# EUROPEAN RESEARCH AND DEVELOPMENT PROGRAMME FOR WATER-COOLED LITHIUM-LEAD BLANKETS PRESENT STATUS AND FUTURE WORK

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## ABSTRACT

The European R&D programme in support of the development of water-cooled Pb-17Li blankets for DEMO aims at improving the data base concerning tritium behaviour and compatibility between blanket materials. The four main areas of the experimental programme are structural material corrosion by Pb-17Li, tritium extraction and permeation control, Pb-17Li physico-chemistry, and water/Pb-17Li interaction. This paper describes the most significant results obtained to date in the various experiments performed in Europe and the future programme required to complete the data base by 1994. (28 ref.)

## 1. INTRODUCTION

The European Test-Blanket development programme, started in 1988, is aimed at the selection by 1995 of two blanket designs to be tested in the next generation tokamak (NET/ITER), one using solid and the other one using liquid breeder materials. In order to solve the many existing uncertainties in the material data base, the implementation of a large experimental programme has been required.

As far as the liquid breeder blankets are concerned, two families of blankets are being studied in Europe, the water-cooled lithium-lead (liquid eutectic Pb-17Li) blankets[1,2] and the self-cooled Pb-17Li blankets[3]. The required experimental programs are different for the two families although they can partly overlap in cases concerning basic Pb-17Li data or Pb-17Li/structural material (martensitic steel MANET) compability data.

This report concerns the experimental programme in support of water-cooled Pb-17Li blanket designs. Four main areas are identified :

1/ Corrosion : the quantification of the corrosion rate of steels (MANET and 316L) by Pb-17Li as a function of the interface temperature and Pb-17Li velocity fixes an upper limit for these design parameters,

2/ Tritium Extraction : the definition of an efficient method of tritium extraction from Pb-17Li is necessary for limiting the T-inventory and permeation within the blanket, and for establishing tritium management procedures. In particular, the limitation of tritium permeation towards the water-coolant is a critical issue for this type of blanket ; therefore the development and testing of permeation barriers is of high priority.

3/ Pb-17Li physico-chemistry : comparison and validation of techniques permitting the purification of Pb-17Li, and the on-line Li-content measurement and adjustment (compensation for Li burn-up),

4/ Pb-17Li/water interaction : the effects of large breaks in water-coolant pipes and the evolution of small leaks (wastage) need to be established for safety, reliability, and availability concerns.

In the next sections, the state of the art and the future working program will be given for all the four areas.

## 2. STRUCTURAL MATERIAL CORROSION BY Pb-17Li

### 2.1. Present Status

Corrosion tests were carried out on two types of structural materials, martensitic steels and 316L austenitic steels (the latter being considered for the cooling tubes), and also on recently developed permeation barriers.

**2.1.1. Martensitic steels.** Corrosion tests carried out in different laboratories and in particular in the CAMILLE loop[4] (at 475°C) have shown an homogeneous dissolution with a linear kinetics and indicated a strong increase of the corrosion rate with the Pb-17Li velocity, passing from 20 μm/y to 100 μm/y when the velocity increases from 2 to 20 cm/s. All tested martensitic steels (1.4914, T91, and EM10) have shown similar behaviour. An equation giving the corrosion rate as a function of Pb-17Li-velocity and temperature[5] has been derived from the compilation of all experimental data obtained in various laboratories (CEA, KfK, ORNL). Various tests carried out at CEA in semi-stagnant Pb-17Li have confirmed the effects of Pb-17Li velocity on corrosion rate.

Experiments in which 1.4914 specimens were subjected to a constant load indicated neither increase in the corrosion rate nor evidence of stress corrosion cracking. Embrittlement by Pb-17Li appears only in simulated welded conditions if the appropriate post-weld heat treatment is not carried out[6].

No effect due to hydrogen loading of 1.4914 specimens has been observed after 2,000 h in Pb-17Li at 450°C. Slow strain rate tests carried out at 250°C in Pb-17Li with an hydrogen atmosphere on notched tensile specimens have confirmed this tendency[7].

**2.1.2. Austenitic steels.** Whatever the thermo-hydraulic conditions and exposure limits, austenitic stainless steel (316L) exhibits a porous ferritic superficial corrosion layer containing a network of Pb-17Li channels. The chemical analysis of this layer shows a strong chromium, nickel, and manganese depletion. Corrosion kinetics appear to be linear after an incubation period and depends on temperature but not on the Pb-17Li velocity as long as the flow remains turbulent. In fact, at 450°C, when the state of semi-stagnant Pb-17Li is reached a reduction of the corrosion rate has been observed[8].

Concerning the influence of magnetic field, a first CELIMENE test[9] carried out at 450°C in semi-stagnant conditions has shown an increase of the corrosion rate especially when the Pb-17Li/steel interface is parallel to the magnetic field. Moreover, a dissymetry in the corrosion product deposition rate seems to be induced by the magnetic field. On the other hand, this influence of magnetic field has not been observed in corrosion tests carried out in other experiments, such as the MALICE loop, with flowing Pb-17Li[10].

No changes on corrosion rate and mechanical properties have been observed when 316L specimens have been loaded with constant or cyclic uniaxial stress[11,12]. Exhaustive tests carried out just above the Pb-17Li melting point revealed that 316L was not subject to embrittlement even when the presence of hydrogen is simulated[11].

The LIFUS2 loop has been designed and constructed by ENEA in order to investigate the low cycle fatigue behaviour of 316L in flowing Pb-17Li. Preliminary tests have been carried out to optimise the geometry of the test samples and to evaluate the performance of a prototype extensometer able to operate at high temperature in presence of Pb-17Li.

**2.1.3. Permeation barriers.** The compatibility of two types of permeation barriers under study at CEA, obtained respectively by aluminization and by oxidation of 316L and 1.4914 steels, has been investigated in quasi-stagnant Pb-17Li at 500°C in isothermal and non-isothermal conditions[13]. The corrosion rate of aluminized specimens, especially of those made of 316L, is very low. On the other hand, the oxide layer present on the surface of the steel specimens was completely dissolved after 1,500 h at 500°C. The aluminized specimens are now being tested in flowing Pb-17Li.

Another type of permeation barrier has been developed at JRC/Ispra. This barrier is obtained by controlled oxidation of 316L in $H_2/H_2O$ atmosphere at 800°C to create a Cr/Mn oxide. This oxide then reacts on exposure to Pb-17Li to form a $LiMn_2O_4$ type oxide, in which chromium occupies some of the manganese sites, and which is stable in Pb-17Li. Preliminary results of corrosion tests in a Pb-17Li filled thermal convection loop indicate that the corrosion rate of the coated steel is a factor 2 to 4 lower than that of AISI 316L[14].

Permeation barrier characterization from the T-permeation point of view will be discussed in section 3.

**2.2 Discussion and Future Work**

Compatibility of Pb-17Li with both austenitic and martensitic steels is now well assessed. Under the temperature and velocity conditions typical for water-cooled Pb-17Li DEMONET2 blankets requiring 20,000 h of continuous operation, the corrosion rate remains within acceptable limits also when the magnetic field is taken into account. In particular for MANET, with a Pb-17Li velocity lower than 1 cm/s, a corrosion rate not greater than 10 μm/y is expected if the maximum interface temperature is limited at 475°C. However, effect of magnetic field on corrosion rate has been tested only for 316L samples. Experiments on MANET samples are in progress in order to complete the program in this area.

Limited results are now available about deposition phenomena. They indicates that these phenomena cannot be easily modelled but their understanding is important to avoid plugging. Experimental work is in progress to simulate DEMONET conditions.

As far as permeation barriers are concerned, tests under flowing Pb-17Li are still in progress and will be completed soon. The results of experiments in stagnant Pb-17Li seem promising for the aluminized samples and for the ternary oxide (Li-x-O) coatings.

The most promising permeation barriers will be tested, both on MANET and on 316L supports, for checking their resistance to thermo-mechanical fatigue, assuming typical water-cooled Pb-17Li DEMONET blanket conditions.

## 3. TRITIUM EXTRACTION

### 3.1. Present Status

In the field of tritium extraction different research subjects are included. They have the common objective of enabling to fully recover the tritium produced in the blanket and to re-inject it into the fuel cycle. The main subjects are tritium desorption and release from Pb-17Li, tritium permeation through steels and barriers and tritium extractor design. Required tests include both out-of-pile and in-pile experiments.

**3.1.1. Kinetics of desoption.** The kinetics of deuterium desorption from Pb-17Li to an helium gas stream at different temperatures (360 to 450°C) and D-partial pressures have been experimentally determined[15]. In the used experimental conditions, the overall process of D-desorption from Pb-17Li is governed by both the diffusion of D-atoms in Pb-17Li and the heterogeneous reaction at the gas/Pb-17Li interface of D-atom recombination.

**3.1.2. Test of extractors.** In order to determine the kinetic parameters of T-transfer to be used for evaluating the performances of various types of T-extractors a new experimental Pb-17Li loop, the MELODIE loop, has been designed and constructed at CEA. In this loop tritium will be simulated by H or D. In order to avoid H-leakage through the walls, the loop has been internally aluminized. The blank test being carried out before the introduction of the Pb-17Li has demonstrated the efficiency of this type of permeation barrier. During these tests the H-meter, that will be used to monitor the H-contents within Pb-17Li, has been tested in $Ar/H_2$ mixture. The ongoing tests examine two kinds of extractors (plate column at first, and later bubble column).

**3.1.3. Permeation barriers.** The diffusivity, solubility, and recombination rate of H in TiC-coated 316L steel samples have been measured by a gas evolution technique[16]. The solubility of H in TiC is about 200 times higher than in 316L but its recombination constant and its diffusion coefficient are several orders of magnitude lower. It can be concluded that, although the TiC-coating acts in a mixed regime (surface limited and diffusion limited regime), the surface limited regime possibly dominates. Therefore, thin coatings should be used in order to eliminate the inventory problems associated with thick coatings (50 $\mu$m of TiC absorb roughtly the same amount of tritium as 1 cm of steel).

Measurements of the hydrogen permeation rates through aluminized discs (d=48 mm) of 316L and 1.4914 steels have been performed in the temperature range 250 to 600°C and at a pressure of 0.1 MPa[17]. In comparison with uncoated samples, the permeation rates through the 1.6 mm-thick 316L samples were reduced of about three orders of magnitude whilst those for 1 mm-thick 1.4914 samples were reduced by up to four orders of magnitude.

However, the definition of an efficient aluminization process[13] needs some technological developments (long tubes of various diameters, welds, large vessel, ...) which are carried out in cooperation with the French firm HEURCHROME, as it was the case for the coating of the MELODIE loop.

In order to compare the efficiency of various permeation barriers in presence of Pb-17Li, a special device has been developed at JRC/Ispra. The tests concern the determination of the permeation barrier efficiency with H and D at first and finally with tritium in the ETHEL laboratory.

**3.1.4. Irradiations in HFR (JRC/Petten).** The objective of the LIBRETTO experiments (in-pile test of Pb-17Li contained in closed steel capsules) is to assess T-release kinetics and T-permeation through reference 316L-steel with and without permeation barrier coating.

*In the LIBRETTO-2 experiment*[18] the comparison between closed uncoated and aluminized 316L capsules has shown that the presence of an aluminide coating increases the tritium residence time by a factor from 3.0 to 4.5 for temperature of 470°C and 300°C respectively. For temperatures lower than 270°C, no permeation was measured after 200 h. With a large plenum volume, no helium bubble retention was observed in Pb-17Li at the end of the experiment.

In the LIBRETTO-3 experiment, planned for the first half of 1992, three types of coatings will be compared : 1/ TiC coating on the outside of the capsule, 2/ $Al_2O_3$ coating on the capsule inside, and 3/ aluminization of the capsule inside.

**3.1.5. Irradiations in OSIRIS (CEA/Saclay).** The objective of the LIPSIE experiment is to study the influence of the $O_2$-concentration in the pressurized cooling water on the permeation rate of tritium generated in Pb-17Li towards the coolant under DEMONET conditions. For this purpose, the ISABELLE-4 pressurized-water loop, set in the OSIRIS reactor pool, has been adapted[19]. Two tritium control devices have been developed for this experiment :

- continuous measurement of the T-content in helium coming from one of the two extraction lines (sweeping of the plenum or bubbling through Pb-17Li). This analysis is used to adjust the T-content in Pb-17Li to a value representative of the T-concentration within Pb-17Li at the Pb-17Li-outlet of a DEMO blanket module.
- continuous analysis of the T-content of the pressurized water to determine the permeation rate of tritium.

In the LIPSIE irradiation (55 full-power days) the capsule material is 316L. The experiment ended in December 1991, the results are not yet available. As a preliminary indication, it can be said that the addition of oxygen into the cooling water for generating an additional oxide-layer did not lead to a significant reduction of T-permeation.

3.1.6. **Numerical Modeling.** A method relying on the use of a 2D finite element simulation model exploiting the analogy between tritium migration and heat conduction has been developed. It should allow to interpret LIBRETTO-type irradiation experiments in terms of reduction of the T-permeation coefficient of the tested coating.

First tests have demonstrated the potentialities of the model. They seem to indicate that, under conditions comparable with the experiment, the aluminide coating tested in LIBRETTO-2 reduces the tritium permeation rate by about two orders of magnitude[20].

### 3.2. Discussion and Future Programme

Tritium control is a critical issue for water-cooled Pb-17Li blankets because the experimental results show that the T-permeation towards the pressurized water-coolant would be inacceptable if uncoated steels were used (in DEMONET conditions, tenths of g/day for a 10,000 s Pb-17Li residence time) in the blanket.

Therefore, a significant development programme for permeation barriers has been planned for the next two years. It includes tests of existing barriers on MANET support, development of new coatings better adapted to martensitic steels, and definition of specific welding procedures on tubular coated samples.

Apart from the thermo-mechanical and the compatibility tests with Pb-17Li, the new coatings will require further in-pile tests (LIBRETTO-4) and the related numerical modeling.

Finally, tests of different types of T-extractors will be performed and their efficiencies will be evaluated.

## 4. Pb-17Li PHYSICO-CHEMISTRY

### 4.1. Present Status

In order to detect composition changes in Pb-17Li, an electrical resistivity monitor, entirely made of steel, has been developed by Nottingham University[21]. The sensitivity of the monitor is ±0.05at% Li. The same university has also experimentally determined solubility constants in Pb-17Li for Ni, Cr, Mn, Fe, Mo, Zr, and V[22].

An electrochemical sensors based on solid electrolyte and fitted to a data acquisition system is under development at SCK/Mol. The reproducibility of such a sensor is better than 1 r~ which corresponds to 0.21at% Li. An ...ortant effort is done to develop a better metal-... mic seal in order to increase the lifetime of the sensor[23,24].

A plugging indicator, previously developed at CEA/Cadarache for Na-technology, is now being tested using Pb-17Li. The Li-content is expected to be determined from the liquidus temperature measurement.

In order to study problems associated with the extended use of Pb-17Li in a blanket, a physico-chemical loop (ANAPURNA) has been designed and is under construction at CEA. At present, the main identified objectives of this loop are : 1/ comparison of on-line Li-content measurement devices (electrochemical probes, resistivity meter, plugging indicator), 2/ Li-content adjustment, 3/ control of metallic impurities, 4/ comparison of various purification techniques.

### 4.2. Discussion and Future Programme

As previously said a specific loop will be used for determining methods to control the Li-concentration and to keep it on stable levels and to purify the Pb-17Li from reaction products, eventually including those due to self-wastage related to water microleaks from the coolant circuits (see section 5).

Purification tests from the expected presence of Polonium-210 in Pb-17Li, although at low level, require a specific loop able to cope with radioactive elements. These tests will be performed at KfK/Karlsruhe, within the experimental programme for the self-cooled Pb-17Li blanket.

## 5. Pb-17Li/WATER INTERACTION

### 5.1. Present Status

Experiments of Pb-17Li/water interaction simulating a large break in a Pb-17Li blanket module have been performed in the BLAST facility[25,26]. The conclusions of the study are the following :

- simulation of large breaks has shown that mixing was the governing factor in the Pb-17Li/water interaction process
- the BLAST experiments, performed at different injection pressures with a connection tube of 50 mm diameter between the reaction vessel and the expansion system, have shown that the pressurization in the reaction vessel did not exceed the actual water-injection pressure, also in the case where a tube bank was mounted in the center of the reaction vessel to intensify mixing between the melt and the water
- on the other hand, when the connextion tube is throttled, the pressurization phase is more rapid than previously, and the pressure peak in the reaction vessel is higher than the injection pressure for a period of time of about 250 ms (the maximum values are 10 MPa for a 6 MPa injection pressure and 13 MPa for 10 MPa injection pressure)
- the melt temperature increase, from 350°C to 500°C, does not appear to have much influence on the pressurization history of the reaction vessel. Moreover, these measurements show that the maximum temperature increase near the tube bank did not exceed 180°C at 250°C and 100°C at 350°C initial melt temperature
- all BLAST experiments indicate that the chemical reaction is self-limiting, due to hydrogen generation and production of solid LiOH and $Li_2O$, with the melt being partially

insulated from the water so that energetic vapour explosions have never been observed.

The reaction water vapour/Pb-17Li has been studied at Nottimgham University[27] using an electrical resistivity meter. The product of the reaction at 450°C was shown to be $Li_2O$, as expected from extensive thermodynamic calculations.

In order to investigate the evolution of water micro-leaks into Pb-17Li, a special device has been constructed at ENEA. The preliminary results indicate that no significant wastage at the micro-cracks was observed over the test duration (3 hours). Also, in the two tests performed, no blockage of the crack tip by the solid reaction products ($Li_2O$ and LiOH) occured[28].

### 5.2. Discussion and Future Programme

The series of large-break BLAST experiments have demonstrated that a serious accident due to Pb-17Li/water interaction is unlikely ; this leads to the conclusion that a large break in the water circuits can probably be handled and controlled if the blanket is properly designed.

The major problem in this area remains the effect of water microleaks during blanket operation. Up to now, self-wastage has not been observed although the particular experiment lasted only 3 h, as compared with the 20,000 h operation of a DEMONET blanket. Therefore, in the next two years similar experiments will be repeated with an expected duration of about 100 h, in order to permit a better extrapolation of the results to DEMONET conditions.

## 6. GENERAL CONCLUSION

In this paper the European research and development programme in support of the water-cooled Pb-17Li blanket studies has been described. The aim is to build a data base concerning the tritium behaviour and the compatibility between blanket materials by 1994 in order to permit the Test-Blanket design selection in 1995.

The state of the art and the tasks still to be completed have been described for the four main experimental areas, that are material corrosion by Pb-17Li, tritium extraction and permeation barriers, Pb-17Li physico-chemistry, and Pb-17Li/water interactions.

In the case of steel corrosion and Pb-17Li/water interaction due to large breaks the available experimental results appear sufficiently complete. They indicate that the particular problems expected in these areas for water-cooled Pb-17Li blankets can probably be handled with a proper design.

Therefore, the future R&D programme includes experiments mainly in the remaining areas, tritium extraction and permeation barriers development being the area requiring the greater effort. Other items of investigation are the adjustment techiques of the lithium content in Pb-17Li and the Pb-17Li purification from reaction products due to water microleaks.

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