Material, ASTM STP 888, American Society for Testing and Materials, Philadelphia, 1986, pp.325-338.
4. K.C.Liu and M.L.Grossbeck. Use of subsized fatigue specimens for reactor irradiation testing, The Use of Small-Scale Specimens for Testing Irradiated Material, ASTM STP 888, American Society for Testing and Materials, Philadelphia, 1986, pp276-289.
5. H.Kurishita, T.Yamamoto, m.Narui, H.Suwarno, T.Yoshitake, Y.Yano, M.Yamazaki and H.Matsui, J.Nucl.Materials, 329-333 (2004) pp1107-1112.
6. C.Petersen, A.Povstynko, V.Prokhorov, A.Fedoseev, O.Makarov and B.Dafferner Mechanical property degradation of ferritic/martensitic steels after the fast reactor irradiation "ARBOR", in proceedings of ICFRM-12, Santa Barbara, USA, 2005, in print.