## 5.2 Fabrication and Characterization of Lithium Orthosilicate Pebbles Using LiOH as a New Raw Material

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## Abstract

For the European Helium Cooled Pebble Bed (HCPB) blanket slightly overstoichiometric lithium orthosilicate pebbles ($Li_4SiO_4$+$SiO_2$) have been chosen as one optional breeder material. This material is developed in collaboration between Research Centre Karlsruhe (FZK) and the Schott Glas, Mainz. The lithium orthosilicate (OSi) pebbles are fabricated by the melting and spraying method in a semi-industrial scale facility. In the past, the not enriched pebbles were produced from a mixture of $Li_4SiO_4$ and $SiO_2$ powders, but due to the fact that enriched $Li_4SiO_4$ is not available on the market, highly enriched carbonate powder was used that finally resulted in nonsatisfying pebble characteristics.

Enriched LiOH powder is commercially available, therefore, a new production route was pursued based on the following, simplified reaction:

$$4\ LiOH + SiO_2 \Rightarrow Li_4SiO_4 + 2\ H_2O$$

The melting process of LiOH and $SiO_2$ is less difficult to control than the melting of $Li_2CO_3$ in spite of the decomposition of water. The pebbles produced from LiOH and $SiO_2$ are similar to those produced from $Li_4SiO_4$ and $SiO_2$. They exhibit a distinctly dendritic structure and show only a small amount of pores and cracks. In addition to the main constituent $Li_4SiO_4$, the high temperature phase $Li_6Si_2O_7$ was detected due to the quenching process and the excess of $SiO_2$. This minor constituent, however, decomposes to $Li_4SiO_4$ and $Li_2SiO_3$ during annealing. In compressive crush load tests of single pebbles a crush load of about 9.5 N was measured for pebbles after drying at 300°C. The chemical analysis revealed a further advantage of the use of LiOH in the melting process. As LiOH is available in high-purity quality, the pebbles contain impurities to a lower degree than pebbles produced from $Li_4SiO_4$ or $Li_2CO_3$. In order to obtain characteristic pebble bed data, first Uniaxial Compression Tests (UCTs) were performed at temperatures between ambient and at 850°C. Compared to results obtained with pebbles produced from $Li_4SiO_4$ and $SiO_2$, the pebble beds with the new material show a softer behaviour, that is, a larger strain for a given uniaxial stress. Thermal creep strains do not differ remarkably.

## Introduction

In collaboration with Schott Glas, Mainz, FZK is developing and investigating slightly overstoichiometric lithium orthosilicate pebbles ($Li_4SiO_4$ + 2.5 wt% $SiO_2$, [1]) to be used in the HCPB blanket. The pebbles are fabricated in a semi-industrial scale facility by a melting and spraying method, which allows a production of 200 - 300 kg/year [2].

The characteristics of the final product are strongly influenced by the fabrication parameters, which are rather difficult to monitor in the small facility and difficult to maintain for different batches. Consequently, the reproducibility from one production run to the other is not very high,

and it is therefore required to control the quality of each batch of pebbles received from the industrial producer, in order to use a well-defined standard material in all experimental activities with pebbles or pebble beds.

Until 2002 $Li_4SiO_4$ and $SiO_2$ powders were used to fabricate not enriched lithium orthosilicate pebbles, whereas $Li_4SiO_4$ had to be totally or partly substituted by $Li_2CO_3$ in order to produce [6]Li-depleted or [6]Li-enriched pebbles. During the melting of $Li_2CO_3$ a large amount of gaseous $CO_2$ was generated, that finally led to an unduly high porosity in the produced OSi pebbles. In 2001 for the first time LiOH and $SiO_2$ had been used as raw materials. This year new batches were produced and the first characterization results will be described in this paper.

As LiOH powder is available with [7]Li or [6]Li, any desired isotope ratio can be obtained with the same process. In spite of the fact that a large amount of $H_2O$ is generated during melting, the melting process is easier to control than in case of $Li_2CO_3$. A further advantage of using LiOH is the high purity level of the material that gives rise to reduced impurities in the product. This is important to avoid possible activation of fusion materials by neutron irradiation [3,4].

## Fabrication Process

$Li_4SiO_4$ pebbles with a slight surplus of $SiO_2$, are produced by melting a mixture of LiOH and $SiO_2$ powders and then spraying the liquid material in air. The sprayed material solidifies during the flight and is collected as pebbles with different sizes. Pebbles having a diameter in the range of 0.25 – 0.63 mm have been selected for the use as breeder materials.

Fig. 1 shows the pebble production unit. The premixed components LiOH and $SiO_2$ are melted at about 1450°C in a 1 l container made of Pt-alloy. During the melting at 1450°C for 1 h and before spraying, the bottom feeder is kept closed by maintaining it at low temperatures. To start the spraying process, the bottom feeder is heated and the melt begins to flow down at a rate of approx. 200 g/min. The glass with a temperature of about 1350°C leaves the bottom feeder with a flow having a diameter between 1.0 and 1.5 mm. The melt is then sprayed with a hot gas jet with a temperature at air nozzle of about 400°C to form hot glass droplets of irregular shape. Due to surface tension the droplets become spherical while flying in air. As the gas jet cannot be well controlled, hot glass droplets are sprayed into a broad area and the flight times vary in a wide range.

By the discontinuous fabrication process in the semi-industrial scale facility two melts/day can be performed with a yield of about 1.5 kg of pebbles each. The production volume is of 200 – 300 kg/year of lithium orthosilicate pebbles.
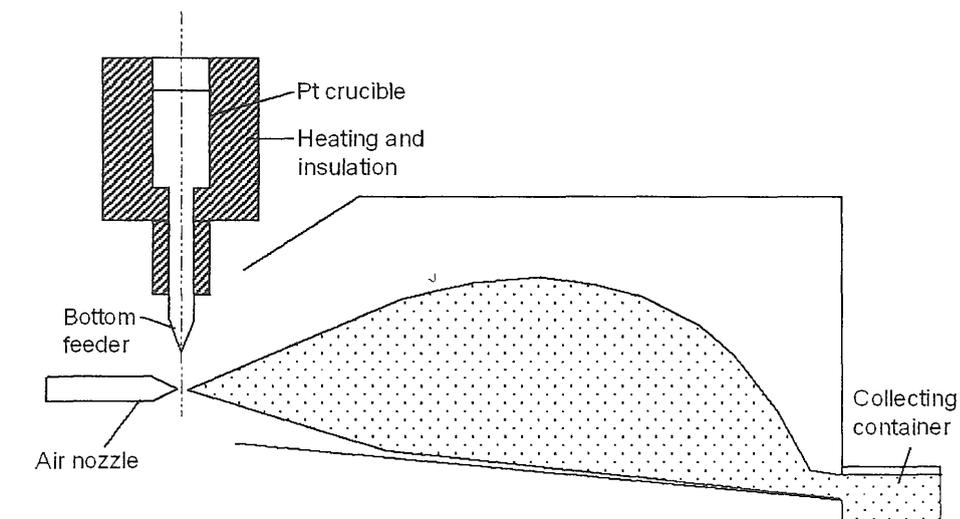


Fig. 1: Schematic drawing of the laboratory unit for the production of lithium orthosilicate pebbles by Schott Glas.

## Results and Discussion

The quality control of the different pebble batches is based on the following tests:

- Measurement of the size distribution and of the microstructural properties of the pebbles by optical and scanning electron microscopy;
- Measurement of density and porosity by Hg-porosimetry and He-pycnometry;
- Compressive crush load tests on single pebbles;
- Chemical analysis (Li, Si, impurity level);
- Phases analysis by X-ray powder diffraction;
- Thermomechanical tests on pebble beds.

### *Microscopy*

The appearance and microstructure of the batches were examined by optical and scanning electron microscopy. Most of the pebbles appear 'pearl white', but there are also some small glassy pebbles (fig. 2). Due to the rapid quenching from the melt, most of the pebbles exhibit the typical dendritic microstructure at the surface as well as in cross sections, spreading out over the whole pebble and containing a network of interdendritic micropores (figs. 3 and 4) [5, 6]. In some cases, if a smaller pebble is captured by a larger droplet, the already solidified small sphere is acting like a seed crystal and the dendrites of the larger pebble set in at the surface of the smaller pebble (OSi 03/1-3 in fig. 4 left.)
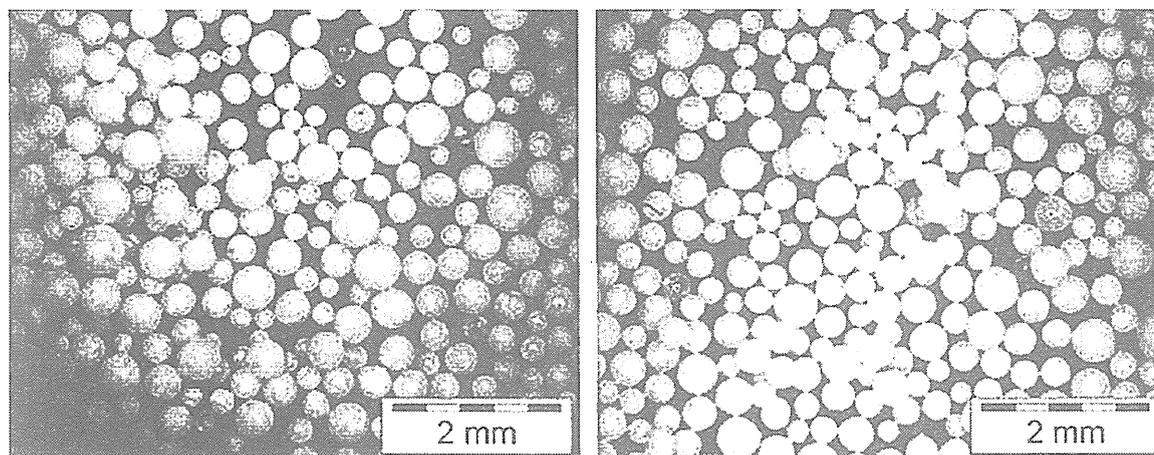


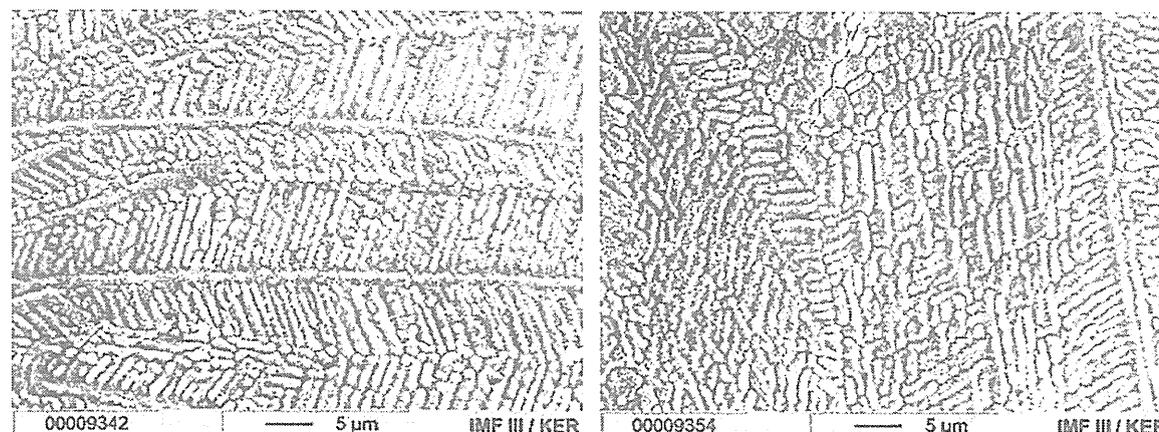Fig. 2: Overview of the batches OSi 03/1-3 (left) and OSi 03/1-4 (right). (OM)



Fig. 3: Dendritic structure at the pebble surface of OSi 03/1-3 (left) and OSi 03/1-4 (right). (SEM)
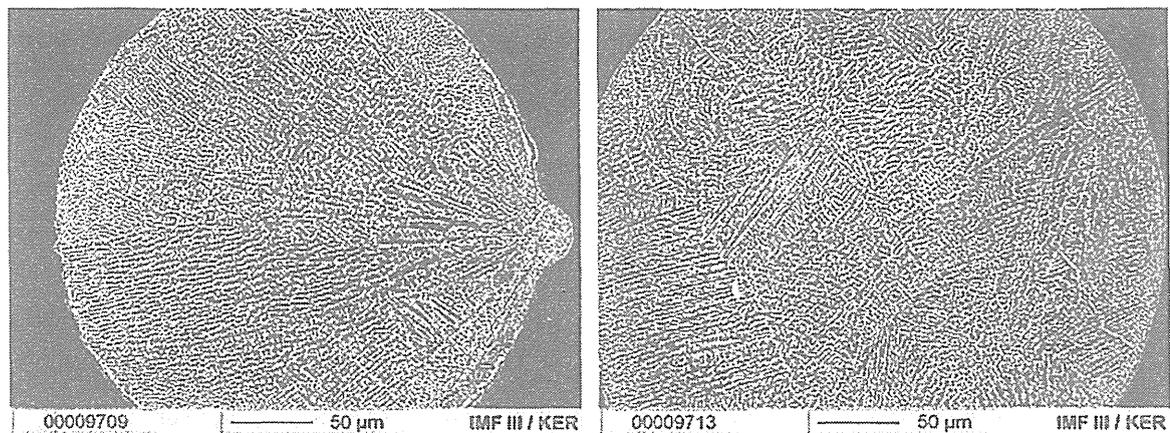
Fig. 4: Dendritic structure at cross sections of OSi 03/1-3 (left) and OSi 03/1-4 (right). (SEM)

The cross sections in figure 5 reveal only a small amount of cracks and pores for OSi 03/1-3, although a few pebbles exhibit larger pores. The batch OSi 03/1-4 has a larger amount of cracks and pores. Particularly at the surface layer of the pebbles a larger amount of micropores was detected.
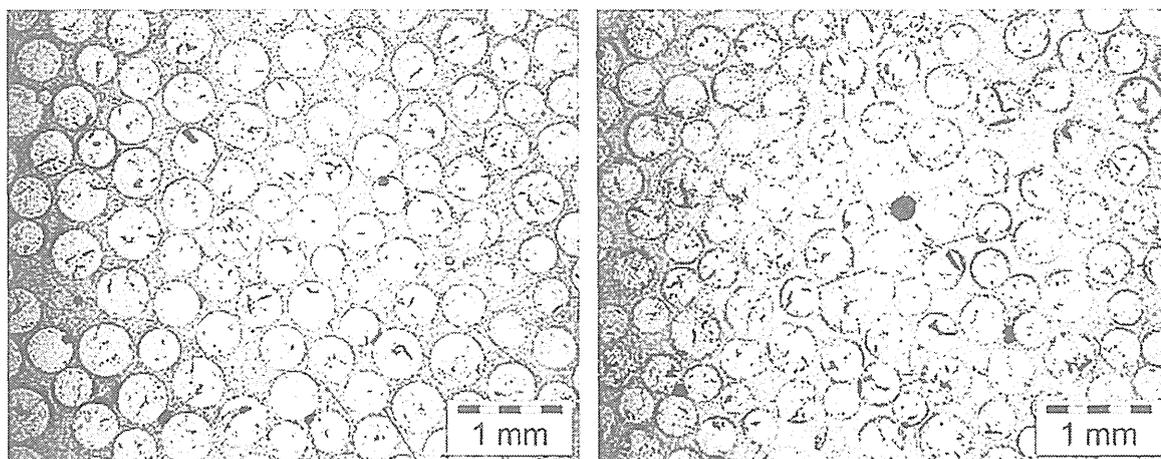


Fig. 5: Cross sections of OSi 03/1-3 (left) and OSi 03/1-4 (right). (OM)

## Size Distribution

The produced pebbles were screened to a diameter range of 250 – 630 µm, resulting in fractions with a mean particle diameter ($d_{50}$) of 310 and 345 µm for the two pebble batches OSI 03/1-3 and 03/1-4, respectively (fig. 6 and tab. 1). The maximum of the distribution is asymmetrically shifted to smaller diameters in both cases, with a more distinct tendency in case of OSi 03/1-3. The overall particle size distribution and therefore also the selected range of 250 – 630 µm is strongly dependent on the flow rate of the melt that is difficult to control in the semi-industrial scale facility.

## Porosity and Density

He-pycnometry was used to measure the bulk density of the pebbles. In this method the open porosity is not included, and the bulk density takes into account only the material density and the closed porosity. To determine the open porosity Hg-porosimetry was used. The measurement of the open porosity by the Hg-intrusion, however, is afflicted with errors, as it is very difficult to distinguish between the intrusion of voids between the pebbles and the intrusion
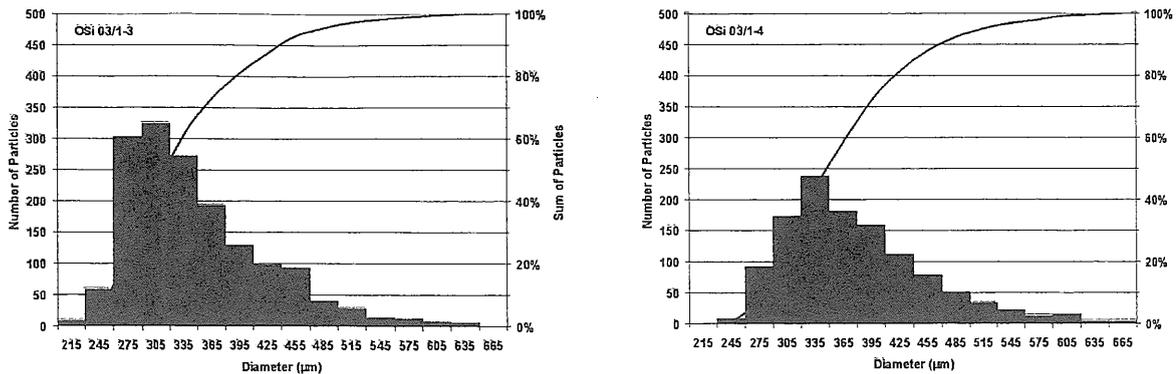
Fig. 6: Diameter distribution of the lithium orthosilicate pebble batches OSi 03/1-3 and /1-4.

of the first pores at low pressures. To make sure that all voids between the pebbles were filled with mercury, two low-pressure cycles were performed before the final run were started. In Table 1 the measured values are given together with the corresponding percentage of the calculated theoretical density of the pebbles (TD = 2.4 g/cm$^3$). For the OSi 03/1-3 batch a closed porosity of 2.7 % were determined by both methods. The open porosity of 3.5 % and a density of about 94 % were measured by Hg-porosimetry. In case of batch OSi 03/1-4 a closed porosity of 3.0 % and 1.4 % were measured by He-pycnometry and Hg-porosimetry, respectively. The higher density of 95 % also does not seem to be in agreement with the impression received from the microscopic studies. In addition to the uncertainty of the methods, these to some extent contradictory results between He-pycnometry, Hg-porosimetry and the microscopic images may be explained by a not representative sampling due to segregation of different pebble sizes. If a small sample quantity varies in the number of little and often glassy pebbles and big pebbles with more cracks and (macro-)pores, the differences between the results may arise.

## Crush Loads

To investigate the differences in the mechanical stability of the pebbles, crush load measurements were performed on 40 single pebbles of each batch. Prior to the crush load measurements the pebbles were dried at 300°C. The load application rate was 0.2 mm/min. Single pebbles with a diameter of 500 μm were placed on a glass plate and pressed by a glass piston to avoid any plastic deformation of the press parts that could influence the measurements. Mean values of 10.1 ± 2.8 (± 27.7%) and 9.0 ± 2.4 N (± 26.7%) were determined for OSi 03/1-3 and OSi 03/1-4, respectively. These values correspond to the crush loads usually measured at the standard OSi materials. Figure 7 displays the distribution of 40 crush load measurements of OSi 03/1-3. In spite of the fact that only pebbles with a diameter of 500 μm were measured, the variation of crush loads is quite large, ranging from the minimum value of 5.7 N to the maximum of 16.2 N. The differences may result from different orientations of the dendritic structure and especially from different crack orientations in the pebbles to the normal force of the crush load.

## Specific Surface Area

The specific surface area of the batches were determined by one-point BET. The OSi 03/1-3 and OSi 03/1-4 pebbles have a specific surface area of 0.20 and 0.25 m$^2$/g, respectively. These values are in the range of the usually measured value of the standard OSi material [7].
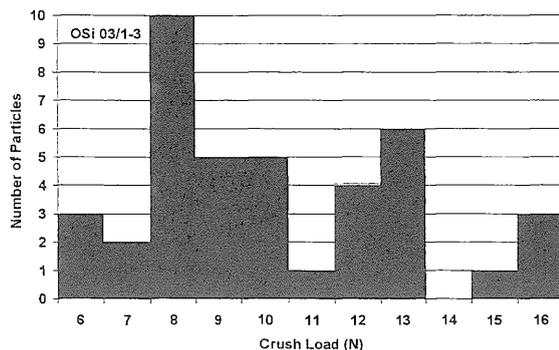
Fig. 7: Crush load distribution of 40 measurements of OSi 03/1-3.

Table 1: Physical properties of lithium orthosilicate pebbles ex lithium hydroxide and silica

| Batch | OSi 03/1-3 | OSi 03/1-4 |
|---|---|---|
| Size Distribution | | |
| $d_{10}$ / μm | 260 | 275 |
| $d_{50}$ / μm | 310 | 345 |
| $d_{90}$ / μm | 435 | 465 |
| He-Pycnometry | | |
| inner density / $g/cm^3$ | 2.34 ± 0.01 | 2.33 ± 0.01 |
| inner density / % TD | 97.3 | 97.0 |
| closed porosity (calc.) / % | 2.7 | 3.0 |
| Hg-Porosimetry | | |
| density / $g/cm^3$ | 2.25 ± 0.05 | 2.28 ± 0.05 |
| density / % TD | 93.8 | 95.0 |
| open porosity / % | 3.5 | 3.7 |
| inner density / $g/cm^3$ | 2.34 | 2.37 |
| inner density / % TD | 97.3 | 98.6 |
| closed porosity (calc.) / % | 2.7 | 1.4 |
| Crush Load Tests | | |
| crush load / N | 10.1 ± 2.8 | 9.0 ± 2.4 |
| Specific Surface Area | | |
| specific surface area / $m^2/g$ | 0.20 | 0.25 |

*Chemical Analysis*

Table 2 shows the results of the chemical analysis of the pebble batches ex lithium hydroxide and silica. The $SiO_2$ excess of the batches is in good accordance with the desired value of 2.5 wt%. A lithium loss by evaporation can be neglected in this process [8]. Due to the high purity level of LiOH the impurities of the product could be reduced. The amount of aluminium in the pebbles should not be higher than 0.006 wt% in order to reduce activation problems with $^{27}$Al

[9,10]. The determined amount of aluminium of about 0.002 wt% is much lower than the maximum limit and meets the demands of the specification.

Table 2: Chemical analysis of lithium orthosilicate pebbles ex lithium hydroxide and silica

| Batch | OSi 03/1-3 | OSi 03/1-4 |
|---|---|---|
| **Principal Constituents / wt%** by Schott Glas | | |
| $Li_2O$ | 48.44 ± 0.10 | 48.34 ± 0.04 |
| $SiO_2$ | 51.12 ± 0.08 | 51.17 ± 0.18 |
| *(excess SiO₂)* | *(2.4)* | *(2.6)* |
| **Impurities / wt%** by FZK, IMF I | | |
| C | 0.054 ± 0.002 | 0.046 ± 0.001 |
| Al | 0.00238 ± 0.00016 | 0.00189 ± 0.00025 |
| Ca | 0.00231 ± 0.00014 | 0.00157 ± 0.00011 |
| Co | < 0.0002 | < 0.0002 |
| Cr | < 0.0001 | < 0.0001 |
| Cu | < 0.0001 | < 0.0001 |
| Fe | 0.00028 ± 0.00011 | 0.00025 ± 0.00008 |
| K | 0.00072 ± 0.00012 | 0.00060 ± 0.00003 |
| Mg | 0.00024 ± 0.00007 | 0.00012 ± 0.00002 |
| Mn | < 0.00005 | < 0.00005 |
| Na | 0.00122 ± 0.00031 | 0.00102 ± 0.00008 |
| Ni | < 0.0001 | < 0.0001 |
| Ti | 0.00032 ± 0.00012 | 0.00024 ± 0.00007 |
| Zn | < 0.0002 | < 0.0002 |
| Zr | < 0.0001 | < 0.0001 |

*Phase Analysis By X-Ray Diffraction*

To determine the phases in the delivered pebbles x-ray powder diffraction were carried out for every batch. As expected, the pebbles consist of $Li_4SiO_4$ as a main constituent and the quenched high-temperature phase $Li_6Si_2O_7$ as a minor constituent [7], which is exemplarily shown by the diffraction diagram of OSi 03/1-3 in figure 8.

*Thermomechanical Pebble Bed Tests*

For the description of the thermomechanical interaction between pebble beds and the structural material of the blanket, characteristic pebble bed data are determined experimentally by uniaxial compression tests (UCTs). In these tests, pebble beds in a cylindrical cavity are compressed in the direction of the cylinder axis and both the uniaxial stress $\sigma$ and the uniaxial strain $\varepsilon$ (defined as ratio of bed deformation to initial bed height) are measured. UCTs are used i) to determine for the first stress increase period the $\sigma$–$\varepsilon$ relation, respectively, the modulus of deformation $E = \sigma/\varepsilon$, and ii) to elaborate thermal strain correlations for $\sigma$ = const. For the thermomechanical tests the pebbles were annealed at 1000°C in air in order to decompose the metastable high-temperature phase $Li_6Si_2O_7$ into lithium orthosilicate and metasilicate. The sample OSi ex
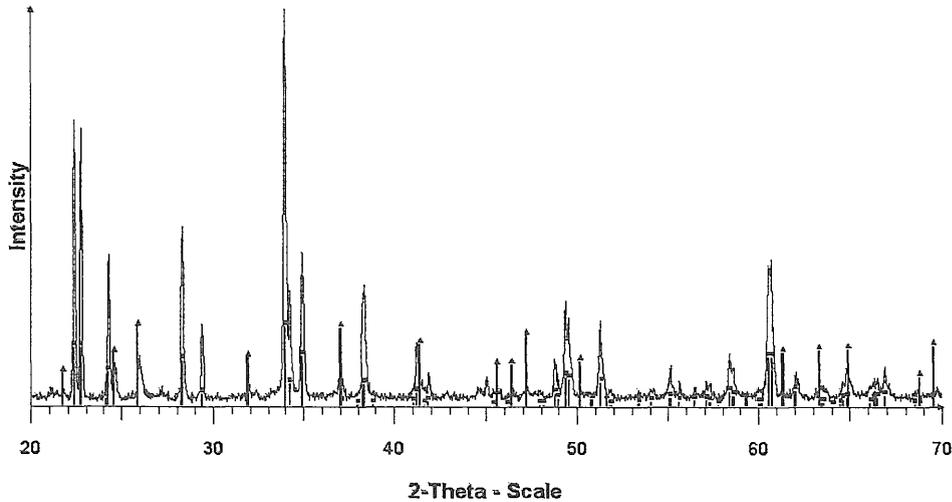
Fig. 8: X-ray diffraction of OSi 03/1-3 ( $\blacksquare$ Li$_4$SiO$_4$ $\triangle$ Li$_6$Si$_2$O$_7$).

silicate were annealed for 2 weeks, whereas the batch OSi 03/1-3 ex hydroxide were treated for 4 weeks.

First UCTs were performed at temperatures between ambient and temperatures of 850°C in a test facility already used previously [11-13]. Figure 9 shows that at ambient temperature the presently developed OSi ex hydroxide (batch 03/1-3) behaves softer (larger strains for a given stress value) than the OSi ex silicate produced until 2002. In agreement with previous measurements, the pebble beds become softer with increasing temperature; at T=850°C, this effect is significantly influenced by thermal creep occurring during the stress increase period (stress ramp $\approx$ 0.6MPa/min). After reaching $\sigma_{max}$ $\approx$ 4MPa, the stress was kept constant for about 10mins in all experiments; at 850°C strain increases remarkably due to thermal creep whereas at ambient temperature strain increases marginally due to relocations of pebbles.

Figure 10 contains the deformation modules E as a function of stress: for a wide range of $\sigma$, the slopes of the curves are fairly constant and independent of temperature; the curves can be well fitted by an expression of the type $E = C\sigma^m$, with $m = 0.5$ and about $C = 125$ for ambient temperature, see Table 3. With increasing T, C decreases as generally observed for all kinds of ceramic granular materials (compare [13]). There had been a proposal to express $C = f(T)$ [11], however, in a subsequent paper [12] it was recommended for pebble bed modelling to use the value of C at ambient temperature and to describe the bed softening by the thermal creep model. For the new OSi pebbles the temperature dependence is larger than for the OSi ex silicate pebbles. The data base for the new OSi is still quite limited. At present, for pebble bed modelling the procedure according to [12] is proposed.

First thermal creep strain results for $\sigma$ = const and different temperatures T are presented in figure 11. In this plot the initial creep strain during the pressure increase period was taken into account (compare [12]). Then, the slopes of the curves become again fairly constant for nearly the total range of creep times and are also independent of temperature and stress. Creep strain is correlated by an expression of the type $\varepsilon_{cr} = A\exp(-B/T(K))\,\sigma(MPa)^p\,t(s)^n$, details are given in Table 3. Compared to OSi ex silicate pebbles, the same value for stress exponent p was observed, and a slightly larger value for the time exponent n. In total, thermal creep strain is slightly larger for the new OSi and the temperature dependence is somewhat more expressed (for $t=10^5$s: $\varepsilon_{cr}/\varepsilon_{cr\,OSi\,ex\,silicate}$ = 1.05, 1.16 for T= 700°C and 850°C, respectively).

Microscopic investigation of both samples revealed a larger amount of lithium metasilicate in case of the longer treated OSi 03/1-3 sample. Most of the metasilicate is located at the surface of the pebbles and at grain boundaries. As the formation of an outer metasilicate layer increases the surface roughness of the pebbles, the softer behaviour of OSi ex hydroxide in the pebble bed tests may arise from the longer annealing time.

**Table 3:** Correlations for modulus of deformation, E, and thermal creep strain, $\varepsilon_{cr}$.

| Granular material | $E(MPa) = C\sigma(MPa)^m$ | | $\varepsilon_{cr}(1) = A\ exp(-B/T(K))\ \sigma(MPa)^p\ t(s)^n$ | | | |
|---|---|---|---|---|---|---|
| | C | m | A | B | p | n |
| OSi ex silicate [12-14] | 180* | 0.47 | 12.1 | 10220 | 0.65 | 0.20 |
| OSi ex hydroxide (OSi 03/1-3) | 125* | 0.50 | 20.1 | 11005 | 0.65 | 0.23 |

*T=25°C
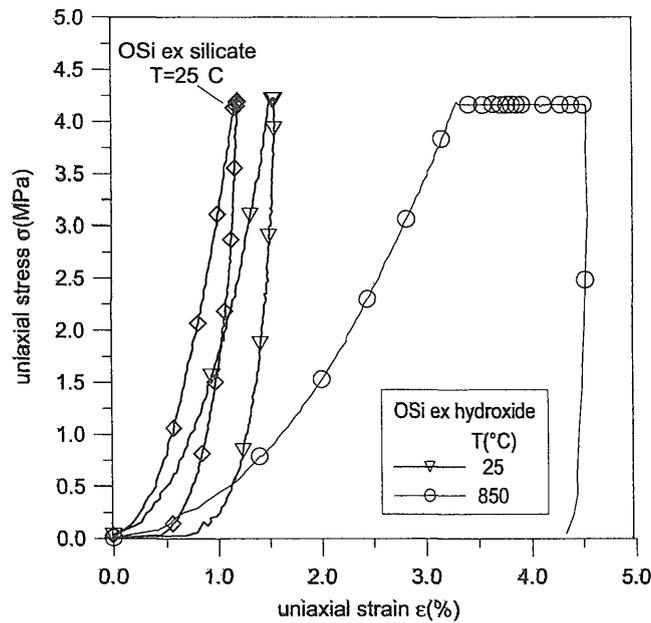


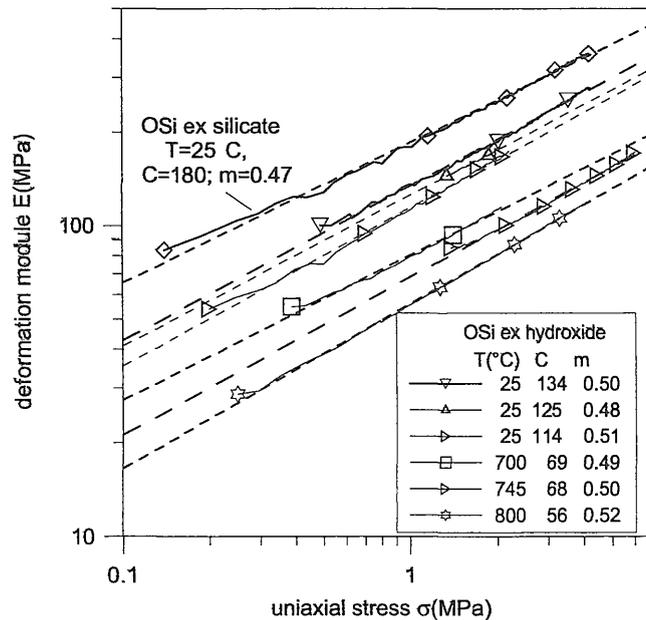Fig. 9: Stress-strain dependence for T=25°C and T=850°C.



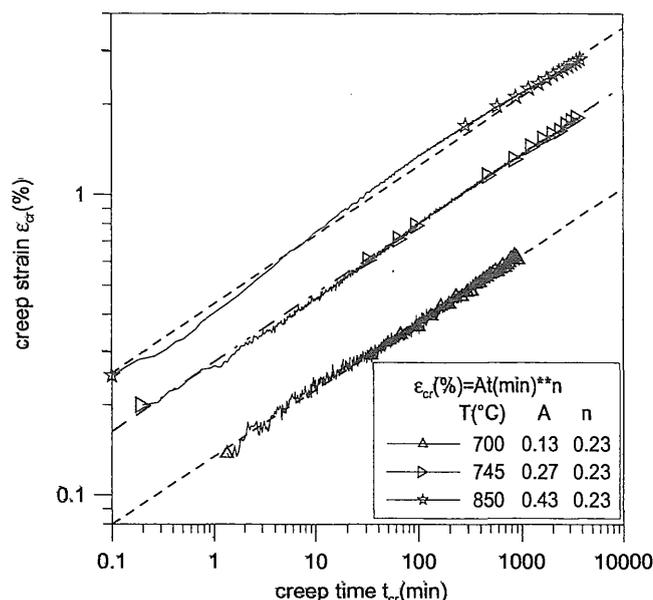Fig. 10: Modulus of deformation as a function of uniaxial stress.

Fig. 11: Creep strain as a function of creep time.

The experimental results of the two batches OSi 03/1-3 and 1-4 demonstrate that the new production route using LiOH and $SiO_2$ as raw materials lead to breeding material with a high quality that meets the demands of the specification. The results, however, also reveal the poor reproducibility of the fabrication process in the semi-industrial scale facility. The differences in size distribution, density, porosity and crush load may be explained by taking into account the technical conditions of this facility:

The heated air jet can only be adjusted in horizontal and vertical direction in front of the flowing melt, therefore hot glass droplets are sprayed into a broad area and the flight times vary in a wide range, affecting the pebble shape.

The flow rate is determined by the static pressure exerted by the weight of the molten lithium orthosilicate in the crucible, which diminishes continuously during the run due to the decreasing glass volume. Further reduction of the flow may be caused by the gradual blocking of the feeder.

The reduction in flow rate and some instability in the gas jet strongly influences the final characteristics of the pebbles, usually only about 50 % of the produced pebbles have acceptable characteristics in terms of size and shape.

Micro cracks arise in the bulk of the pebbles due to the rapid cooling of about 50 K/s and may also be generated in the pebbles by hitting the wall of the collecting container. Pores may be caused by entrapped air during the formation of droplets or by the volume contraction during solidification and crystallization. Both, cracks and pores, strongly influence the mechanical strength of the pebbles.

To determine all parameters that influence the pebble characteristics and the reproducibility, fundamental experiments were recently carried out at Schott Glas. In these experiments parameters such as the pressure and temperature at the air jet and the melt temperature were varied. In addition, at each experiment some pebbles were quenched in liquid nitrogen. First observations show that the pebble shape is strongly affected by the decreasing flow rate of the melt, i. e. the decreasing hydrostatic pressure. In the early stages of the experiments all produced pebbles exhibit round spheres. But within a short time, i. e. with decreasing flow rate, more irregular parts are formed. Reproducibility and the rate of yield should therefore be increased in a continuous process in an upgraded facility, which provides a better control of the process parameters.

## Conclusions

The new pebble batches OSi 03/1-3 and 03/1-4 produced from lithium hydroxide and silica show properties very similar to the previous standard material produced from $Li_4SiO_4$. Compared to the first batches made from LiOH (OSi 01/3-4) the porosity and the amount of cracks could be reduced. In the new batches the amount of the $SiO_2$ excess could be adjusted to $2.5 \pm 0.1$ wt %. In addition, the high purity level of LiOH leads to reduced impurities in the final product, which now meets the requirements of the specification. Summarizing the first experimental results from the thermomechanical tests it can be stated that pebble beds consisting of the first batches of OSi ex hydroxide pebbles are characterized by a softer behaviour during the first pressure increase period and, to a smaller extent, in respect to thermal creep compared to the previously produced OSi ex silicate. A softer behaviour results in smaller stresses during the heat-up phase and faster stress relaxation processes at high temperatures; effects which are presumed to be favourable for blanket operation. In the future, more experiments are required in order to enlarge the pebble bed data base for this new material.

Taking into account the availability of LiOH with different isotopes, the new fabrication route, using LiOH and $SiO_2$ for the melting and spaying process, should be favoured for the production of further lithium orthosilicate pebbles. An important advantage of the melting and spraying process is the possibility to recycle all rejections as well as depleted material from the breeding process.

## Acknowledgement

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