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Twin Astir: An irradiation experiment in liquid Pb–Bi eutectic environment

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ABSTRACT

The Twin Astir irradiation program, currently under irradiation in the BR2 reactor at SCK.CEN is aimed at determining the separate and possibly synergetic effects of a liquid lead bismuth eutectic (LBE) environment and neutron irradiation. It will lead to a parameterisation of the key influencing factors on the mechanical properties of the candidate structural materials for the future experimental accelerator driven system (ADS). The experiment consists of six capsules containing mainly mini tensile samples and one capsule containing mini DCT's (disc shaped compact tension specimens). Three of the tensile containing capsules and half of the DCT containing capsule are filled each with approximately 20 ml of low oxygen (10^{-6} wt%) LBE. To complete the filling of these capsules with LBE under controlled conditions a dedicated filling installation was constructed at SCK.CEN. The other three tensile containing capsules are subjected to PWR water conditions, in order to discriminate the effect of PbBi under irradiation from the effect of the irradiation itself. To extract the effect of the PbBi corrosion itself on the material properties, one of the capsules is undergoing the thermal cycles of the BR2 reactor without being subjected to irradiation. This results in a matrix of three irradiation doses in LBE (0, 1.5 and 2.5 dpa) and two environments (PbBi and PWR water conditions). Here we will present the detailed concept and the status of the Twin Astir project, describe the materials under irradiation and report on our experience with the licensing of the experiment.

1. Introduction

Lead bismuth eutectic is selected to be both coolant and spallation target material for the future experimental accelerator driven system (MYRRHA) [1,2]. This new concept of reactor type might be one of the possible solutions for the nuclear waste problem as it is conceived to be able to burn up high level radioactive waste and long lived actinides [3].

The ADS technology, however, requires special operating conditions. The materials need to withstand temperatures ranging between 200 and 550 °C under high neutron flux and in contact with the liquid lead bismuth eutectic (LBE). This liquid metal contact does not only result in liquid metal corrosion but might also facilitate liquid metal embrittlement [4].

While both the liquid metal embrittlement phenomenon and liquid metal corrosion in lead bismuth eutectic (LBE) are currently widely under investigation by numerous laboratories around the globe [5–13], little is known about the possible synergy between irradiation and liquid metal corrosion and embrittlement. There is a lot of experience in the field of liquid sodium from the fast reactor community, however, the current knowledge on liquid lead bismuth technology is still rather scarce and mostly limited to the military experience of the Russians [14].

Materials are known to undergo irradiation hardening when subjected to neutron irradiation which may make them prone to liquid metal embrittlement as it was observed by Vogt et al. when the material was hardened by a dedicated heat treatment [13]. Few tests in LBE have been performed on materials irradiated under PWR conditions [15], however, there is no data available on the possible synergetic effect of the irradiation hardening and the liquid metal environment of materials irradiated in contact with LBE.

The environmental and therefore also licensing problems caused due to the creation of Po^{210} when irradiating Pb–Bi (neutron caption of Bi^{209}) do not facilitate the progress of the lead bismuth expertise in an active environment.

SCK.CEN is one of the candidate sites to actually build the XT-ADS and has therefore also engaged itself within the European FP6 programme EUROTRANS to further investigate the influence of liquid lead bismuth eutectic on the candidate structural steels in a neutron irradiation environment.

The irradiation experiment called Twin Astir, which stands for ADS Steel T91 irradiation up to two doses (hence Twin) is the first

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of its kind and started receiving neutron irradiation during the second cycle, 2006 of BR2 in April 2006. The knowledge and expertise that was built up in the MEGAPIE initiative, which received its first proton beam on target only a few months later, was of great importance and facilitated the licensing and realisation of Twin Astir.

The Twin Astir experiment has the purpose to distinguish and quantify the most important influencing parameters on the liquid metal corrosion and/or embrittlement in LBE and to examine possible synergetic effects of these parameters.

In this paper, the concept and realisation of the Twin Astir irradiation project will be explained as well as the licensing issues that influenced the design. The materials under irradiation will be described and finally a brief overview of the current status of the experiment as well as of the issues still to be resolved will be explained.

2. Concept and purpose

The concept of the Twin Astir experiment is a parameterisation study of the most important degradation phenomena of the structural material for the future accelerator driven system at 350 °C. The future ADS is expected to have a window of operational temperature (core inlet–outlet) between 300 and 550 °C. The lower boundary of the heavy liquid metal coolant core inlet temperature under operation is a very important input for the design. However, this leads to discussion among material scientists due to the known irradiation hardening problems at 300 °C [4]. For this reason the irradiation temperature of the Twin Astir programme was chosen to be on the lower temperature boundary for operation. The parameters that are thought to be dominating in the material degradation process are irradiation hardening (accumulated dose), liquid metal corrosion, liquid metal wetting and liquid metal embrittlement. These will be assessed by performing corrosion examinations and tensile tests, crack growth tests both in inert environment as in liquid lead bismuth eutectic environment.

Thus it is expected to gain a better idea of the key influencing factors and possible synergies between these factors on the material degradation in a liquid lead bismuth environment under irradiation. This may allow us to focus on replacing or inhibiting the weakest link in the chain of materials degradation and therefore possibly increasing the operation lifetime of the accelerator driven system (ADS) facility.

3. Safety and design

The irradiations were to be performed at 300 °C up to a fluence of 1.2 and 2.5 dpa. A large part of the desired irradiation conditions were compatible with what the PWR loop in BR2 (CALLISTO) can provide. Therefore, it was decided to perform the irradiations of Twin Astir inside this loop. The samples are inserted in open tubes if they are irradiated under PWR conditions and in closed tubes if they are irradiated in lead bismuth eutectic. As we will explain in detail further on, however, the necessity to use double walled capsules out of safety considerations for the irradiation in contact with LBE increased the irradiation temperature by 50 °C. This temperature increase is caused by the gamma heating of the samples and the capsule itself and the subsequent temperature gradient over the gap between the inner and outer tube of the capsule. Because the PWR water temperature in CALLISTO could not be lowered due to other experiments running at the same time, the overall irradiation temperature for both open (PWR conditions) and closed (LBE filled) tubes of the Twin Astir experiment was set to be 350 °C.

The IPS2 of CALLISTO gives an accumulated dose of 0.25 dpa per cycle or 1.25 dpa/year which results in an irradiation period of one year for the low dose and two years for the high irradiation dose. Furthermore it was desired that the dose difference between the samples in the experiment would not exceed 15%. For this reason the length of the stack of samples needed to be restricted to a maximum length of 300 mm. The limited neutron dose difference across the length of the capsule and relatively small length of the capsules also result in a negligible temperature difference across the stack off samples.

The issue remained, however, that LBE and especially its activation product polonium are not well known materials. This is particularly true for the Po^{210}, which is difficult – and dangerous – to handle and is not available in quantities suitable for the accurate determination of its properties [16]. Therefore, the conditional tense is widely used in what follows.

Likewise, PbBi is not a usual reactor coolant. Before it gained a renewed interest for ADS-related studies, the only practical experience came from the Russian nuclear submarines programmes and therefore references are scarce in the open literature. Basically, in terms of the licensing of the experiment there were three major concerns being the corrosion of steels, the volume change of the LBE after solidification and the activation of Bi^{209} into Po^{210}.

Concerning the corrosion it is well known that the LBE preferentially dissolves the nickel in stainless steels. However, the dissolution kinetic is slow below 600 °C and the native oxide layer of the steels should be able to protect them during irradiation.

The volume change of LBE, however, was considered to possibly pose a problem in terms of the structural stability of the irradiation capsule as the capsule loaded with LBE needed to undergo freezing–defreezing cycles with the BR2 operating cycles. This volume change of the LBE upon cooling has been studied quite extensively [17–19] and depends on the solidification and cooling profile. The faster the cooling, the more pronounced the expansion. To experimentally verify the risk posed by this volume change of the LBE several freezing–defreezing cycles were performed on a mock-up capsule. Additionally another installation similar in volume and shape to the capsules to be used in the irradiation was left idle for two months to allow the possible problem to develop. No permanent deformation or crack of the PbBi container was ever noticed.

The integrity of the irradiation capsules was considered crucial by the licensing authorities due to the presence of Po^{210} after irradiation. This pure alpha-emitter could pose a very serious health hazard in case the capsule would be breached. Polonium is supposed to be volatile. However, research conducted in the frame of the ADS programmes found that, in PbBi, Polonium forms lead polonide that remains in solution in the bulk and is not released until a temperature of about 600 °C is reached [20]. Above 600 °C, all the stored Po is released abruptly, but this is not a situation which could occur in CALLISTO.

However, regarding the possibility of volume expansion causing cracking of the capsule and resulting in a possible Po contamination, the capsule needed to have a double wall. In the worse case scenario, that is the failure of both tubes of a capsule the Po would come into contact with the PWR water of the CALLISTO loop.

In presence of water, the Po is expected to form the volatile and unstable PoH_2 [20]. At the process temperature, PbBi is liquid and Po should be mobile within the liquid metal. There is the possibility that PoH_2 would be formed at the interface between a droplet of PbBi/PoPb and CALLISTO water. The PoH_2 would then be taken away in the water stream, where it could decompose further away. After decomposition, Po could be adsorbed on walls, or integrated in crud, or could reform another PoH_2. Ultimately, Po would be transported everywhere, including to the Surge Tank where the hydrogen gas bubbling will likely extract the volatile PoH_2 from the water and mix it with the air stream of the non-recyclable ventilation. So, there is a possibility that the contamination would be spread across the whole building ventilation and outside, in the environment. As Po is a pure alpha-emitter, there is little way to detect it specifically at an early stage and prevent the spreading, e.g. by isolation of the Surge Tank.

The thermochemical properties of Po are, however, not sufficiently known to assess its chemical behaviour with a reasonable degree of certainty [22–25]. Furthermore, there is no such thing as stable Po which could be used to perform measurements or tests. Therefore, it is not possible to predict the behaviour of the Po contamination of liquid PbBi in presence of water and it is not possible to exclude the scenario described in the above paragraph [21].

Hence, it is difficult to predict which proportion of Po could be released in the water. This is why we opted for the double wall solution. The double wall – with monitoring of the pressure of the intermediate space – allows a permanent control of the integrity of the capsule. A leak – be it on the inner or the outer capsule – will be detected and adequate action can be taken in time, that is before the second barrier fails.

If any permanent deformations of the capsule walls would be induced by the cooling of the LBE, the expansion could possibly modify the temperature control of the capsule by reducing the gap thickness but the influence would be limited to the decrease of the irradiation temperature by a few degrees.

By using double wall capsules, the release of Polonium in CALLISTO water – and further – is ruled out as design basis accident.

The experiment consists of six capsules containing mainly mini tensile samples and one capsule containing mini DCT's. Three of the tensile containing capsules and half of the DCT containing capsule are double walled and filled each with approximately 20 ml of low oxygen (10^{-6} wt%) LBE. The final concentration of oxygen in the capsule, however, can differ from 10^{-6} wt%, as it depends on when a thermodynamic stability of the system will be reached. There will be a thermodynamic balance or equilibrium between the dissolution of oxygen in the LBE and the reduction or oxidation of the oxide film on the stainless steel surfaces. Moreover, the solubility of oxygen and other chemical elements in the LBE is not constant during the irradiation experiment due to the fluctuations in temperature of the LBE caused by the cycles of the BR2 reactor. The final oxygen concentration is therefore difficult to predict.

To complete the filling of these capsules with LBE under controlled conditions a dedicated filling installation was constructed. The other three tensile containing capsules are foreseen with holes to allow the PWR water to enter and flow thru. Thus these capsules are subjected to PWR water conditions, in order to discriminate the effect of PbBi under irradiation from the effect of the irradiation itself. To extract the effect of the PbBi corrosion itself on the material properties, one of the capsules is undergoing the thermal cycles of the BR2 reactor without being subjected to irradiation.

The tensile filled capsules are designed to contain mostly tensile specimens which are screwed in a retaining plate in an arrangement of three specimens per stage. One stage is schematically represented in Fig. 1 showing the three tensile specimens screwed in

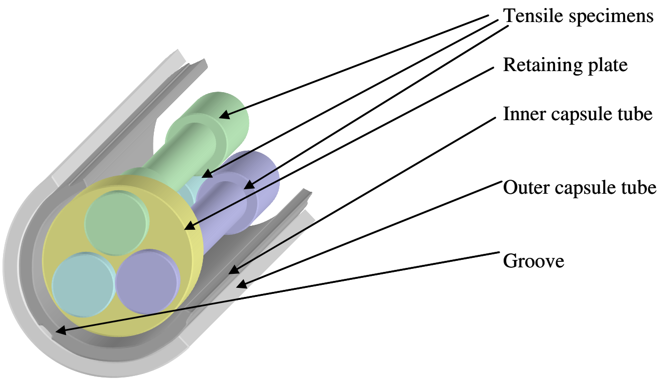

Fig. 1. Schematic representation of tensile samples and retaining plate inside double walled capsule.

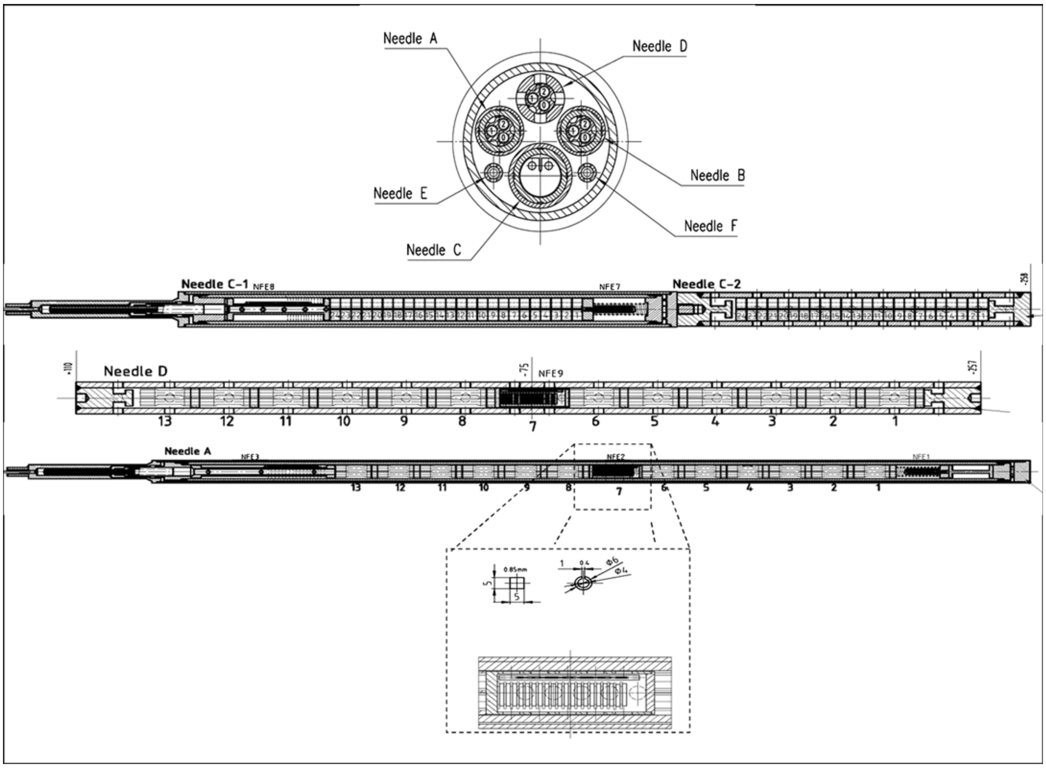

Fig. 2. Design drawings of the capsules of Twin Astir. Cross-section showing the position of each capsule (left); capsule with CT specimens (upper right). PbBi filled section on the left and open section in contact with PWR water on the right; capsule for tensiles open to PWR water (middle right); and capsule for tensiles in contact with PbBi (lower right).

their retaining plate inside the double walled capsule. The drawing of the capsules is illustrated in Fig. 2 showing both types of tensile capsules (middle and lower right) as well as the CT filled capsule (upper right). The tube containing the corrosion plates is being magnified in the figure. Due to the vertical position of the capsule in the reactor, the corrosion plates would be pressed together by the buoyancy forces preventing adequate liquid metal contact. To ensure contact with the LBE, spacers (rings provided with an opening) are placed in between the plates. The CT filled capsule consists of two parts. Half of the capsule containing CT's is in contact with PWR water, the other half, also containing the same amount of CT's is closed, filled with LBE and foreseen of a double wall. Because the PbBi filled capsules are submitted to a strong external pressure of the PWR water, they were pressurised at room temperature by 50 bar of helium. The pressure control of the LBE filled capsules will be discussed more in detail further on.

There are several materials being studied in the Twin Astir experiment, being the most important structural material candidates for ADS, T91 and 316L as well as their welded joints (TIG and EB) and four experimental Si-enriched steels. Different samples of these materials are stacked in an identical order for each of the capsules.

4. Neutronics

MCNP calculations of the detailed axial distributions of the neutron fluxes and gamma heating were made based on the design of the needles of Twin Astir located in the IPS2 channel of CALLISTO and the dpa rates, production of Po^{210} and the reactivity effect were evaluated. The calculations were performed for the full scale 3D heterogeneous MCNP & ORIGEN-S model of BR2 with detailed 3D fuel depletion distribution in the fuel elements.

The calculated accumulated dpa for each of the capsules based on 10 operating cycles of BR2 are given in Table 1. Note that needle A will be removed after only 6 cycles. The production of Po^{210} in these needles after 10 cycles of irradiation was estimated to be around 0.01 g using MCNP. This amount of Po corresponds to a total activity of 1.66×10^{12} Bq or, given a total PbBi volume of 67 cm^3, the volumic activity is 2.48×10^{10} Bq/cm^3. The axial distribution of the fast and the thermal neutron flux for needle A as well as the axial distribution of the gamma flux for each needle are depicted in Fig. 3.

Table 1
Calculated accumulated dpa in the needles of Twin Astir based on 10 BR2 operating cycles

Needle A (%)	Needle B (%)	Needle D (%)	Needle C1 (%)
2.68 ± 2.3	2.67 ± 2.3	2.82 ± 2.2	2.46 ± 1.6

Values for the regions around the mid plane of the reactor core given in dpa.

5. Temperature and pressure control

With a double wall, there is an additional resistance to heat transfer that needs to be taken into account. The high gamma heating, in the range of 4 W/g for IPS 2, does not make things easy. Hence, the tubes are matched as close as possible, coming possibly into contact at several spots. Four grooves, 0.25 mm deep were machined in the inner tube to facilitate the control of the gap. Even if the capsule tubes are machined in order to get a close fit, the tolerances leave a thin gap between the tubes (see Tables 2 and 3). According to the tolerance range, the gap thickness varies between 0.01 and 0.05 mm. Due to further circularity and cylindricity errors, the gap could close at some locations. Hence, the temperature gradients across the capsule tubes will vary according to the gap thickness, as indicated below in Table 2. In addition, there is a gradient at the interface tube/CALLISTO water (forced convection in CALLISTO water) and another 5 °C temperature gradient within the capsule (gamma heating of the materials contained by the inner tube). The results of the heat transfer calculations are summoned in Table 2 for the tensile containing capsule and in Table 3 for the CT containing capsule.

As the capsules temperature control does not rely on the fluid heat conductivity for heat transfer, the 'closed gap' technology makes the system insensitive to the fluid used for the intermediate room control. Therefore the control fluid could be gas, despite its low thermal conductivity. In this case, the integrity control will be performed on the pressure in the intermediate room between the inner and the outer capsule tube. Three pressure sensors (one per capsule) are installed in the instrumentation head to monitor the capsule integrity.

When heating a given volume of gas from 20 to 310 °C, the pressure increases by a factor 2 as calculated in the following equation (the volume remains almost constant):

$$\frac{p_1}{T_1} = \frac{p_2}{T_2}, \quad \text{thus} \quad p_2 = p_1 \cdot \frac{273.15 + 310}{273.15 + 20} \cong 2 \cdot p_1. \qquad (1)$$

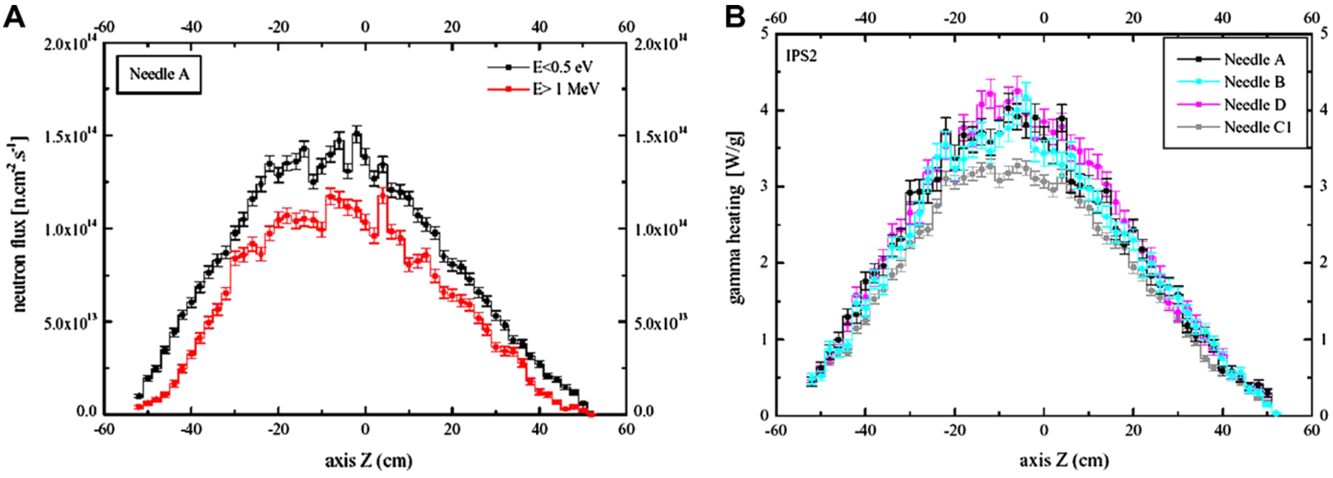

Fig. 3. Axial distribution of neutronics; A: axial distribution of the fast and thermal neutron flux (shown for needle A – other needles are comparable) and B: axial distribution of the gamma flux.

Table 2
Calculation of the temperature gradient across the tensile loaded capsule tube based on the gap thickness

Temperature gradients across the capsule Tensile-loaded capsule	Hot plane Contact	 Minimum gap	 Average gap	 Maximum gap
Geometry data				
Inner tube inner diameter (mm)	10	10	10	10
Inner tube thickness (mm)	2	1.89	1.88	1.88
Inner tube outer diameter (mm)	14	13.78	13.77	13.76
Gap thickness (mm)	0.000	0.110	0.125	0.140
Outer tube inner diameter (mm)	14	14	14.02	14.04
Outer tube thickness (mm)	1	1	0.99	0.98
Outer tube outer diameter (mm)	16	16	16	16
Tubes Δt				
Inner volume Δt (°C)	10.3	10.3	10.3	10.3
Inner tube Δt (°C)	11.1	10.4	10.3	10.3
Gap Δt (°C)	0.0	32.0	36.9	41.8
Outer tube Δt (°C)	4.1	4.1	4.1	4.0
Δt convection (°C)	1.9	1.9	1.9	1.9
Tubes Δt (°C)	17.1	48.4	53.2	58.0
Total Δt	27.4	58.7	63.5	68.3

Table 3
Calculation of the temperature gradient across the CT loaded capsule tube based on the gap thickness

Temperature gradients across the capsule CT-loaded capsule	Hot plane Contact	 Minimum gap	 Average gap	 Maximum gap
Geometry data				
Inner tube inner diameter (mm)	14	14	14	14
inner tube thickness (mm)	2	1.99	1.98	1.97
Inner tube outer diameter (mm)	18	17.98	17.96	17.94
Gap thickness (mm)	0.000	0.010	0.030	0.050
Outer tube inner diameter (mm)	18	18	18.02	18.04
Outer tube thickness (mm)	1.5	1.5	1.49	1.48
Outer tube outer diameter (mm)	21	21	21	21
Tubes Δt				
CT Δt (°C)	18.70	18.70	18.70	18.70
Inner tube Δt (°C)	13.3	13.3	13.2	13.1
Gap Δt (°C)	*0.0*	*5.7*	*17.0*	*28.2*
Outer tube Δt (°C)	12.5	12.4	12.3	12.2
Δt convection (°C)	2.38	2.37	2.36	2.36
Total Δt in capsule tubes (°C)	25.8	31.4	42.5	53.5
Total Δt with Δt in CT specimens (°C)	46.9	52.4	63.6	74.6

The inner PbBi capsules could be pressurised to 2.5 MPa (cold condition), resulting in a pressure of 5 MPa in hot condition whereas the intermediate room is inflated to 5 MPa, becoming 10 MPa in hot condition. This inner pressure is sufficient to counter the external pressure applied to the outer tube by CALLISTO (0–1 MPa in cold condition and 15.5 MPa in hot condition).

In order to get an efficient leak detection, i.e. a significant pressure change in case of leak, the intermediate volume should be small compared to the inner volume (half the volume would be nice). In practice, the control volume should be built as close as possible to the PbBi capsule.

In case of a leak of the inner tube, considering a volume $V1$, filled at $P1 = 5$ MPa, in communication with a volume $V2 = 0.5 \cdot V1$, filled at 10 MPa, the pressure at equilibrium is +/−6.6 MPa. As the helium molecule is very thin, it should be able to find the leak path even in the ‘closed’ gap. The pressure change could, however, be very gradual.

A leak of the outer tube would obviously increase the pressure to 15.5 MPa, which is unmistakable.

6. LBE filling

For the filling of the capsules with PbBi a dedicated filling installation was designed and built at SCK.CEN. As oxygen content is the key parameter for corrosion control in PbBi systems, particular emphasis was put on this aspect during the filling procedure. The filling installation is schematically represented in Fig. 4 and is composed of a PbBi conditioning tank in which the PbBi is melted and its chemistry set to the required O_2 concentration (10^{-6} wt%), a vacuum pump, a set of heaters, which have to keep the whole installation at or above 150 °C, to prevent the freezing of PbBi during the filling process, and a filling connection, connecting the tank, the capsule to fill and the vacuum pump (see Fig. 4).

The filling connection itself is schematically represented in Fig. 5. The purpose of this system was to allow the evacuation of the capsule, its bakeout, and it’s filling without opening to the atmosphere and contamination of the PbBi by air.

The filling needle is inserted in the filler neck of the capsule to avoid any risk of contamination of the latter by the PbBi, which would prevent the execution of a tight sealing weld. The filling needle is withdrawn after filling. With adequate isolation valves, as shown in Fig. 4, the conditioning and PbBi filling could be performed in air without contamination of the PbBi melt. The capsule would then be disconnected from the conditioning

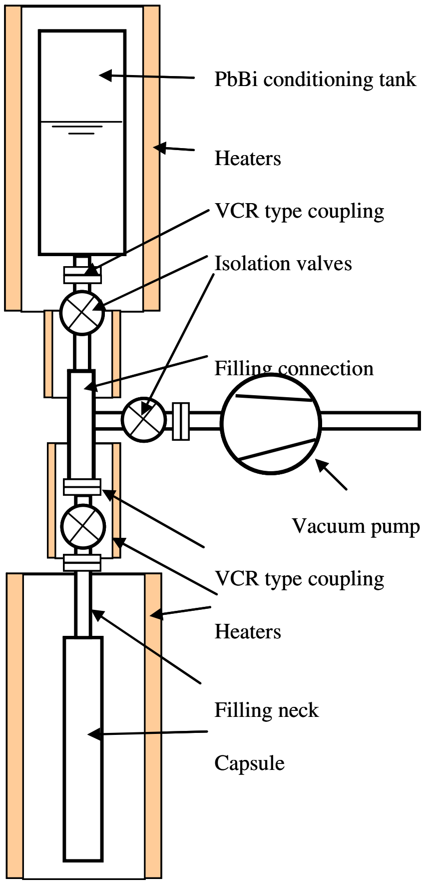

Fig. 4. Schematic representation of the PbBi filling system for the Twin Astir capsules.

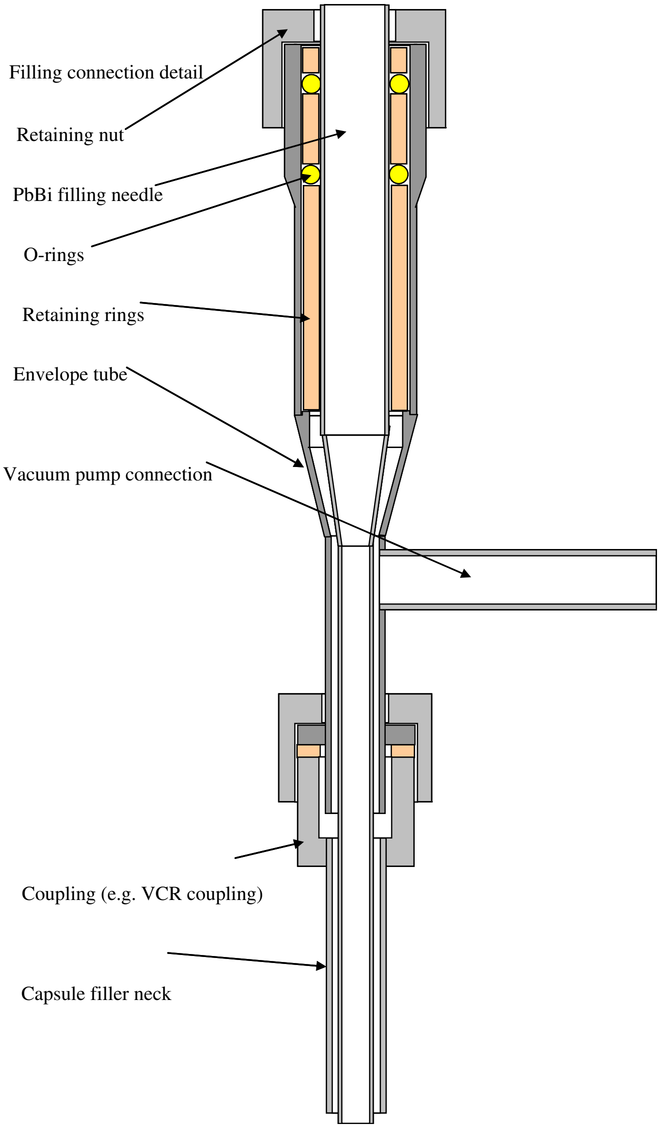

Fig. 5. Schematic representation of the filling connection (detail of Fig. 4).

Table 4
Chemical composition of the LBE used in the Twin Astir programme

	Sample 001	Sample 002
Bi (%)	54.3%	54.3%
Pb (%)	45.7%	45.7%
Cu (μg/g)	3.6	4.6
Ag (μg/g)	13	13
Sn (μg/g)	4.2	4.3
Tl (μg/g)	2.3	2.2
Na (μg/g)	<50	<50
Ca (μg/g)	<250	<250
Cr (μg/g)	<10	<10
Fe (μg/g)	<100	<100
Ni (μg/g)	<5	<5
Mo (μg/g)	<1	<1
Cd (μg/g)	<1	<1
Th (μg/g)	<0.5	<0.5

tank and the vacuum pump and brought into a glove box where it is pressurised and seal welded. The chemical composition of the LBE used in the Twin Astir programme is given in Table 4.

7. Status of the Twin Astir experiment

The Twin Astir programme is currently under irradiation in the Belgian BR2 reactor at SCK.CEN and has successfully been irradiated for 4 cycles. The capsules tightness, more precisely the intermediate space of the double walled capsule used for the integrity surveillance, has proven very difficult to maintain. For this reason two of the capsule were re-inflated to raise the pressure after the second cycle. No polonium was detected during the process which implies that the inner capsules were still intact.

8. Open issues for the continuation of the work

Even though the experiment is currently under irradiation many questions remain unsolved and a certain amount of open issues will need to be tackled to facilitate the post irradiation examinations (PIE). Due to the very strict legislation concerning Po^{210} and the uncertainty regarding the quality of the LBE after irradiation issues such as the secure retrieval of the samples in the hot cell and the decontamination of polonium contaminated materials need to be resolved.

At SCK.CEN a hot cell was designed and built, dedicated to performing mechanical tests in heavy liquid metal environment under well controlled temperature and LBE chemistry conditions [26,27]. This facility is currently licensed to perform tensile tests in PbLi environment but still needs to be licensed for tests in PbBi environment. Additionally tensile tests as well as crack growth tests are foreseen to be performed using this facility in both inert gas and LBE environment. Regarding the crack growth measurement in LBE adequate measurement techniques are currently under development.

9. Conclusions

The Twin Astir experiment has been successfully designed, realised and implemented at SCK.CEN and is currently under irradiation in BR2. The programme has a relatively large test matrix and is foreseen to examine the separate and combined effects of LBE exposure and neutron irradiation by tensile testing, crack growth measurements and corrosion examinations. The production of Po^{210} due to the irradiation of bismuth forced the implementation of double walled capsules for the irradiation and severely complicates the handling of the material for PIE. Issues regarding the licensing of the hot cell testing facility to work with polonium contaminated materials in an LBE environment remain to be solved.

Acknowledgements

Projects such as this are obviously the work of a group of people. Therefore I would like to take the opportunity to thank all people who participated in the realisation of the Twin Astir irradiation. Special thanks go to Philippe Benoit, the project engineer of Twin Astir but of course also to all technicians who worked on the sample preparation and the rig assembly. A special word of gratitude is also in place to all people in SCK.CEN's management who made it possible to give the necessary priorities to this project.

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