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## Development, characterization and testing of a SiC-based material for Flow Channel Inserts in high temperature DCLL blankets

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Abstract—Flow Channel Inserts (FCIs) are key elements in the high temperature DCLL blanket, since in this concept the flowing PbLi reaches temperatures near 700 °C and FCIs should provide the necessary thermal and electrical insulations to assure a safe blanket performance. In this work, the use of a SiCsandwich material for FCIs is studied, consisting of a porous SiC core covered by a dense CVD-SiC layer. A fabrication procedure for porous SiC is proposed, being the resulting materials characterized in terms of thermal and electrical conductivities (the latter before and after being subjected to ionizing irradiation) and flexural strength. SiC materials with a wide range of porosities are produced; additionally, preliminary results using an alternative route based on the gel-casting technique are also presented, including the fabrication of hollow samples to be part of future lab-scale FCI prototypes. Finally, to study the corrosion resistance of the material in hot PbLi, corrosion tests under static PbLi at 700 °C and also under flowing PbLi at ~10 cm/s and 550 °C, with and without a 1.8-2T magnetic field, were performed to materials coated with a 200-400 µm thick dense SiC layer, obtaining promising results.

*Keywords*— Flow Channel Insert; DCLL blanket; porous SiC; electrical conductivity; thermal conductivity; corrosion by PbLi

### I. INTRODUCTION

The Dual Coolant Lead Lithium (DCLL) breeding blanket is being investigated as a candidate for the DEMO reactor, where a major requirement will be the tritium self-sufficiency to sustain the thermonuclear reaction with the required efficiency in converting the energy coming out from the burning plasma to provide several hundreds of MW [1]. The DCLL principle is based on the use of a eutectic PbLi alloy in liquid state acting as breeder and coolant, flowing through long channels while heating up to high temperatures. A low temperature version of the DCLL is being currently designed by CIEMAT [2], where the PbLi stays lower than ~550 °C; the potentiality to elevate the PbLi operating temperature up to  $\sim$  700 °C with the consequent increase of efficiency in the power conversion system makes the DCLL an interesting concept for the future [1], although high temperature materials must be developed and tested to face this concept.

In the DCLL blanket, the liquid PbLi will be in direct contact with the blanket structure, which is expected to be made of a Reduced Activation Ferritic Martensitic (RAFM) steel like EUROFER; to assure the corrosion stability against hot PbLi of all materials in contact with it is thus crucial to ensure a reliable operation. The corrosion behavior against PbLi of RAFM steels is strongly dependent on the temperature and the flow characteristics of the PbLi [3][4]. Although the experimental data regarding the corrosion rate of RAFM in flowing PbLi differ in a wide range, corrosion issues are supposed to appear at temperatures near 450-550 °C; besides, the presence of a magnetic field may affect severely the corrosion rate [4]. Therefore, to assure that the steel is maintained at a temperature at which severe corrosion issues are avoided, in a high temperature DCLL the PbLi should present a sufficient thermal insulation with respect to the blanket structure. In addition, to reduce magnetohydrodinamic (MHD) effects that may affect the flow velocity and the heat transfer, the PbLi flow should be also electrically decoupled from the conducting steel. Studies and experimental tests under relevant conditions must be performed to assure the correct performance of all components.

The blanket components responsible of the insulation tasks are a key element in the DCLL design, especially in the high temperature concept, and are called flow channel inserts (FCIs). FCIs are typically designed as hollow square channels of a few mm thick, containing the hot flowing PbLi and being separated from the RAFM steel wall by a thin gap also filled with PbLi. In the framework of the low temperature DCLL design, FCI prototypes made of a steel-alumina-steel sandwich material are being tested [2]. In a high temperature approach, however, extra challenges must be overcome regarding the FCI material, especially concerning the corrosion phenomena and the thermal stresses that will be present in the channels due to the considerably high thermal gradient across its walls. For these reasons, silicon carbide (SiC) is considered as a candidate for FCIs in a more advanced high temperature DCLL approach, due to its good behavior in high radiation environments, its chemical stability under PbLi and its high mechanical strength; however, the relatively high thermal and electrical conductivities of this ceramic requires the development of a low-conductivity SiC material. The insulation requirements of FCIs depend on their design parameters, such as its projected thickness and shape, as well as on other aspects like the PbLi flow velocity profile; all these dependencies are being studied and discussed in several works. In the analysis of the technical challenges on the pathway to DEMO made by Abdou et al. [5], the requirement for effective flow channel insert performance in terms of thermal conductivity is considered in the range 1-5 W/m·K and in the electrical conductivity of 1-10 S/m; in the review of the status of the DCLL made by Smolentsev et al. [6], the most restrictive goal regarding to the FCIs of the inboard blanket is to achieve a thermal conductivity of 1-2 W/m·K and an electrical conductivity of about 1 S/m. Besides, recent studies like the one presented by Chen et al. [7] show the dependence between the FCI design and the MHD effects and the heat transfer profile, being specially influent the thickness

of FCIs; in this work, the authors suggest a 5 mm FCI, assigning them a 8 W/m·K thermal conductivity and a considerably low electrical conductivity of 0.0001 S/m. The results pointed out by the different studies are consistent with a previous study presented by the authors of this paper [8], where a 5 mm FCI with a thermal conductivity near 7 W/m·K is suggested as a possible option. Essentially, it can be concluded that R&D efforts must be done to develop a low-conductivity, high resistant SiC material with a wide enough design window to be consistent with the blanket requirements, characterizing the candidate materials under relevant conditions, and considering the manufacture possibilities.

Together with the development of the SiC/SiC composites [9], other potential candidates for FCIs are SiC-sandwich materials, based in a core of porous SiC covered by a dense SiC coating typically produced by the Chemical Vapor Deposition (CVD) method. Following this concept, foambased SiC FCI prototypes have been produced by Ultramet, tested in UCLA using the MaPLE facility and analyzed at CIEMAT [10][11][12]; in this material, a highly porous SiC foam infiltrated with silica aerogel is covered by a CVD-SiC coating of about 1 mm thick.

In the present work, a different SiC-sandwich material is presented, based in an alternative production method of porous SiC. Following this method, materials with a wide range of porosities and hence of properties can be produced; the fabrication process, as well as the properties of the material obtained, is shown in section II. Porous SiC samples produced by this method have been also coated with a dense CVD-SiC layer with thicknesses between  $\sim$ 200-400 µm; such a thickness has been chosen to study if it is enough to provide protection against PbLi corrosion while reducing the high mechanical stresses that seem to concentrate in this dense layer, according to the results presented in our previous work [8]. The coating process was performed by Archer Technicoat Ltd, UK. To determine the insulating properties of the material, its thermal and electrical conductivities have been studied; in the latter case, measures after submitting the samples to ionizing irradiation have been also performed, using a 1.8 MeV Van de Graaff accelerator at CIEMAT. These results are presented in section II. Furthermore, to study the viability of a method that allowing the fabrication of hollow samples of required size and shape for FCIs, an adaptation of the procedure to the gelcasting technique is being studied; preliminary results obtained by this method are presented in section III.

To characterize the material's behavior against hot PbLi corrosion two different experiments have been conducted. Firstly, porous samples coated with a ~200  $\mu$ m CVD-SiC layer were tested under static PbLi at 700 °C for 1000 h [13]. A second corrosion experiment has been recently performed, testing a new batch of coated samples under flowing PbLi at ~10 cm/s and 550 °C for 850 h; in order to study the possible influence of the presence of a magnetic field in the corrosion phenomena, some samples were subjected to a 1.8-2 T magnetic field during the experiment. The results of these corrosion tests are presented in section IV. The mentioned experiments have been performed using lab-scale flat samples; in the future, further tests will be performed with hollow labsize FCI prototypes.

### **II. SIC PRODUCTION**

#### A. Experimental procedure

As mentioned in the previous section, the proposed production method of porous SiC is based in the *sacrificial template* technique, previously used in the fabrication of porous ceramics. In this procedure, the material is obtained from a powder mixture with a sacrificial phase added to it, being this phase later removed leaving pores in the structure.

In this work, SiC powder (Superior Graphite, 0.3 µm) together with 1.5 wt.% Al<sub>2</sub>O<sub>3</sub> (0.4  $\mu$ m) and 1 wt.% Y<sub>2</sub>O<sub>3</sub> (1 µm) were used as initial powders, the last two acting as sintering additives. Spherical mesocarbon microbeads (MCMB, 15 µm) were also added to the mixture as the sacrificial phase, in quantities ranging from 0 to 22 wt.%. The sintering additives together with the SiC powder and a 3 wt.% of an aqueous polymer as the binder agent were mixed in ethanol for 18h, being the MCMB added afterwards to the solution and mixed for another 30 min. With the resulting blend, samples with the required geometries were produced by uniaxial pressing at 100 MPa. The green compacts were then sintered at 1900 °C for 30 min, and to burn out the carbonaceous sacrificial phase, the sintered samples were subjected to an oxidation treatment, heating them up to 700 °C for 10h.

The porosity of the resulting samples was calculated from the comparison between its measured and theoretical densities: the density of the samples was determined from its geometry and weight, and the theoretical one was calculated by the rule of mixture. Microstructure was studied by field emission gun scanning electron microscopy (FESEM). Thermal conductivity as a function of temperature was determined from the specific heat capacity obtained from [14], density and thermal diffusivity of the samples, measured by the Laser Flash method. Regarding the electrical properties, electrical conductivity measurements were performed in the Van de Graaff accelerator of CIEMAT, subjecting the samples to an ionizing irradiation of 1.8 MeV up to 140 MGy to study also the behavior of the material after irradiation. Flexural strength was determined at room temperature by three point bending tests (3PBT) using 4 samples for each condition.

### B. Results and characterization

In Fig.1, the final porosity of the samples as a function of its initial amount of MCMB (sacrificial phase) is shown for 5 different compositions (15, 18, 20 and 22% of initial MCMB, along with the material fabricated with 0% initial MCMB for reference). As can be observed, the final porosity increases with increasing the sacrificial phase content, being the sintering process less effective in the samples with high MCMB; in this materials the porosity increase in a more severe way, showing rather an exponential relationship with the amount of sacrificial phase.



Fig. 1. Porosity of the final material as a function of its initial amount of sacrificial phase (error bars are showed only for deviations  $\geq 1\%$ )

The microstructure of materials produced with porosities of 35, 40 and 55% are shown in fig. 2 a). The spherical pores are formed by the burn out of the sacrificial phase; the reduction of the sintering grade while increasing MCMB content can be seen. This effect causes residual porosity in the SiC matrix, so it is detrimental to the mechanical strength; to improve sintering in the high porous samples would be desirable if a material as resistant as possible is desired. In fig 2. b), details of the microstructure of the pores and of the surrounding SiC matrix can be seen in a sample produced with 18% initial MCMB, resulting in a final porosity near 40%.

The insulating properties at high temperatures of the produced porous SiC materials have been characterized. In fig. 3, the thermal conductivity as a function of temperature of samples with different porosities (6, 36 and 43%) can be seen. As expected, the thermal conductivity decreases with temperature and with porosity. As the projected operation temperature of FCIs at the high temperature DCLL is near 700 °C, the thermal conductivity at this temperature of SiC samples with different porosities are shown in fig.4. The thermal conductivity of the material decreases exponentially with porosity, being the most interesting materials for FCIs in terms of thermal insulation those with porosities up from ~40%, as they correspond to the required thermal conductivity of <10 W/m-K at 700 °C.

Concerning the electrical properties, the electrical conductivity versus the inverse of temperature of three different SiC materials, before and after being submitted to an irradiation of 140 MGy, can be seen in fig. 5. Two porous samples with porosities of 34 and 50% were measured; also, the electrical



Fig. 2. a) Low magnification FESEM micrographs of samples produced with: 1) 15% initial MCMB (final porosity 35%); 2) 18% initial MCMB (final porosity 40%); 3) 22% initial MCMB (final porosity 52%); b) Microstructure of the pores in a 40% porous sample



Fig. 3. Thermal conductivity as a function of temperature of samples with different porosities (6, 36 and 43%)



Fig. 4. Thermal conductivity at 700  $\,^{0}\mathrm{C}$  as a function of porosity of SiC materials with different porosities

conductivity of a sample coated with a dense CVD-SiC layer, whose microstructure can be seen at fig. 6, is presented. This sandwich material was formed covering a 34% porous sample with a dense coating of ~230-300  $\mu$ m thick, being the thickness of the porous core similar to those of the two porous samples measured. The values of the electrical conductivity of each sample at the highest temperature tested, 550 °C, are shown in table I.



Fig 5. Electrical conductivity versus the inverse of temperature of three different samples, before and after a 1.8 MeV irradiation up to 140 MGy. Two porous samples (with 34 and 50% porosity) and a coated SiC-sandwich sample (formed by a porous core of 34% porosity covered by a dense CVD-SiC layer of 230-300  $\mu$ m thick) are shown

 TABLE I

 ELECTRICAL CONDUCTIVITIES AT 550 °C BEFORE AND AFTER IRRADIATION

		$\sigma$ at 550 °C (S/m)
	Before irradiation	
50% porous sample		2.8.10-5
	After irradiation	1.2.10-4
34% porous sample	Before irradiation	0.11
	After irradiation	0.23
	Before irradiation	0.92

As can be seen, porosity has a great influence in the electrical conductivity, being increased by several orders of magnitude in the 34% porous sample compared to the 50% one. The electrical conductivity of the coated material is also higher than those of the completely porous one, although the width of the porous layers of both materials were very similar. This is an unexpected result, which may indicate that changes occur in the porous SiC during the CVD treatment inducing an increase in its electrical conductivity. However, this effect should be confirmed with further measurements. Regarding the influence of the ionizing radiation, an increase in the electrical conductivity after irradiation is observed in all samples; still, the greatest value measured is near 1 S/m, which would fit the electrical insulation requirements.

Finally, to end with the characterization of the material, its flexural strength in function of the porosity can be seen in Fig. 7. As expected and introduced before, the strength decreases

considerably as the porosity increases, even though the important dispersions of the measured values prevent to draw



Fig. 6. FESEM micrographs of the coated sample whose electrical conductivity is showed in fig.5 and table I. The dense coating covered the top and bottom of the sample (the sides remain uncoated to measure the conductivity through the thickness, avoiding short-circuits) having a thickness between  $\sim$ 230-300 µm

clear conclusions. Obtaining different values of flexural strength in samples with the same composition is attributed to the presence of defects like cracks; the uniaxial pressing step contributes considerably to its appearance. The highest dispersion was obtained in the samples pressed with any sacrificial phase, which are the ones presenting the most difficulties in the compaction step due to the absence of the lubricant effect provided by the presence of graphite. The results of a previous work [8] which studied the distribution of the stresses caused by the thermal gradient across the FCI showed that, in a SiC-sandwich FCI like the one proposed, the highest stresses would be present in the dense coating, being the stresses supported by the porous core an order of magnitude lower. According to this study, the flexural strength of the porous materials tested would be enough to support the stresses present in the core of the FCI; nevertheless, further work should be done in order to assure the production of a material free of defects, being also desirable to increase the strength of the highly porous materials to assure a safe operation.



Fig. 7. Flexural strength versus porosity

### III. PRODUCTION OF POROUS SIC BY THE GELCASTING TECHNIQUE: PRELIMINARY RESULTS

The consolidation techniques commonly used in powder metallurgy imply limitations, like the relatively reduced size of the samples that can be manufactured; also, techniques like uniaxial pressing can easily lead to the presence of defects like cracks in the samples, being this especially true in the manufacturing of hard, brittle ceramics like SiC. These problems added to its hard machinability once sintered and the impossibility of manufacture it in green state, due to the poor density of the green samples produced by pressing, has led to an increased interest in the developing of new methods for producing high quality green ceramics, avoiding the pressing step. In the case of the production of FCIs, where the fabrication of complex shapes with a relatively big size will be possibly needed, these alternative methods are especially interesting, as they could allow the manufacture of samples with no limitations regarding its size or geometry.

In this aspect, one of the promising methods to produce high-quality ceramic bodies is gelcasting, which is based in the dispersion of the initial ceramic powders in an aqueous monomer solution to form a slurry that is subsequently gelled inside a mold. After unmolding and eliminating the aqueous medium by drying the sample, the result is a material with homogenous chemistry and density, containing a certain amount of polymer similar to the amount of binder that is commonly used in pressed ceramics [15]. The principal advantages of the method are its reduced cost, the high strength of the produced green samples and its capability of industrial implementation, being possible to use it for almost all sizes allowing complex geometries. However, key aspects of the process must be well optimized to take full advantage of the method, like the use of high solid content suspensions with an adequately low viscosity, to adequately eliminate the trapped air of the suspension before gelation, or to successfully control the dying step avoiding internal stresses in the part; this aspects, among others, are well summarized in [16].

To produce simple hollow square FCI prototypes (firstly in lab-scale, with the possibility of increase its size or vary its shape in the future) an adaptation of the procedure presented in section II, incorporating the gelcasting method to porous SiC production, is being currently studied. The first hollow samples, together with flat samples of different geometries, have been produced, by using acrylamide (AM) as the main monomer and N,N',-methylenebisacrylamide (MBAM) as the cross linker.

A powder mixture containing SiC powder, a 20 wt.% of MCMB and a 2.5 wt.% of sintering additives  $(Al_2O_3 \text{ and } Y_2O_3 \text{ in a } 3:2 \text{ ratio})$  was added to a premix of the monomers in water (the quantities used were a 10 wt.% of AM and a 5 wt.% of MBAM, with respect to the amount of water). To trigger the gelation reaction, N,N,N,N-tetramethylethylenediamine (TEMED) was used as a catalyst, together with a 5 wt.% aqueous solution of ammonium peroxydisulfate (APS) as initiator. The slurries were subjected to vacuum during a few seconds to remove the air trapped in the suspension, and then casted; when the gelation was completed, samples were unmolded and dried at room temperature. The next steps of the

process, i. e. sintering and oxidation, were performed in the same way than in the traditional route previously presented.

TABLE II PROPERTIES OF THE MATERIAL PRODUCED BY GELCASTING

Initial MCMB content (wt.%)	Final porosity (%)	Flexural strength (MPa)
20	44	<b>79</b> ± 11

The porosity and the flexural strength of the final material (using in the gelcasting mixture a 42 vol.% solid content) are presented in Table II, while its microstructure can be seen in fig. 8. The thermal conductivity of the material, which depends mainly on the amount of porosity, can be deduced by the relationship of fig. 4 ( $\sim$ 8 W/m·K). In fig. 9, hollow square samples produced to become the core of future SiC-sandwich lab-size FCI prototypes are shown. The properties obtained in this gelcasted material are quite similar to those of the uniaxially pressed one if the same amount of initial MCMB is used (42% porosity with a strength of  $84 \pm 31$ MPa in the pressed samples vs the 44% and  $79 \pm 11$  MPa obtained in the gelcasted ones). No cracks were detected in the gelcasted samples; however, extra pores of a greater size than desired (~100 µm) were observed, caused by the trapped air not adequately removed in the degassing step. The additional porosity measured in this samples is attributed to the presence of this undesired pores, whose relatively big size deteriorate the flexural strength; the deairing process will be optimized in the future to avoid this effect. Other important elements for the optimum performance of the gelcasting method, like is the use of a dispersant in the adequate amount to permit high solid contents while maintaining low viscosity, is also still not incorporated to the process used in this work, so it is believed that exists a large margin in the improvement of the final properties of the material.

These first results show that the gelcasting technique is a promising method for the fabrication of ceramic FCIs, being possible to be applied in the fabrication of more complex shapes by the design of proper molds; further results, together with the progresses achieved, will be presented in future publications.



Fig. 8. FESEM micrographs of the material produced by the geleasting method (final material; after sintering and oxidation)



Fig. 9. Hollow porous SiC channels produced by the gelcasting method (final material; after sintering and oxidation) IV. CORROSION EXPERIMENTS

A crucial issue in the DCLL and FCIs design, especially regarding the high temperature concept, is to test the materials that will be directly in contact with the hot flowing PbLi to assure that no corrosion phenomena would be altering the performance of the components. To characterize the behavior of the produced SiC, corrosion experiments under relevant conditions have been performed with lab-scale flat samples, consisting in a porous SiC core (fabricated by the traditional route including uniaxial pressing) covered by a CVD-SiC dense coating. New experiments with lab-size hollow FCI prototypes will be performed in the future.

### A. Test under static PbLi

For the first corrosion experiment, 14x14x5 mm<sup>3</sup> porous SiC samples with a porosity around 50% were covered with a dense CVD-SiC coating of ~200 µm thick. Six samples were tested, being partially immersed in static PbLi at 700 °C for 1000h; photographs of some of the samples at the finish of the test are shown in fig. 10.

After the experiment, samples were cut and its inside was studied by SEM, EDS and X-ray analysis. No lead was detected inside them and no significant reduction of the thickness of the dense CVD-SiC coating was found (as can be observed in the micrographs showed in fig. 10) although some damages in the dense coating occur after the experiment. Details of these results can also be found in [13].



Fig. 10. Samples after the corrosion experiment under static PbLi; a) in the experiment assembly; b) Surface of two samples, with PbLi adhered to the surface; c) Cross-sections of cut samples, where the inner porous SiC can be seen



Fig. 11. Cross-section FESEM micrographs of the inside of samples after the corrosion experiment under static PbLi

### B. Test under flowing PbLi

For this second experiment, a new batch of porous SiC samples with porosities near 40% were coated with a CVD-SiC layer. In this case, the thickness of the coating varied from ~200 to 400  $\mu$ m, due to inaccuracies that take place in the CVD procedure; these problems should be solved to assure the greatest accuracy as possible in the thickness of the coating in future processes. In this experiment, 11 samples were tested; 8 of them were subjected to a 1.8-2T magnetic field throughout the test, while 3 remained outside the magnetic influence as control samples. The PbLi was flowing at ~10 cm/s and 550 °C, being the duration of the experiment 850 h. Photographs of the samples after the test can be seen in fig. 12.



Fig. 12. Samples after the corrosion test under flowing PbLi; a) in the experiment assembly; b) Samples outside the magnetic field; c) Samples subjected to the magnetic field. The ones marked with \* presented damages in the surface

Surface damages were detected in two samples (marked with \* in fig. 12), consisting in a partial detachment of the dense layer that occur during the dismantling of the experiment. The surface was examined by FEGSEM and EDS analysis were performed, confirming that no Pb was present in the zones without dense coating. The reason of these detachments is unknown; severe temperature changes could have affected the integrity of some dense coatings during their extraction of the test assembly, or other followed procedures could have caused damages in the samples. More studies will be done to identify the problem and assure that these conditions would not be repeated in service.

During the test, no failure happened in any case, and no weight gain was presented in any of the samples. Micrographs of the inside of a sample that remained outside the magnetic field can be seen in fig. 13; no cracks or failures were detected in the dense coating, which provided protection against PbLi ingress. Likewise, micrographs of a sample subjected to the magnetic field are shown in fig 14. As can be observed, no substantial differences were found between the Hartmann and side walls, remaining the whole dense coating undamaged also in this series of samples.

### V. CONCLUSIONS

Some brief conclusions can be drawn from the presented results:

- Porous SiC with a variety of properties can be produced by the procedure followed in this work, being possible to adjust the porosity according to the required needs. A SiC material with reduced thermal and electrical conductivities can be fabricated if enough porosity is added.
- A fully dense, free of defects CVD-SiC coating offers a reliable protection against hot PbLi corrosion. No remarkable effects of the application of a magnetic field in the corrosion behavior have been observed.
- The gelcasting method is a promising route in the fabrication of ceramic FCIs.



Fig. 13.Cross-section FESEM micrographs of the inside of a sample after the corrosion test under flowing PbLi; sample located outside the magnetic field, with a  $\sim$ 250 µm dense coating



Fig 14. Cross-section FEGSEM micrographs of the inside of a sample after the corrosion test under flowing PbLi; sample subjected to a magnetic field of 1.8T throughout it, being a) the Hartmann walls, and b) the side walls. Dense coating of ~400  $\mu m$  thick

### VI. ACKNOWLEDGMENTS

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