# TECLA PROJECT
# CONTRACT No. FIKW-CT-2000-00092
# WORKING PACKAGE 4
# Effects of irradiation on lead alloy corrosion
# D22 : Report on experimental details

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# LiSoR

Liquid Solid Reaction under Irradiation

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## Abstract :

The present document is the D22 deliverable report of work-package 4 (*Effects of irradiation on lead alloy corrosion*) from the TECLA (*TECHNOLOGIES, MATERIALS AND THERMAL- HYDRAULICS FOR LEAD ALLOYS*) project of the 5$^{th}$ European Framework Program.

It concerns the problematic of irradiation effects on the Liquid Metal Embrittlement in structural materials for liquid metal targets of spallation neutron sources (sub-task 4.1).

This report on the experimental program gives an overview of the main motivations of the LiSoR project. It summarises the experimental objectives and the consequences on the design of the experiment. The experimental program is presented and a short overview on the post irradiation examination is given.

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# 1 INTRODUCTION

## *1.1 General background in the framework of ADS*

Accelerator driven spallation devices are receiving growing attention world wide both as research neutron sources and for application in future energy systems. While the physics of these devices is well understood, their feasibility and reliability hinge on materials issues. Very little is known, so far, on the effects of spallation type radiation on the properties of materials. The main difference relative to thermal and fast neutron irradiation is the high ratio of Hydrogen and Helium production per atomic displacement (dpa).

For a spallation neutron source with power of ≥~1MW, liquid metal (Hg, Pb-Bi, Pb) targets are considered to be the best choice[1]. The main advantage of a liquid metal target is that the target material itself will not suffer from radiation damage, which is the main factor limiting the life-time of a solid target. However, in a liquid metal target, some structural components, particularly the beam window and the containment vessel, will be subjected to not only the intensive radiation damage produced by protons and neutrons but also great mechanical loads generated by the pressure of the liquid metal and the temperature gradient in the material, and moreover, significant or even serious liquid metal corrosion, erosion and embrittlement effects. As a consequence, the components may be damaged soon.

One of the critical issues in liquid metal target development is related to the question, whether **Li**quid metal-**So**lid metal **R**eactions (LiSoR) are enhanced under irradiation in the presence of mechanical stress. Since this is a question that must be answered before a liquid metal target can be used in a spallation neutron source, the LiSoR experiment has been initiated to use 72 MeV protons generated by the Phillips cyclotron at PSI to irradiate stressed steel specimens in contact with flowing liquid metal. Scooping calculations showed that the damage and gas production rates of 72 MeV protons are, within reasonable limits, comparable to those of 600 MeV protons, while much less radioactivity in the liquid metal will be produced. Also, the beam parameters can be adjusted in such a way to obtain relevant heating rates at the solid-liquid interface. The original idea and purpose of such an irradiation experiment can be found in PSI Research Proposal [2].

## *1.2 Scientific background*

For the design and construction of liquid metal (Pb-Bi or Hg) spallation targets, several kinds of steel (martensitic and austenitic) have been identified as the candidate structural materials [3,4]. Whereas for Hg targets, austenitic steels seem the prime candidates, ferritic/martensitic steels are favoured for LBE targets. The following points summarises the main arguments for this choice.

- These steels offer a good mechanical strength and a good compromise between heat conductivity and thermal dilatation lowering the thermal constraints in the material;
- They have a satisfying resistance against liquid metal corrosion due to the contact with the eutectics PbBi and PbLi, due to only moderate dissolution of Fe and Cr into the liquid metal;
- The steels have been applied successfully in Russian reactors cooled by liquid PbBi;
- Mechanical tests of martensitic steels in contact with PbBi did not show significant liquid metal embrittlement;

- The ductile to brittle transition temperature (DBTT) changes under neutron irradiation, however only slightly for steels with 9wt% of Cr.
- The swelling performances are very good under fast neutron irradiation up to a level of approximately 10 appmHe/dpa;
- From the manufacturer point of view the selected steel candidates have good machining properties (e.g. for welding)

The target window, 9%Cr martensitic steels, like EM10 (9%Cr1%Mo) or T91 (9%Cr1%MoVNb), satisfy *a priori* the required metallurgical criteria under irradiation. On the other hand, the liquid metal (LM) must be a satisfying spallation neutron source and maintain the durability of the target structural materials at long term (months to year). In other words, the resistance to corrosion and Liquid Metal Embrittlement (LME) of structural materials for the LM target of the spallation neutron source in ADS are critical issues and must be carefully investigated, without and under proton irradiation.

### 1.2.1 Liquid metal corrosion

The corrosion attack in LM is a dissolution of elements out of the matrix. The corrosion attack by LM increases with increasing solubility of the elements and it depends on :

- the composition of the steel and its impurities content and stress;
- the impurity content of the LM;
- the exposure time of the steel to the LM
- the exposure temperature and temperature range
- the circulating or static conditions of the LM on the material surface;
- the LM velocity across the steel surface.

The corrosion attach of steels by LM leads to the following phenomena :

- dissolution of material (uniform attack) into the LM;
- preferential leaching of one or more components out of the steel matrix;
- solid phase transformation;
- leaching decarburization;
- intergranular attack;
- diffusion of liquid metal atoms into the solid metal lattice;
- mass transfer;
- with increasing temperature and time the corrosion rate increases as well;

Very recently, in the frame of the European research projects related to ADS, was studied the long term corrosion behaviour (in the range of 800 to 3000 h) of martensitic and austenitic steels in lead or lead alloys under stagnant [5] or dynamic conditions [6] in the temperature range expected in ADS.

### 1.2.2 Liquid metal embrittlement

LME is defined as the degradation of the mechanical properties of otherwise ductile metal induced by some interaction with a liquid metal and under mechanical sollicitation [7,8].

The characteristics of liquid metal embrittlement are :

- brittle failure of a normally ductile metal in contact with LM;
- the embrittlement is severe, and the propagation of fracture is very fast in the case of LME;
- the fracture is usually brittle intergranular;
- complete coverage of the solid by the LM, which is usually difficult to remove

It is known that Fe – Pb and Fe – Bi are not known to be embrittling couples [9]. The same observation can be made with steel in lead alloys. However, the signature of an embrittling effect of lead (reduction of area, plastic strain at rupture, UTS) is well mentioned in some cases. For example, it is the case of leaded steels [10], of 8-9%Cr martensitic steels after some specific heat treatments [11]. On the other hand, it was shown recently that notched specimens exhibit brittle rupture under some particular metallurgical and mechanical testing conditions [12]. In case of observed LME the phenomena were :

- severe embrittlement occurs near the melting point of the LM
- around 50 °C above or below $T_m$
- propagation of cracks appears to occur immediately
- presence of liquid at the moving crack tip appears necessary for fast fracture
- reduction in fracture stress, strain or both
- increase of the test temperature decreases embrittlement, which leads to a brittle-to-ductile transition

Figure 1 gives a schematic view of the change of ductility influenced by LME as function of the LME temperature.

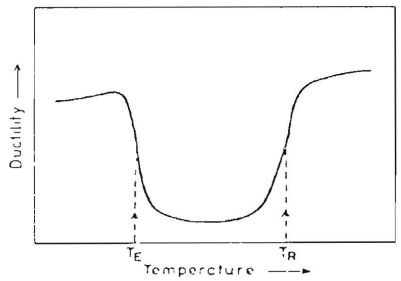


Figure 1 : Change of ductility versus temperature ($T_E$ : ductile – brittle temperature; $T_R$ : brittle – ductile temperature)

On the other hand for martensitic steels with standard heat treatments and specimens without notches the following observations can be made regarding LME :

- no corrosion, dissolution or diffusion-controlled intergranular penetration process;
- solid metal has little or no solubility in the LM;
- no formation of intermetallic compounds;
- little or no penetration of LM into the solid metal;
- time and temperature dependent processes are not considered to be responsible for LME

A general overview on liquid metal and a presentation of the state-of-art appraisal can be found in [13].

# 2 PROJECT OVERVIEW

The LiSoR experiment constitutes task 4.1 of WP4 of the TECLA project and provides an important contribution for the choice of materials for the MEGAPIE target to be built by the MEGAPIE initiative. MEGAPIE [14] (MEGAwatt PIlot Experiment) is a joint initiative between PSI, CEA, CNRS, ENEA, FZK, JAERI and SCK/CEN to design, build and operate a liquid lead-bismuth spallation target at a proton beam power of 1 MW, taking advantage of using the existing spallation neutron facility SINQ at PSI. The project complements the investigations of WP1 to 3, in which the corrosion and mechanical behaviour of materials under off-beam conditions are investigated.

The LiSoR project aims to irradiate steel specimens in a lead-bismuth loop under operating conditions representative for future liquid metal spallation targets. The loop contains test samples that will be exposed simultaneously to the proton irradiation, flowing liquid metal at elevated temperatures, fast temperature changes due to the discontinuous operation of the beam (up to 1000 beam interruptions within 6 days) and mechanical stress.

It is foreseen to perform 5 irradiation experiments with different irradiation times with the longest being 40 days

After irradiation, the test samples will be analysed in the hot cells at PSI.

The MEGAPIE project has chosen the ferritic/martensitic T91 steel (9Cr1MoVNb) as the prime material for the beam window.

As summarised in the WP 4 description of the TECLA project, LiSoR was planned to investigate ferritic/martensitic steels (9Cr1Mo, 9Cr1MoVNb), clean martensitic steels such as F82H and Optifer (moderate DBTT shift, good creep behaviour under irradiation [15]), and the austenitic stainless steel, type 316. Irradiation experiments were planned to run under conditions with both static and dynamic mechanical loads. To investigate the irradiation effects, the material from both the irradiated region and the adjacent unirradiated regions of the test samples will analysed. Control tests will be performed at the same Pb-Bi flow conditions without irradiation. This needs an adequate design of the samples taking into account local beam shielding and temperature gradient calculations. The post irradiation examination will be done in hot labs where a number of devices and methods are available: gamma spectrometry, optical microscopy, Scanning Electron Microscopy (SEM), Electron Probe Microanalysis (EPMA),Secondary Ion Mass Spectrometry (SIMS), Inductive Coupled Plasma Mass Spectrometer (ICPMS).

In the framework of the project, five irradiation experiments are planned. The number of experiments is limited by the costs for each test section to be irradiated and post irradiation examinations, the availability of Injector-I at PSI and the duration of the project. These boundary conditions required a detailed revision of the relevant experiments to be performed with LiSoR in order to make sure to have pertinent and concluding results to verify T91 for MEGAPIE. For this reasons and for the time being, the other steel candidates (Optifer, F82H and others) had to be cancelled from the initial program. Additional tests on these materials could be envisaged if a project prolongation with additional financial support could be obtained.

Therefore, the objective of the LiSoR project is re-defined as to expose T91 steel samples simultaneously to irradiation, mechanical load and liquid Pb-Bi. After irradiation the material properties will be examined to identify any modifications in the microstructure and mechanical behaviours.

# 3 LISOR MATERIAL SELECTION

The ferritic-martensitic steel T91 has been chosen as reference material for the construction of the MEGAPIE liquid metal target window. This selection was based on the following arguments [16]:

Firstly, the radiation effect and heat deposit resistance, as well as the Pb-Bi compatibility of the structural material of the window, the container hull of MEGAPIE should be as high as possible. In any case it should be prototypical of the 1 to 5 MW spallation target required for the European Demonstrator of Accelerator Driven System.

Secondly, due to the tight schedule of MEGAPIE, the structural material has been selected among industrial grades used in the core structure of experimental or power plant reactors. Al alloys have been rejected due to a too high operating temperature in MEGAPIE and Pb-Bi compatibility. Zr alloys have been discarded due to their limited resistance to radiation effect and ability to work at temperature up to 500 – 550°C in the perspective of the European Demonstrator. Ni based alloys such as 706 and 718 have also been discarded due to heavy inter-metallic phase precipitation under fission neutron irradiation, that results in high embrittlement and premature 706 fuel-cladding brittle failure in fast reactors. Austenitic steels have not been selected on the basis of a low heat deposit resistance and chemical composition dependence of swelling resistance, that rule them out as prototypical materials for the ADS European Demonstrator.

Finally, the 9Cr1MoNbV (industrial grade T91) has been selected on the base of the in-service properties database obtained on 9-12%Cr ferritic-martensitic steels after irradiation under fission neutron and proton neutron mixed spallation spectrum (SINQ and LANSCE). The 9-12%Cr martensitic steels present an excellent compromise between mechanical strength, resistance to heat deposition and radiation under fission neutrons. The compatibility with Pb-Bi is reasonable without irradiation, and is reported as good from russian experience under fission neutron spectrum.

The main chemical compositions of T91 (wt%) from the manufacturer Creusot Loire Industrie, used for a certain number of LiSoR samples, is shown in table 1. Slight variations can be possible when using T91 from a different material provider. For the experiment only samples with standard heat treatment will be investigated (see table 2).

| C | Mn | P | S | Si | Cr | Mo | Ni |
|---|---|---|---|---|---|---|---|
| 0.105 | 0.38 | 0.009 | 0.003 | 0.43 | 8.26 | 0.95 | 0.13 |
| V | Nb | $N_2$ | Al | Cu | As | Sn | Ti |
| 0.2 | 0.08 | 0.055 | 0.024 | 0.08 | 0.02 | 0.008 | 0.014 |

**Tabel 1 :** Main chemical composition of the LiSoR T91 (wt%, balance Fe) samples.

| |
|---|
| Phase 1 :<br>Annealing temperature : 1040 C for 60 minutes |
| Air quench |
| Phase 2 :<br>Annealing temperature : 760 C for 60 minutes |

**Tabel 2 :** Standard heat treatment for T91 samples.

In figure 2 to figure 5, the principle mechanical parameters of the T91 steel are compared to those of solution annealed 316L (SA316L). The data are extracted form the French design code RCC-MR[17]

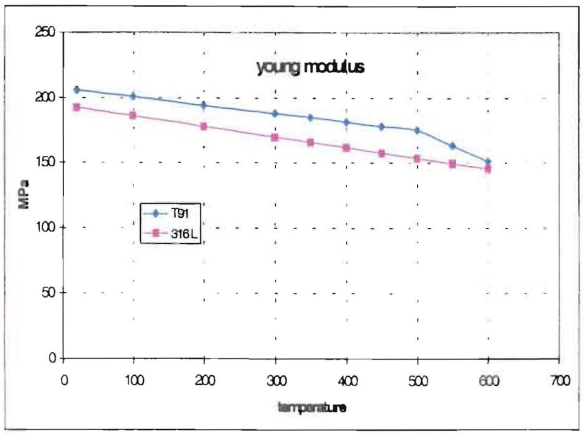


**Figure 2.:** 316 L and T91 young modules

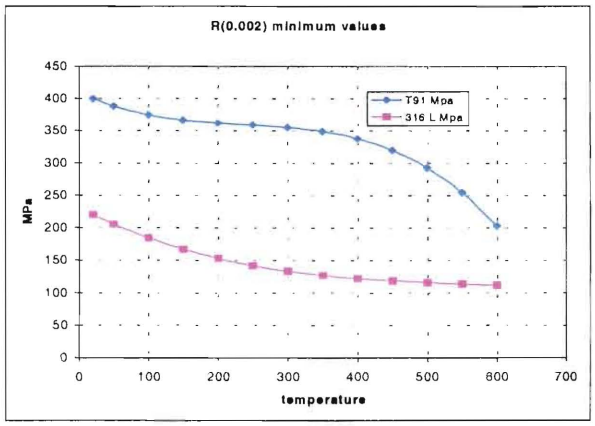


**Figure 3.:** Minimal yields strength for T91 and 316L

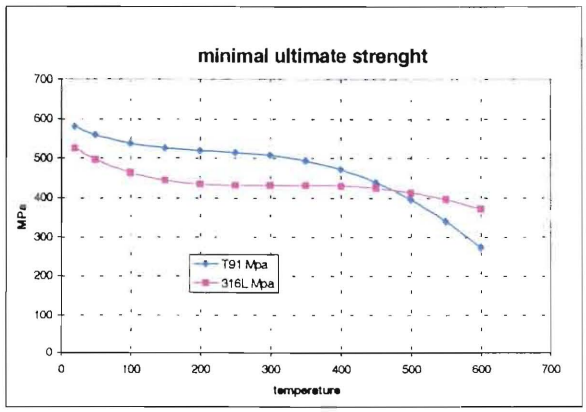


**Figure 4.:** Minimal ultimate strength from RCC-MR

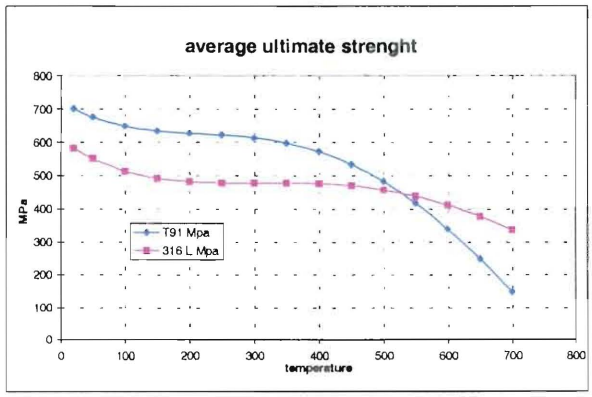


**Figure 5.:** Average ultimate strength from RCC-MR

# 4 THE LISOR EXPERIMENT

## *4.1 Experimental parameters*

The main concern about the significance of the work is whether 72 MeV protons can produce similar irradiation effects in materials as the higher energy protons in real neutron source targets do. To answer this question, we have performed calculations using the HETC code to compare the results from 72 MeV and 600 MeV proton irradiation.

Using the HETC code available at PSI, one can calculate radiation damage rate, gas (He and H) and transmutation nuclei production rates, as well as energy deposition in materials produced by high energy (generally say ≥ 20 MeV [18]) protons. Instead of performing complicated calculations for steels, we just use results of Fe as references. Table 3 presents the calculation results for a 1 mm thick Fe plate irradiated with 72 MeV and 600 MeV protons.

| $E_p$ (MeV) | dpa* / p/cm$^2$ | $\sigma_{He}$ (mb) | $\sigma_H$ (mb) | $\sigma_Z$ ** (mb) | $E_{dep}$ ***(MeV/p) |
|---|---|---|---|---|---|
| 72 | $5.2\times10^{20}$ | 130 | 1020 | 800 | 5.25 |
| 600 | $4.4\times10^{20}$ | 460 | 1980 | 770 | 3.0 |

**Table 3** Results of HETC code calculation for a 1 mm thick Fe plate irradiated with 72 and 600 MeV protons.
* dpa, displacement per atom. The number means on average every atom in the Fe plate has been displaced for one time by 72 MeV proton at a fluence of $5.2\times10^{20}$ /cm$^2$.
** $\sigma_Z$, production cross section of total transmutation elements except for H and He.
*** $E_{dep}$, energy deposited in the Fe plate per incident proton.

It can be seen that, as compared to 600 MeV protons, 72 MeV protons produce about the same damage, little less than 1/3 He, about ½ H, but generate similar amount of transmutation impurities and deposit more energy. Although He and H production is lower, the important parameter, He to dpa ratio (He/dpa), for 72 MeV proton is about 68 appm/dpa which is still much higher than in any neutron (fast, thermal and fusion) irradiation case, where the numbers are 0.5 to 20 appm/dpa. He and H effects will be seen if they really play an important role. On the other hand, in the target of a neutron source the window receives not only proton irradiation but also more or less equivalent neutron irradiation, which brings the He/dpa down to about 100 appm/dpa. The neutron production by 72 MeV protons is much lower and the contribution of neutrons to radiation damage is almost negligible.

Nevertheless, the similar radiation damage rate and reasonably high H and He production rate provide the fundamental significance of the experiment.

By varying the beam spot size, from about 10 mm$^2$ to 4 cm$^2$, and the beam current, from 10 to 50 μA, one can easily change the power density at the beam window in a very large range from about 150 W/cm$^3$ to 25 kW/cm$^3$,which covers all the maximum values at target windows of current spallation source projects such as SINQ, ~700 W/cm$^3$, and ESS (5 MW European Spallation Source), ~1400 W/cm$^3$. With a suitable geometry of the window and flow rate of the liquid metal in the loop (see next section), the temperature and (thermal) stress at the beam window can be changed in a wide range, too.

Therefore, we believe that this experiment can provide a very good simulation for the situation at SINQ liquid metal target window and can generate information which is indispensable to the design and safe operation of liquid metal target windows.

Table 4 summarises the experimental parameters of the LiSoR experiment. This parameter set assures to be in MEGAPIE representative operational conditions for the irradiation of the samples.

| **Parameter** | **Value** |
|---|---|
| Irradiation conditions : | |
| ▪ Beam energy : | 72 MeV |
| ▪ Max. beam current : | 50 μA |
| ▪ Max. proton flux : | 3,1 $10^{14}$ p/cm$^2$/s |
| ▪ Max. current density allowed : | 50 μA/cm$^2$ |
| ▪ Max. fluence : | $10^{21}$ p/cm$^2$ |
| ▪ Beam size: | 10 mm (2sx=2sy); FWHM=1.18 mm |
| ▪ Beam structure: | wobbling horizontal $x_{max}$ = ± 2.75 mm |
| ▪ | wobbling vertical $y_{max}$ = ± 7 mm |
| ▪ Beam time per test : | 10, 20 days, 40 days |
| ▪ Radiation damage: | ~ 0.5, 1, 2 dpa |
| Test section material choice : | |
| ▪ Test section tube material: | T91 martensitic steel and MANET II |
| ▪ Test section tensile specimen : | T91 or back-up material |
| Liquid metal (LM) characteristics : | |
| ▪ LM : | lead-bismuth eutectic |
| ▪ LM temperature (design temperature for the loop) : | max. 300°C |
| ▪ LM speed in the test section : | ~1 m/s |
| Mechanical load conditions on the test sample : | |
| ▪ Kind of load : | Static load |
| ▪ Maximum stress (including thermal stress) : | 0.5 $R_{p0.2}$ (at 300 °C) |

**Table 4.:** Experimental configuration for the sample irradiation.

## *4.2 The experimental program*

To get sufficient and relevant data for the validation of the MEGAPIE target window material 5 irradiation experiments are foreseen.

In the first experiment the sample will be exposed to irradiation only during a short time. In this case the dpa will not be a relevant parameter. The irradiation by the proton beam will just assure the local heat deposition in the sample. The obtained tensile curve will be compared to data performed without irradiation. The temperature of 200°C is chosen to stay in the range of

100K above the lead-bismuth eutectic melting temperature (125°C), which corresponds to the critical region for liquid metal embrittlement (see chapter 1.2.2).

All other test are planned at 300°C. This temperature corresponds to the temperature of the liquid metal at the MEGAPIE window surface. The main parameters that can vary between the different experiments will be the irradiation time, the load condition and the material of the specimen.

Table 5 gives an overview of a possible experiment matrix for the five irradiation experiments. The feed back of each experiment will determine the final parameter set for the next one. Hence, modifications are possible and a final decision on the type of the back-up material and the load conditions will be taken later. The duration of the experiment will mainly depend on the available beam time at the PSI Injector I. A minimum of 20 days however will be requested.

| No | Material Test section | Material Specimen | dpa | Duration | Load conditions | Temperature |
|---|---|---|---|---|---|---|
| 1 | MANET | T91 | <0.01 | short | Tensile test | 200°C |
| 2 | T91 | T91 | 1 | 20 days | 50% $R_{p0.2}$ (at 300 °C) | 300°C |
| 3 | T91 | T91 | 2 | 40 days | 50% $R_{p0.2}$ (at 300 °C) | 300°C |
| 4 | T91 | T91 or back-up | 1 | 20 days | ≥ 50% $R_{p0.2}$ (at 300 °C) | 300°C |
| 5 | T91 | T91 or back-up | 1 | 20 days | ≥ 50% $R_{p0.2}$ (at 300 °C) | 300°C |

**Table 5.:** Summary of the 5 irradiation experiments planned with LiSoR

# 5 DESIGN OF THE TEST UNIT

The central unit of the LiSoR liquid metal loop is the test section with the test sample to be irradiated. In order to be able to identify the origin of any changes in the material properties, the design of the test section allows to examine samples from different parts of the test section at conditions of both with and without mechanical load, and with and without irradiation. This specification lead to a special test section design with two T91 components : the test section tube and the test specimen.

## *5.1 The test section tube*

The test section tube as shown in figure 6 is a part of the liquid metal loop. The liquid metal is flowing inside of the tube. The outside of the tube is the vacuum of the proton beam line. The test specimen is located inside the tube in the flowing liquid metal (see next paragraphe).

Two zones are indicated on the test section tube. The lower spot corresponds to the zone irradiated by the proton beam. The upper spot is a part of the non-irradiated part of the test section tube. There is no additional mechanical load to be applied on the tube. The stress is induced by the pressure difference between the vacuum outside and the liquid metal inside the tube. Compared to the mechanical stress added to the test specimen it can be neglected.

**Figure 6.:** The test section tube of T91. The lower spot shows the region impacted by the beam. The upper region is a non-irradiated zone.

## *5.2 The test specimen*

The inner test specimen, as shown in Figure 7, is 50 cm long, 20 mm wide and 5 mm thick with a reduced section of 10 mm long and 1 mm thick. This 10 cm section will be located at the position corresponding to the test section tube. Since the proton beam size is about 12×6 mm$^2$, the rest part will be not irradiated or irradiated with some spallation neutrons.

All test specimens for irradiation will have an additional weld in the region above the irradiation spot. The weld will only be exposed to the neutrons produced by the incident proton beam in the liquid metal. Although the neutron production rate is not very high, the weld will be analysed for any modifications.

**Figure 7.:** Test specimen with the irradiated (lower spot) and non-irradiated (upper spot) zone.

Although the proton energy is smaller and the beam spot size slightly larger on the inner test samples, the heat density at the inner sample is comparable to the one dissipated in the front wall of the tube.

Figure 7 demonstrates the specimen. The lower and upper spots are the irradiated and non-irradiated areas which correspond to those indicated in Figure 6.

By this special design of the test tube and the inner test specimen, four zones are identified having different constraints but the same experimental conditions (e.g. liquid metal velocity). This allows the experiment to extract information from material with or without mechanical load, with or without irradiation under the same operating conditions, resulting in a significant gain of beam time.

# 6 THE POST IRRADIATION EXAMINATION

The goal of the post-irradiation examination (PIE) is to study microstructural changes in surface layers which contact with Pb-Bi and the mechanical properties degradation of the material after irradiation. PIE will be performed in Hotlabor at PSI. The investigations and experiment procedures are described as what follows (slight modifications in the final PIE program might be possible).

1) Gamma Scan

After the test section tube and sample being transported to Hotlabor and loaded in a hot-cell, γ-scan has to be performed with a spatial resolution of 2×2 $mm^2$ and steps of 1 mm to give a precise irradiation intensity distribution.

2) Cutting samples

The samples will be cut with a diamond wire saw machine. The samples to be cut from the irradiated sample area are shown in Figure 5. The same type of specimens will be cut from un-irradiated area for comparison.

3) Gamma Spectrometry

With γ-spectrometry one can analyse quantitatively the radioactive nuclei produced by the spallation reaction and measure the activity of specimens. Small samples from different places will be measured.

4) Optical Microscopy (OM), Scanning Electron Microscopy (SEM) and Electron Probe Microanalysis (EPMA)

Optical microscopy and the SEM can be used to observe the morphology of the surface and transverse section of the specimens, see Figure 8. Observation on the transverse section will show whether there are cracks. The SEM in the Hot-lab is equipped with the Energy Disperse Spectroscopy (EDS) which can analyse "semi-quantitatively" chemical compositions of an area as small as 2 μm in diameter. However the SEM analysis can be made only on specimens with low dose rate, <2 mSv/h at 1 cm. For specimens with higher dose rate, chemical analysis and surface analysis (topography) can be made with the shielded EPMA.

5) Secondary Ion Mass Spectrometry (SIMS)

SIMS can, in principle, provide the depth-profile for a number of isotopes in the specimen. The goal is to investigate possible change of chemical compositions in the PbBi corroded surface layer. This could provide important information for understanding the corrosion mechanism.

6) Transmission Electron Microscopy (TEM)

The changes of microstructures during irradiation, namely the changes in the precipitate structure, the dislocation structure, the micro-chemistry at and near grain boundaries, can be analysed with the Transmission Electron Microscopy (TEM) which will be located at building OFLB. This basic information is needed in order to explain eventual changes in mechanical properties.

7) Hydrogen and Helium analysis

Hydrogen and helium effects on the mechanical properties of materials are well known. It is important to measure their concentration precisely. H and He can be measured by gas extraction method. Few samples will be sent to Pacific Northwest National Laboratory (PNNL) in USA since the facility in PSI Hotlabor is not available for Helium measurement by the time.

8) Micro-hardness and tensile tests

The change of mechanical properties will be accessed by perform micro-hardness test and tensile test. Due to relatively small area to be irradiated, only few tensile samples can be cut

from the area. Micro-hardness test will be associated with the optical microscopy observation and can be only performed at room temperature.

9) Inductive Coupled Plasma Mass Spectrometer (ICPMS)

The change of chemical compositions in the PbBi due to irradiation and corrosion can be measured precisely by ICPMS. The information obtained is also very useful to reveal the corrosion mechanism. Samples will be taken after each irradiation.

10) Auger Electron Spectrometry

AES has a spatial resolution as small as 100 nm and can yield the information from several atomic layers at a surface. It is an ideal method to analyse chemical changes in grain boundaries. The Auger devices at CIEMAT can be used for low active samples. Few samples are planed to be analysed there.

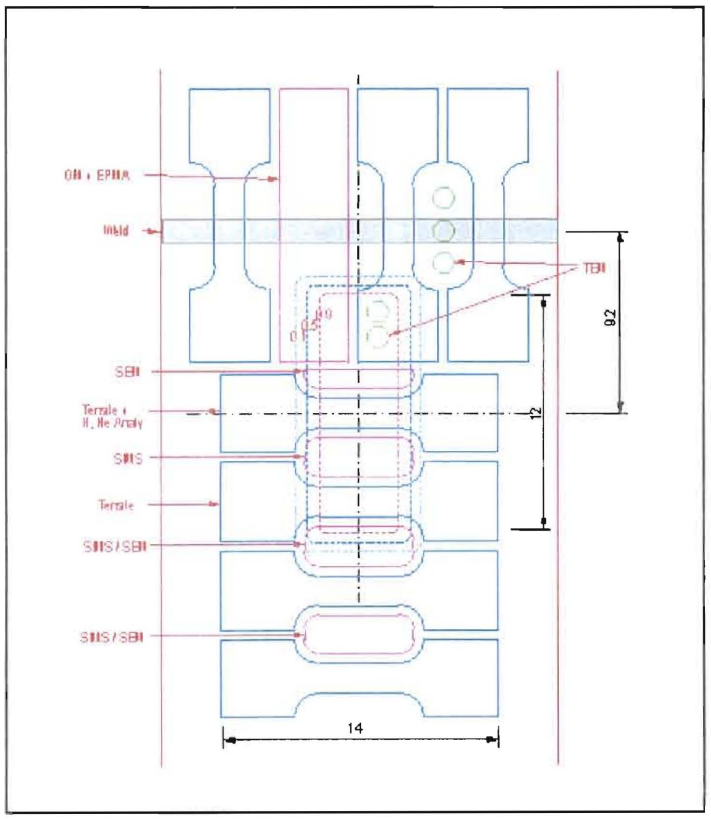


**Figure 8.:** Contour of the different samples to be cut for the PIE with their corresponding analysis method.

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