

The chemical compatibility of oxide ceramic candidates in static PbLi for advanced breeding blanket channel components

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ABSTRACT

The use of a PbLi eutectic alloy is extended in the Dual Coolant Lead Lithium (DCLL) and Water Coolant Lead Lithium (WCLL) Breeding Blanket (BB) design concepts in future fusion reactors. However, the reactivity of PbLi is a challenging problem, as the flow liquid metal can damage metallic and ceramic materials. For the development of BB technologies, to conduct chemical compatibility studies of materials that will be exposed to the liquid metal is therefore critical. The advanced DCLL concept considers the use of ceramic channels, which could help to extend the BB operating window above 550 °C while improving the tritium-breeding ratio. Since BB components will be highly exposed to neutrons, the influence of PbLi in neutron-like damaged components is also of high concern as compositional changes may modify properties and functionality of the exposed material. Consequently, selected ceramics were submitted to long-term chemical compatibility experiments using COES, a lab-scale facility located at the Liquid Metal Laboratory at CIEMAT, Spain. Commercial Al₂O₃ ceramics were tested in the as-received and light-ion-implanted conditions, the resultant surfaces after static PbLi exposition being studied by SEM-EDX microstructural and SIMS analytical techniques. The conclusions after the long-term chemical compatibility experiments suggest that the use of bulk alumina ceramics in contact with PbLi is promising. Although lithium diffuses inside the ceramic transforming the initial alumina stoichiometry, the surface structural damage is limited to the first 50 μm in the worst case, and could be still considered as a candidate ceramic material for the advanced DCLL BB.

1. Introduction

In the framework of FP8 and FP9 EUROfusion activities, alumina ceramics are proposed as insulating channel components in the advanced Dual Coolant Lead Lithium Breeding Blanket (DCLL BB) to electrically decouple the PbLi breeding flow from the magnetic field [1]. One of the technical approaches considered is the location of sandwich-like insulating steel-ceramic-steel structures inside the channels, the so-called Flow Channel Inserts (FCI) [2]. However, while simulating the FCI performance at CIEMAT, some functional issues have arisen, favoring the development of new designs in which the steel protection disappears [3]. Evolving since 2018, the new DCLL design aims to balance the optimal tritium reproduction, the secure shielding and the mechanical response of the BB system, among others [4,5,6]. In this advanced design, the insulating ceramic is the main channel component, between the walls of which the PbLi flows in a poloidal direction at moderate velocities (few cm/s). The BB operating window could then be extended above 550 °C, a limit temperature due to the steel corrosion,

with an increase in the TBR. Consequently, the chemical compatibility of the candidate ceramic in direct contact with the PbLi requisites to be assured.

At the same time, the BB ceramic component will be exposed to a continuous flux of neutrons from the plasma. While neutrons are required to guarantee the tritium breeding functionality, they are known to have a detrimental effect on materials, causing radiation damage and transmutation of matrix constituents and impurities, which will modify the material chemistry and physical properties. Furthermore, slow and shallow but effective diffusion of lithium has been observed experimentally in alumina ceramics [7]. Within the solid, neutron transmutation of lithium would generate H-isotopes and He (${}^6\text{Li} + n(\text{slow}) \rightarrow {}^4\text{He} + {}^3\text{T} + 4.8 \text{ MeV}$), altering its crystalline structure and consequently its mechanical stability and functional properties. Besides, during BB operation, the diffusion of bred T from the liquid metal to the materials in contact should not be ruled out, leading to uncontrolled tritium inventory and involving serious safety issues.

One way or another, the presence of H-isotopes and He might be an

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issue in the crystalline structure of insulating ceramic components of the PbLi flow channel. The effect of light ions in alumina materials under conditions expected in fusion facilities has been the subject of study over the last decades [8,9,10]. However, the synergetic effects between H and He ions on the structural and functional properties of alumina together with the prolonged exposure to liquid lithium lead is an issue worthy to be addressed and remains to be elucidated. For this reason, this is the first lab-scale essay achieved to clarify the role of light ions produced by neutron transmutation in materials and their possible involvement in the chemical compatibility experiment with liquid metals.

In order to better understand the behaviour of candidate ceramics, chemical compatibility experiments have been designed to test commercial alumina samples with static PbLi and relevant temperatures. The originality of this work lies in the exposure to liquid metal action of the ceramic implanted with light element impurities. To experimentally simulate the presence of light gases in the alumina matrix due to neutron transmutation processes, the ceramic oxide surface has been implanted by hydrogen and helium ions prior to be tested in PbLi.

2. Experimental

2.1. Samples and sample implantation

Two high purity and high density (better than 99.7 %, 3.95 g/cm³) technical alumina ceramics manufactured by a slip-casting process (SL) and by extrusion (EXT) were tested. While the SL alumina plates were purchased from Nanoker (Oviedo, Spain), the 99.8 % purity EXT alumina substrates were acquired at Ceramic Powders SL (Pontevedra, Spain). Both bulk ceramics were perfectly polycrystalline corresponding to alpha-alumina (ASTM pattern No. 00-005-0712). The PbLi tolerance of both materials was evaluated in $10 \times 5 \times 3$ mm samples at three different conditions: i) as-received, ii) after He ion implantation and iii) after He and H implantation. Samples implanted by helium and hydrogen ions were studied to simulate the generation of light gases in the alumina matrix due to neutron transmutation effects, assessing whether the presence of these species and the ion-induced damage affects the chemical compatibility experiment. Consequently, the PbLi tested specimens listed in Table 1 were named as follows: EXT1, EXT2, EXT3, SL1, SL2 and SL3.

Ion implantations were carried out at the Centre for Microanalysis of Materials (CMAM, Cantoblanco, Spain). To select irradiation conditions, simulations were previously run employing *The Stopping and Range of Ions in Matter (SRIM)* software [11]. The calculated Bragg peaks are shown in Figs. 1 and 2. The so-called **He-implanted** samples (EXT2 and SL2 in Table 1) were irradiated with a 15 MeV He ion beam using a beam degrader, the total dose being 1.1×10^{16} ions/cm². Fig. 1 shows the SRIM simulations yielded values of displacement per atom (dpa) and ion concentration (appm) at five different depths according to the initial

He⁺ beam energy and the thickness of the aluminum beam degrader windows (18, 50, 75 y 100 µm). The use of an energy degrader allows the sample *homogeneous* irradiation in the first 75 µm from surface. In a second irradiation campaign, an H and He ion implantation was carried out to an in-coincidence ion projection of 27 µm inside the ceramic. The aim of the double implantation is to simulate the coexistence of these light gases, likely resulting from the transmutation induced by plasma neutrons of either matrix and impurity elements, and lithium from the liquid metal composition. Without the interception of the beam degrader during this implantation trial, a 7.6 MeV He ion beam followed by a 1.6 MeV H⁺ beam was accelerated towards the alumina targets up to a total dose of 4.23×10^{15} and 1.53×10^{16} , respectively. Resulting samples were labelled as EXT3 and SL3 (Table 1). Similar to the previous implantation, the dpa values and He and H implanted concentrations were plotted versus depth distribution in Fig. 2.

2.2. The COrrOsion Experiment in Static conditions (COES)

Hence, in order to evaluate the influence of the molten PbLi on the ceramic materials properties, long-term chemical compatibility experiments were conducted at the Liquid Metal Laboratory (LML), a group of facilities located at CIEMAT, Spain, aimed at developing new technologies required for liquid metal-based breeding blanket approaches. The exposure of as-received and ion-implanted samples to liquid PbLi was carried out in the COES facility (COrrOsion Experiment in Static conditions), a testing equipment among others located in the LML (CICLO (Ciemat Corrosion LOop) and CLIPPER). Specifically designed to test material compatibility with the PbLi liquid eutectic alloy, COES has conducted over 40 experiments testing different materials since it became operational in 2021. The apparatus shown in Fig. 3 is an AISI 316 stainless steel chamber with a volume capacity of 2 dm³, able to allocate up to 8 small alumina crucibles (41 × 30 mm²). The heating system is a tubular furnace (JH Hornos- HEE-C) with an integrated K thermocouple and a PID controller (CONATEC-REGULATOR 4801). Two Swagelok® valves close the internal gaseous atmosphere and the pressure is monitored by an Edwards® active strain gauge (ASG2, 2.000 mbar⁻¹.) Pressure and temperature are controlled by a National Instruments chassis 4-slot USB (CDAQ-9174, compactdaq).

The PbLi alloy was purchased by CIEMAT to CAMEX GmbH. Several physicochemical analyses of this alloy were carried out at CIEMAT with different techniques to certify the lithium and impurities contents [12]. The eutectic melting temperature is 235 °C with a lithium content of 14.7 ± 1.99 at.%, quite close in composition to the fusion reference Pb-16Li eutectic alloy.

The protocol for testing materials compatibility with PbLi must ensure the minimum presence of oxygen, water and oxides in PbLi. Therefore, the ingot of eutectic PbLi was cut in small pieces with a SiC disc and a cutting liquid free of water using a Secotom-6 saw (Struers S. A) and grinded with a SiC sandpaper to avoid the presence of oxides and other surface impurities. This piece of solid PbLi was placed in the crucible with the sample before locating the crucible inside of the COES chamber. Once the chamber was closed, and before the test, five cycles of vacuum and argon filling were performed, and finally, the chamber was pressurized with 1 bar of 99.999 % purity Ar (Air Liquid). The compatibility experiment was carried out at 550 °C for 1000 h.

2.3. Microstructural and compositional study

After the compatibility test and before the characterization, samples were immersed during 15 min in a cleaning bath of ethanol (98 %): hydrogen peroxide (30 %vol.): acetic acid (glacial) in a 1:1:1 ratio, analytical quality liquids purchased from MERVLAB S.A., after which they were raised with deionized water and dried in air. Tested and cleaned samples were mirror-like polished prior to study the ceramic microstructure near the surface in contact with PbLi. The specimen cross section is encapsulated upwards in a 30 mm in diameter plastic

Table 1
Summary table of damage depths calculated by SEM for the alumina samples with different treatments.

Sample	15 MeV He ⁺ implantation	7.6 MeV He ⁺ & 1.6 MeV H ⁺ implantation	PbLi treatment	EXT Mean grain size	SL Mean grain size
0	–	–	–	21 ± 0.5	1.2 ± 0.2
				Damaged depth (±1 µm)	
1	–	–	–	30	10
2	✓	–	–	51	9
3	–	✓	–	41	10

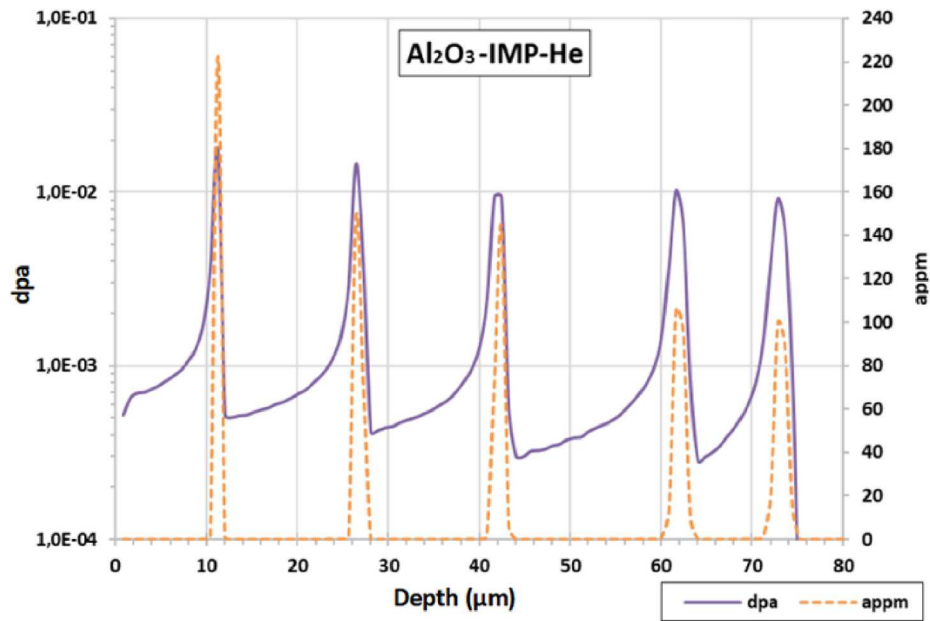


Fig. 1. Dpa and helium ion concentration depth profiles as obtained for the EXT2 and SL2 alumina ceramics using the SRIM code.

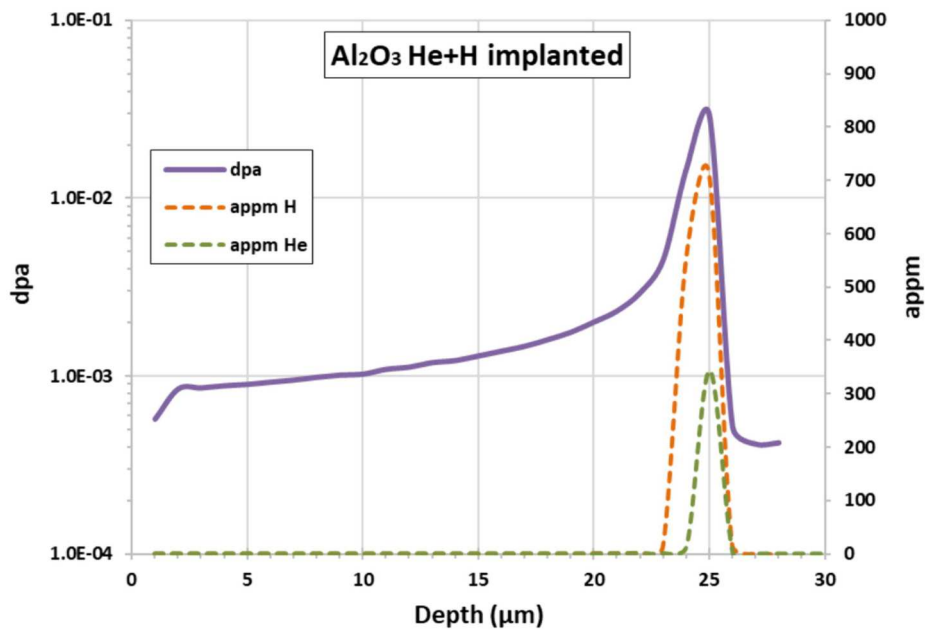


Fig. 2. Dpa, helium and hydrogen ion concentration depth profiles as obtained for the EXT3 and SL3 alumina ceramics using the SRIM code.

transparent disc, to be firstly grinded during 2 min using a 400-grit, water lubricated, SiC wheel, and then polishing on napped cloths with a sequence of normally 5 min-each 15 μm , 6 μm and 1 μm diamond particles suspended in a water-based lubricant. The alumina polished surface was then thermal etched during 15 min at 1500 $^{\circ}\text{C}$ to reveal the grain boundaries.

The FESEM Zeiss Auriga Compact, available at CIEMAT, Madrid, Spain, was the equipment used to study the microstructural features developed at the ceramic surface in contact with the liquid metal, in comparison with the material without implantation or PbLi exposure, named as EXT0 and SL0. The SEM acquired micrographs and calculated degradation depths are shown in Fig. 4 and Table I.

Secondary Ion Mass Spectrometry in the dynamic mode (D-SIMS) is the analytical method applied to determine the in-depth elemental

composition [13]. The HIDDEN SIMS workstation uses an internally generated O_2^+ primary ion beam, working at 5 keV and $3.8 \cdot 10^{-5}$ mbar and optimized to 700 nA of ion current, that strikes the solid sample surface. The generated secondary ions are then accelerated and focused inside a quadrupole mass analyzer. Since the analyzed target matrix are insulators in nature, an electron gun set at 400 μA was used to compensate the surface charging. It is appropriate to mention that the base oxygen signal corresponding to the primary ion gun is only 1×10^4 au and is therefore included under the curves recorded for these ceramic oxides. The in-depth investigated masses were ^7Li , ^{16}O , ^{27}Al and ^{208}Pb . Once the analysis is finished, the SIMS crater depth is measured by means of a profilometer. Considering the total analysis time, the matrix sputtering rate is then calculated for each profile to be properly plotted as a function of depth.

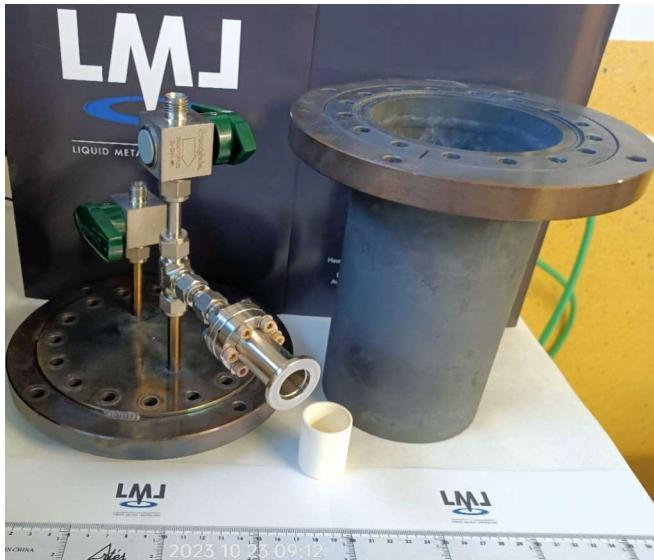


Fig. 3. COES experimental facility at LML-CIEMAT.

3. Results and discussion

3.1. SEM

The SEM micrographs in Fig. 4 show the cross-sectional surfaces of the as-received and implanted samples after the PbLi compatibility test. Micrographs a) and e) correspond to fresh fractures of the as-received samples, the grain size being larger for the EXT0 ($21 \pm 0.5 \mu\text{m}$) compared to the SL0 ($1.2 \pm 0.2 \mu\text{m}$). Fig. 4 also shows the micrographs of ceramic samples after the 1000 h treatment in contact with PbLi. In the upper part of the images and limited between horizontal green dashed lines, the most superficial region has been highlighted, where the microstructure has suffered an evident attack. Actually, the EXT alumina shows two zones with different degrees of damage, separated by a third, white dashed, horizontal line. Below and towards the bottom of the images, the PbLi undamaged material is imaged.

After 1000 h of compatibility test in static PbLi at 550°C , the microstructural evolution of SL and EXT alumina is found different. In the comparative analysis with the two as-received alumina grades (SL1 vs EXT1), the action of the liquid metal seems to have weakened the alumina chemical bonds, the aggressiveness appearing to be more effective in the sample characterized by a higher grain size. Furthermore, the region in contact with the liquid metal in the beforehand ion-implanted samples (SL2, SL3, EXT2 and EXT3) has also suffered from an evident damage (Fig. 4-c-to-h). In the SL alumina affected by He implantation, the attack progresses to a depth similar to that measured for SL1 (Table I), the affected region being slightly deeper in the doubly implanted one. The SL alumina degradation reaches a thickness below or almost equal to the calculated He and H Bragg peaks (Figs. 1 and 2).

However, the chemical effect in the microstructure is more pronounced in the EXT samples. The SEM damage has proceeded up to $30 \pm 1 \mu\text{m}$ in depth from the surface in the as-received EXT alumina, the influenced region increasing to 51 and $41 \pm 1 \mu\text{m}$ in the ion-implanted EXT2 and EXT3 samples, respectively. The reaction rate is then higher when the surfaces have been subjected to the prior penetration of high-energy ion beams. Furthermore, cracking formation is observed in these EXT samples at a depth of about $15 \mu\text{m}$ from the surface, separating two damaged layers of different textures (Fig. 4 c and d). In close coincidence with the He and H ion projected range predicted by SRIM at about $25 \mu\text{m}$, the embrittlement revealed at this depth is a consequence of the damage induced along the high energy ions projected range. In fact, the loose grains observed in the superficial area of the EXT3 SEM preparation suggest that the weaker grains are either eroded by the slow liquid

metal movement or washed out during sample cleaning.

Based on the SEM images, the observed effect is compatible with the so-called intergranular corrosion, also known as intergranular attack, widely described in alloys [14,15]. The resistance of these alumina samples to the PbLi is hampered by the more susceptibility of grain boundaries to the chemical reaction than the material itself. The resistance of alumina ceramics is known to be influenced by its purity. Since impurities are segregated to the grain boundaries during ceramic sintering, the presence of metal oxides (SiO_2 , CaO , Fe_2O_3 , MgO , Na_2O , ...) give rise to the formation of an easily dissolved silicate-rich glassy phase. While the grain boundaries in the EXT alumina appear to have been dissolved, in the SL grade the strong gap between grains strongly supports the mechanism of the liquid metal interaction occurring in several sequential stages. Starting from the lithium diffusion into the alumina ceramic through the grain boundaries, chemical reactions may take place, likely transforming the initial crystal lattice, modifying the phase volume and therefore causing the accumulation of tension forces that debilitates the intergranular cohesion.

The behaviour in PbLi could be explained as a consequence of the lower mechanical strength of alumina implanted with light elements. Studies on the mechanical resistance of SL alumina [16] point out that the strength resulting from compressive loading significantly decreases in the proton-implanted sample in comparison with the alumina in the as-received condition, the origin being found linked to irradiation-induced changes in the grain size. Besides and relying on previously published work, the formation of bubbles, constitutional vacancies as well as irradiation-induced defects in alumina is known to occur depending on He implantation dose and temperature [17,18]. Likewise, Sasajima et al [19] carried out a study with sequential He and H implantation, where bubbles, point defects and vacancies were also observed but dislocation loops were found larger under hydrogen irradiation. Considering the simultaneous He and H irradiation, Zinkle [20] reported the formation of cavities and nucleation of dislocation loops at temperatures up to 650°C covering the entire irradiation zone, while these defects are mainly located in the peak implantation in other ceramics. Therefore, it can be concluded from literature that light ion irradiation is associated with microstructural damage promoting defects, interstitial vacancies, ... among other phenomena, that can facilitate lithium diffusion path and lead to matrix degradation.

The ceramic channel under consideration for the advanced DCLL BB approach is being designed with a maximum estimated thickness of 25 cm [6]. Considering the depths of the damaged layers (summarized in Table I), the degradation rate of the commercial EXT alumina is estimated to be $0.71 \pm 0.02 \mu\text{m/day}$ for the as-received sample, the light ion implantation increasing this value to 1.20 ± 0.02 (EXT2) and $0.98 \pm 0.02 \mu\text{m/day}$ (EXT3). The previous values calculated from SEM micrographs could enlarge by an additional error of about 15% , considering the possible loss during washing of detached crystallites as discussed above. On the other hand, the SL alumina shows a degradation factor of $0.24 \pm 0.02 \mu\text{m/day}$ without a significant influence on the damaged layer thickness due to the implantation treatment. Since the damaged depth due to PbLi could be then estimated at $88 \mu\text{m/year}$, the SL alumina could be considered a promising candidate for a potential ceramic channel.

3.2. SIMS chemical determination

The observed changes in the microstructure seem to be reflected in the SIMS compositional analysis. In order to register the depth evolution of the chemical elements involved in the PbLi compatibility test, the intensity of ^7Li , ^{16}O , ^{27}Al and ^{208}Pb mass signals as a function of time was acquired by means of the dynamic SIMS technique. Due to the longer times involved in SIMS analysis, the in-depth qualitative determinations were carried out down to the first $8 \mu\text{m}$ below the surface in contact with the PbLi. It is worth underlying that the SRIM simulated, maximum structural damage and light ion projected ranges (Fig. 2)

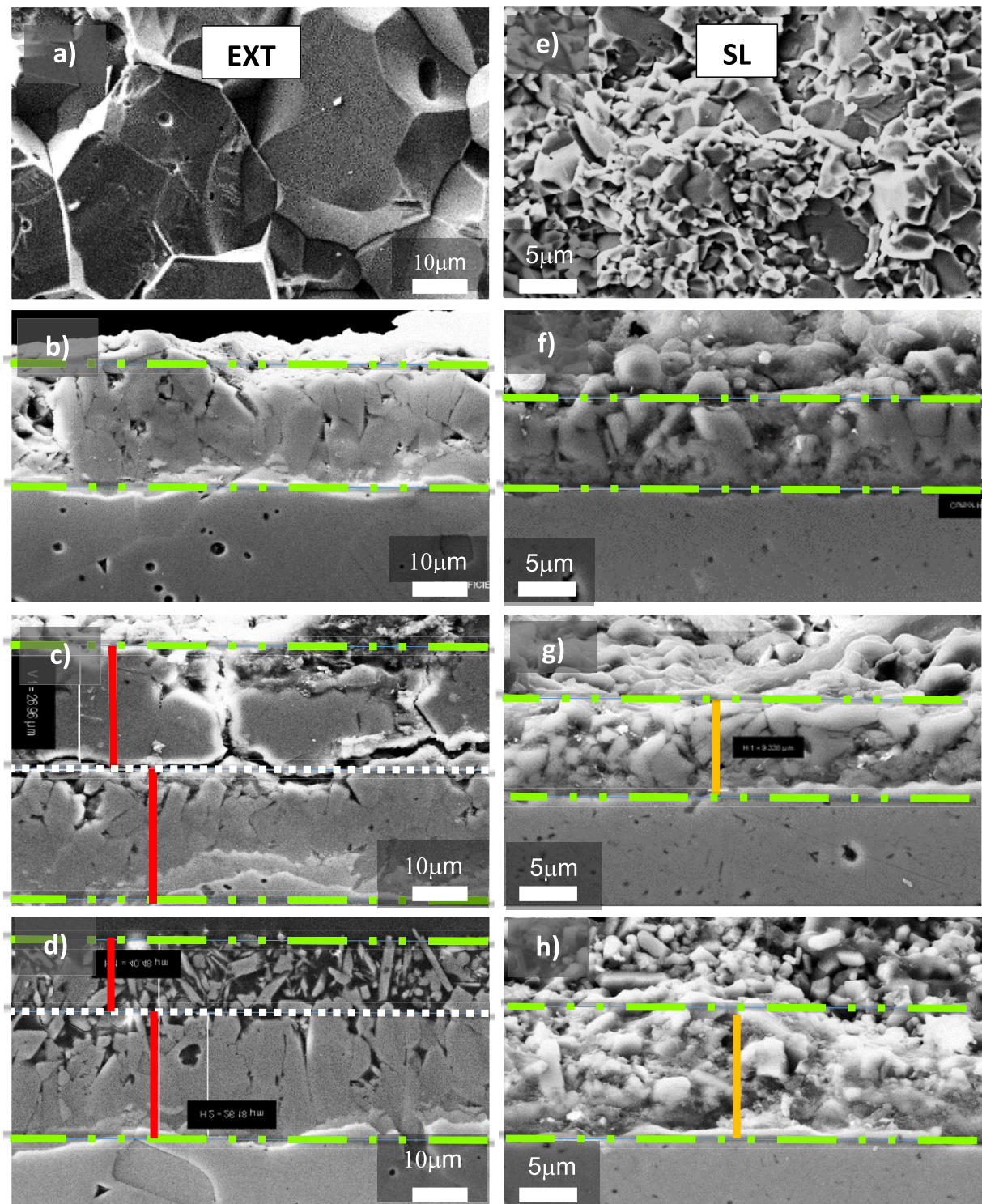


Fig. 4. Series of images displaying the fresh fracture microstructure of the EXT and SL alumina grades in the as-received state (a and e), and as polished cross-sections after the PbLi compatibility test: in the non-irradiated (b and f), the He-irradiated (c and g) and the He + H-irradiated (d and h) condition.

should be found at several microns beyond the SIMS analyzed region. The less disaggregated SL alumina samples were then chemically analyzed.

The normalized elemental trends shown in Fig. 5 as a function of matrix depth demonstrate the surface deterioration of the SL alumina subjected to the long PbLi exposition. In this comparative study, as the SIMS analysis is performed from the as-received (SL0) to the non-implanted (SL1), single (SL2) or double (SL3) implanted samples

exposed to PbLi, the evolution of the registered elements as a function of depth become more significant. While the intensity of Al signals decreases with time and ion implantation, the Li signal is made evident in all PbLi-affected samples (Fig. 5-a and c, respectively). Of the two major components of the ceramic, O seems to be less influenced by the liquid metal and the damage caused by irradiation (Fig. 5-b). In agreement with the cross-sectional micrographs where the liquid metal appears solidified at surface without diffusing into the matrix, Pb is not detected

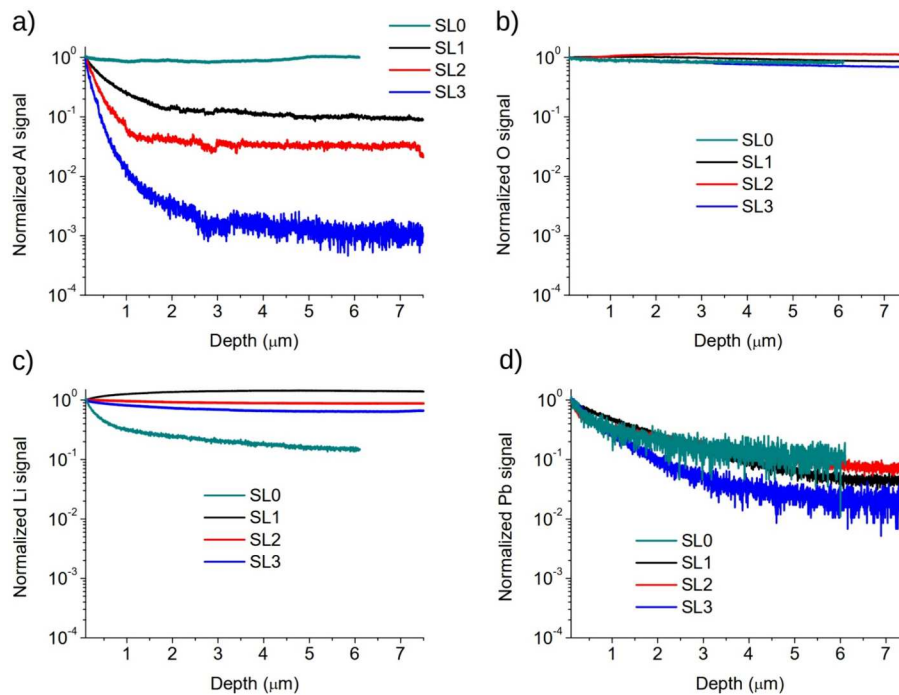


Fig. 5. SIMS depth profiles of a) Al, b) Li, c) O and d) Pb elemental signals for the as received (SL1), He-implanted (SL2), and He + H-implanted (SL3) alumina samples. The in-depth elemental evolution curves registered for the SL alumina without exposition to PbLi (SL0) are included for comparison.

at depth, as shown by the similar trend in the evolution curves (Fig. 5-d). The slow signal decrease with depth showed is characteristic of this element.

At least several insights from the compatibility of alumina with the PbLi liquid breeder can be concluded from the analytical results. On the one hand, a clear diffusion of lithium is determined as the loss of the matrix Al also diminishes. In coincidence with the depths microstructurally affected by the liquid metal, an effective change in the matrix stoichiometry is therefore revealed. On the other hand, the deviations undergone by the Al SIMS signal become more relevant as the matrix is ion-implanted, being highly marked in the double-implanted material. Two scenarios could therefore be considered to explain the action of PbLi in ion-implanted alumina: i) Li diffuses shallower than the light ions projected range, or ii) lithium penetrates the He and H-influenced depth, although no additional effect in the mechanism of degradation can be stated.

The results of the SEM microstructural observations and SIMS elemental determinations point out the relative attack of the liquid metal components. Indeed, the Ellingham diagrams and Gibbs energies [21] suggest the chemical reaction of alumina with lithium at 550 °C. According to the revised literature [22–25], the interaction of alumina with PbLi alloy is indeed driven mainly by an intergranular diffusion of the alkaline element in the matrix, causing the reduction of the matrix cation and giving rise to the formation of lithium aluminate (LiAlO_2) and non-stoichiometric Li-Al alloys. As a consequence, the attack may involve dimensional and compositional grain changes, due to lithium diffusion, chemical reaction, crystal lattice deviations and likely surface erosion. The weak cohesion of the transformed grains in this damaged layer could then be evidenced as the large cracks in the microstructure (Fig. 4), enabling a certain degree of mass loss during the ceramic immersion in PbLi and the washing process prior to characterization.

4. Conclusions

SEM imaging and SIMS compositional analyzing techniques have been applied to study the compatibility of two commercial alumina ceramics against the action of PbLi eutectic alloy. Alumina is a

promising candidate material as electrical insulating BB channel component of the advanced DCLL fusion concept. For the first time, the chemical resistance to the liquid metal has been tested on the H-isotopes and He implanted insulator, simulating the presence of the light elements generated by the neutron transmutation of matrix elements and their impurities.

Alumina samples were subjected to 1000 h in static PbLi at 550 °C before being analyzed. The chemical compatibility test concludes that alumina ceramic is attacked on surface. The SEM microstructure appears to be degraded, showing dissolution of the component at grain boundaries and detachment of alumina grains, together with partial loss of material. The damage progresses deeper in the largest grain size alumina, the best behaviour having found in the commercial alumina produced by slip casting (SL), which deteriorates by only 10 μm from the surface. The SIMS analytical determination concludes that lithium diffuses in the alumina matrix. Furthermore, the loss of aluminum is registered as the alumina is transformed by the diffusion and chemical reaction of lithium.

A more thorough study considered the alumina chemical resistance with the presence of light elements in the ceramic surface. When cast alumina is ion-implanted with 15 MeV He^+ up to 1.1×10^{16} ions/cm² total dose, or with 7.6 MeV He^+ followed by 1.6 MeV H^+ up to approx 2×10^{16} ions/cm² total dose, the surface degradation rate is maintained, the PbLi reaction progressing down to similar depths as in the as-received condition. In operation, this alumina would lose approximately 87 μm per year, a negligible thickness in a DCLL ceramic channel wall so far designed with thicknesses ranging from 25 to 12 cm [6]. Having verified the effective diffusion of lithium in alumina, the presence of tritium by neutron transmutation of lithium is plausible. As a result, the issue of tritium inventory becomes important for alumina components in the fusion reactor.

CRediT authorship contribution statement

J. Patiño: Writing – original draft, Methodology, Investigation, Formal analysis, Conceptualization. **J.M. García:** Writing – review & editing, Methodology, Investigation, Formal analysis, Data curation. **G.**

D. Soria: Writing – review & editing, Methodology, Investigation, Formal analysis, Conceptualization. **M. González:** Writing – review & editing, Validation, Supervision, Resources, Project administration, Investigation, Funding acquisition, Formal analysis, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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