Fusion Engineering and Design 89 (2014) 1426–1430

Contents lists available at ScienceDirect

Fusion Engineering and Design

journal homepage: www.elsevier.com/locate/fusengdes

Influence of chemisorption products of carbon dioxide and water vapour on radiolysis of tritium breeder

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HIGHLIGHTS

- Chemisorption products affect formation proceses of radiation-induced defects.
- Radiolysis of chemisorption products increase amount of radiation-induced defects.
- Irradiation atmosphere influence radiolysis of lithium orthosilicate pebbles.

ARTICLE INFO

Article history:
Received 19 August 2013
Received in revised form 9 December 2013
Accepted 8 January 2014
Available online 31 January 2014

Keywords:
Tritium breeder
Lithium orthosilicate pebbles
Radiolysis
Chemisorption products

ABSTRACT

Lithium orthosilicate pebbles with 2.5 wt% excess of silica are the reference tritium breeding material for the European solid breeder test blanket modules. On the surface of the pebbles chemisorption products of carbon dioxide and water vapour (lithium carbonate and hydroxide) may accumulate during the fabrication process. In this study the influence of the chemisorption products on radiolysis of the pebbles was investigated. Using nanosized lithium orthosilicate powders, factors, which can influence the formation and radiolysis of the chemisorption products, were determined and described as well. The formation of radiation-induced defects and radiolysis products was studied with electron spin resonance and the method of chemical scavengers. It was found that the radiolysis of the chemisorption products on the surface of the pebbles can increase the concentration of radiation-induced defects and so could affect the tritium diffusion, retention and the released species.

1. Introduction

Lithium orthosilicate pebbles (0.25–0.63 mm) with 2.5 wt% excess of silica are the European Union's reference tritium breeding material for the Helium Cooled Pebble Bed (HCPB) Test Blanket Modules (TBMs) [1–3]. Pebbles with lithium orthosilicate as main phase have appropriate tritium breeding parameters, i.e. good tritium release behaviour, high melting point and lithium density [3]. Beside the main task to produce and release tritium, the pebbles also must be able to withstand the harsh conditions as expected in the TBMs over the long time of operation [3]. In the HCPB TBMs tritium breeding material will be exposed to an intense neutron flux ($\Phi \leq 10^{18}$ neutrons m^{-2} s^{-1}), a high magnetic field ($H = 7$–10 T) and temperature ($T = 573$–1193 K) [1,2].

The most promising method for the lithium orthosilicate pebble fabrication is a melt-based process [3–8]. For the synthesis, lithium hydroxide and silica are used as raw materials [4,6,8]. The 2.5 wt% excess of silica is added to increase mechanical stability of the pebbles [3]. The mixture of raw materials is heated to about 1723 K and the then formed liquid is sprayed in dry air, to obtain pebbles [6–8]. Due to the excess of silica (Eq. (1)) and the rapid quenching, the resulting product has two phases – lithium orthosilicate as the main and lithium orthodisilicate as the minor phase [6–8]. After annealing at 1243 K in air, lithium orthodisilicate phase decomposes (Eq. (2)) and pebbles mainly consist of 90 mol% lithium orthosilicate and 10 mol% lithium metasilicate [6–8].

$$10\mathrm{LiOH} + 3\mathrm{SiO_2} \rightarrow \mathrm{Li_4SiO_4} + \mathrm{Li_6Si_2O_7} + 5\mathrm{H_2O}\uparrow \tag{1}$$

$$\mathrm{Li_6Si_2O_7} \rightarrow \mathrm{Li_4SiO_4} + \mathrm{Li_2SiO_3} \tag{2}$$

Latest analysis of the pebbles surface [7] showed that after annealing in air also carbon dioxide may accumulate and is only released at temperatures >773 K [8]. The carbon dioxide mainly accumulates as chemisorption product, i.e. lithium carbonate (Eq. (3)) [9]. Traces of lithium carbonate on the surface of the pebbles were observed in depths less than 1 μm [7]. The formation

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http://dx.doi.org/10.1016/j.fusengdes.2014.01.005

of lithium carbonate probably occurs due to a too slow cooling rate after annealing or contact with air at storage stage.

$$Li_4SiO_4 + CO_2 \rightarrow Li_2CO_3 + Li_2SiO_3 \quad (3)$$

The chemisorption process of carbon dioxide can be catalyzed by air humidity (Eqs. (4) and (5)) [10]. Therefore small amounts of lithium hydroxide and absorbed water may also accumulate on the surface of the pebbles.

$$Li_4SiO_4 + H_2O \rightarrow 2LiOH + Li_2SiO_3 \quad (4)$$

$$2LiOH + CO_2 \rightarrow Li_2CO_3 + H_2O \quad (5)$$

The lithium carbonate and hydroxide are radiation unstable compounds [11] and may affect the formation of radiation-induced defects (RD) and radiolysis products (RP) during neutron and other type irradiation. The importance and role of RD and RP on the tritium diffusion and retention has been established [12–15]. Most crucial are electron type RP, i.e. colloidal lithium, which can interact with tritium and may disturb tritium diffusion and decrease its retention [15]. The colloidal lithium forms at the radiolysis of the lithium orthosilicate (Eq. (6)) as result of coagulation of the electron type RD [15].

$$Li_4SiO_4 \xrightarrow{\text{Ionizing radiation}} 2Li + Li_2SiO_3 + 0.5O_2 \quad (6)$$

The aim of this study is to estimate the influence of the chemisorption products of water vapour and carbon dioxide on the radiolysis of the lithium orthosilicate pebbles. In addition, nanosized powders were used to determine and investigate the factors which can influence the formation and radiolysis of chemisorption products.

2. Experimental

2.1. Fabrication of ceramic specimens

The lithium orthosilicate pebbles (screened to 0.45–0.56 mm), together with nanosized powders with three different compositions were selected for investigation (Table 1). The nanosized powders were used due to their high surface area and smaller grain size compared to the pebbles. The increased surface area increases the amount of chemisorption products [8,11].

The pebbles were produced by a melt-spraying method at Schott AG (Mainz, Germany) [4–8] and were annealed at 1243 K for 168 h in air.

The nanosized "pure" lithium orthosilicate powder was produced by plasma synthesis [16] at Institute of Inorganic Chemistry (Riga Technical University, Latvia). For the synthesis, lithium carbonate and silica were selected as raw materials. To eliminate residues of raw materials, the obtained powder was annealed at 890 K in air. The formation of 2 mol% lithium metasilicate phase can be explained by un-stoichiometry of the raw materials.

The 3 mol% (2 wt%) excess of silica was added to the "pure" powder. The mixture of both powders was homogenized by milling for 3 h in a ball mill and then annealed at 920 K for 3.5 h in air. The excess of silica was added to obtain similar composition as in the pebbles.

2.2. Preparation and irradiation of samples

The lithium orthosilicate pebbles were encapsulated in quartz tubes either with dry argon or air and were irradiated by accelerated electrons (Table 1). Irradiation was performed with the linear electron accelerator ELU-4 (Salaspils, Latvia), up to 4 h per day.

To understand the processes, which may occur during one of the irradiation cycles (up to 4 h) in air, the nanosized powders were used instead of the pebbles. The irradiation type practically do not influence the formation of RD and RP [17], and thus gamma rays instead of accelerated electrons were used, due to smaller dose rate and lower irradiation temperature. The electron flux of the linear electron accelerator was converted to gamma rays by a tungsten plate.

The nanosized lithium orthosilicate powders with three different compositions were irradiated by gamma rays at room temperature in air (Table 1), to investigate the formation and radiolysis of the chemisorption products. The dose rate and absorbed dose was reduced, to avoid formation of RP to the chemisorption products [18]. In order to identify RD of the chemisorption products, lithium carbonate and hydroxide powders were irradiated ($D = 56$ kGy, $T \approx 300$ K) as well. To investigate the thermal stability of RD, the irradiated nanosized powders were thermally treated up to 620 K for 30 min in air.

To simulate the processes, which may occur on the surface of the pebbles during one of the irradiation cycles, the nanosized lithium orthosilicate powder with 6 mol% of lithium metasilicate phase was thermally pre-treated ($T = 570$ K, $t = 4$ h) and then irradiated with gamma rays ($D = 56$ kGy, $T \approx 300$ K) in air.

2.3. Methods of characterization

The chemical composition of the ceramic specimens was analyzed by qualitative powder X-ray diffractometry (p-XRD), thermogravimetric analysis (TGA) and Fourier transformed infrared spectroscopy (FT-IR). The surface area and grain size were investigated by BET adsorption and by scanning electron microscopy (SEM), respectively. The accumulated RD and RP were analyzed with electron spin resonance (ESR) and with the method of chemical scavengers (MCS) [15–17].

The p-XRD patterns were obtained by a Bruker D8 (10–60° 2Θ, CuKα, $\lambda = 0.15418$ nm), the FT-IR spectra by a Perkin Elmer Spectrum Two (450–4000 cm^{-1}, pressed in KBr pellets) and the TGA curves by a Seiko EXTAR 6300 (290–1270 K, 2–10 K min^{-1}, dry argon and air). The ESR spectra were recorded by a Bruker BioSpin X-band radiospectrometer (300–400 mT, 30 dB, 9.83 GHz) operating at 100 kHz field modulation in room temperature. The grain size of the nanosized powders was analyzed with a Hitachi S-4800 SEM.

The MCS is based on the difference of red-ox properties of the hole and electron type RD and RP in acid containing solvents [15]. The irradiated ceramic specimens were dissolved in 0.1 M sulphuric acid solution with 1 M ethanol, to analyze the total amount of the electron type RD and RP, and with 1 M sodium nitrate, to analyze the amount of the electron type RP. In the acidic solutions the generated gaseous molecular hydrogen was obtained [17] and analyzed by gas chromatography.

3. Results and discussion

3.1. Radiolysis of lithium orthosilicate pebbles at elevated temperature in dry argon

Using MCS, it has been determined that after irradiation up to 11 GGy absorbed dose at 550–590 K in dry argon, lithium orthosilicate pebbles accumulated both simple and multi-electron centres. Up to 95% are multi-electron centres. Multi-electron centres consist of colloidal lithium aggregates, whereas simple centres – localized electrons in oxygen vacancies, so called F^+ and F° centres, and E' centres (ion-radical $SiO_3{}^{3-}$) [17]. The estimated radiation chemical yield (5.1×10^{-5} localized electrons per 100 eV) and the decomposition degree (0.15 mol%) were much smaller than determined in previous studies [15–17], most likely due to the high absorbed dose and elevated irradiation temperature.

Table 1
Characterization of the investigated lithium orthosilicate ceramic specimens and irradiation conditions.

Parameter	Lithium orthosilicate pebbles	Nanosized lithium orthosilicate powders		
		"Pure"	With 3 mol% silica	With 6 mol% lithium metasilicate
Chemical composition	90 mol% Li_4SiO_4 10 mol% Li_2SiO_3	98 mol% Li_4SiO_4 2 mol% Li_2SiO_3	95 mol% Li_4SiO_4 2 mol% Li_2SiO_3 3 mol% SiO_2	92 mol% Li_4SiO_4 8 mol% Li_2SiO_3
Grain size	10 μm [11]	200–300 nm	200–400 nm	300–600 nm
Specific surface area ($m^2\,g^{-1}$)	0.20–0.25	23 ± 2	22 ± 2	17 ± 2
Irradiation conditions	Accelerated electrons, $E = 5$ MeV, $D = 0.7–11$ GGy, $P = 88$ MGy h^{-1}, $T = 550–590$ K, dry argon and air	Gamma rays, $D = 7–56$ kGy, $P = 14$ kGy h^{-1}, $T \approx 300$ K, air		

In the ESR spectra of the pebbles, which were irradiated in dry argon, only one signal ($g = 2.001$, $\Delta H = 1$ mT) was observed (Fig. 1). It was attributed to the paramagnetic E′ centres, which have been investigated and characterized in previous experiments [11,15,17]. Presumably, the signal of colloidal lithium ($g = 2.0025$, $\Delta H \leq 10^{-2}$ mT [15]) in the ESR spectra cannot be observed due to aggregation, and the signal of F^+ centres is too broad to be analyzed.

The E′ centres together with HC_2 centres (ion-radical $SiO_4{}^{3-}$), are the primary stage electron and hole type RD of the lithium orthosilicate (Eqs. (7) and (8)) [17]. However due to the second and third stage reactions of the radiolysis [17], the concentration of the E′ centres is quite small (10^{15} radicals g^{-1}), in contrast, HC_2 centres practically do not accumulate.

$$\mathrm{SiO_4^{4-}} \xrightarrow{\text{Ioniz. radiat.}} \mathrm{SiO_4^{3-}}(\mathrm{HC_2\ centre}) + e^- \tag{7}$$

$$\mathrm{SiO_4^{4-}} + e^- \xrightarrow{\text{Ioniz. radiat.}} \mathrm{SiO_3^{3-}}(\mathrm{E'\ centre}) + O^{2-} \tag{8}$$

3.2. Influence of air on radiolysis of lithium orthosilicate pebbles at elevated temperature

Using air instead of dry argon as irradiation atmosphere of the lithium orthosilicate pebbles, not only signals of the E′ centres were observed in the ESR spectra, but also two symmetric signals ($g = 2.193$ and $g = 1.898$) with 50.2 mT splitting and two signals with g-factor 2.008 and 2.014 (Fig. 1). The signals with g-factor 2.008 and 2.014 were attributed to the HC_2 centres, whereas the two symmetric signals were attributed to atomic hydrogen.

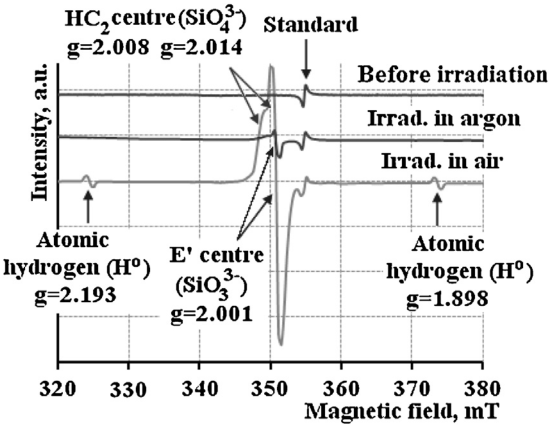

Fig. 1. ESR spectra of the lithium orthosilicate pebbles before and after irradiation ($D = 11$ GGy, $T = 550–590$ K) in dry argon and air.

In a previous study [11] the formation of atomic hydrogen was related to the radiolysis of the chemisorption products of water vapour (Eq. (9)). However the increase of the RD concentration (from 10^{15} to 10^{17} radicals g^{-1}) and the decomposition degree (from 0.15 to 1.5 mol%) as well as the formation of HC_2 centres was not completely understood.

$$\mathrm{LiOH} \xrightarrow{\text{Ioniz. radiat.}} \mathrm{LiO}^{\bullet} + \mathrm{H}^{\bullet} \tag{9}$$

In this research it has been suggested that these effects could be related to the radiolysis of the chemisorption products of water vapour (Eq. (10)) and carbon dioxide (Eq. (11)), which may form during the irradiation in air.

$$2\mathrm{LiOH} \xrightarrow{\text{Ioniz. radiat.}} 2\mathrm{Li} + \mathrm{H_2O} + 0.5\mathrm{O_2} \tag{10}$$

$$\mathrm{Li_2CO_3} \xrightarrow{\text{Ioniz. radiat.}} 2\mathrm{Li} + \mathrm{CO_2} + 0.5\mathrm{O_2} \tag{11}$$

Up to 70% of RD localize in a 50 μm subsurface layer of the pebbles, due to intrinsic structural defects [12,15]. The radiolysis of the chemisorption products on the surface of the pebbles could influence the formation processes of RD and so a rapid increase of the RD concentration and decomposition degree could be detected.

The formation and radiolysis of the chemisorption products during the irradiation could be influenced by several factors: irradiation temperature and time, absorbed dose and dose rate, chemical composition and surface area of the pebbles etc. [8–11,15–18]. Therefore to understand the processes, which may occur during one of the irradiation cycles ($D \approx 350$ MGy, $t \approx 4$ h, $T = 550–590$ K) in air, the influence of the irradiation atmosphere, the chemical composition and the irradiation temperature on the radiolysis of the pebbles were investigated separately.

The formation of RD and RP in the lithium orthosilicate pebbles take place in three main stages. After irradiation with the absorbed dose >10–20 MGy at 400–600 K mainly colloidal lithium forms, due to the coagulation of the electron type RD [15]. To avoid the formation of colloidal lithium and to accumulate mainly primary stage RD, the dose rate and absorbed dose was reduced (from 350 MGy to 56 kGy).

3.3. Influence of air atmosphere on radiolysis of lithium orthosilicate powder at room temperature

To increase the surface area and thus the amount of chemisorption products, the nanosized powders were selected for further investigations instead of the pebbles.

The "pure" lithium orthosilicate powder showed impurities of lithium carbonate and hydroxide before irradiation. In the p-XRD patterns traces of additional signals were observed (Fig. 2A). In

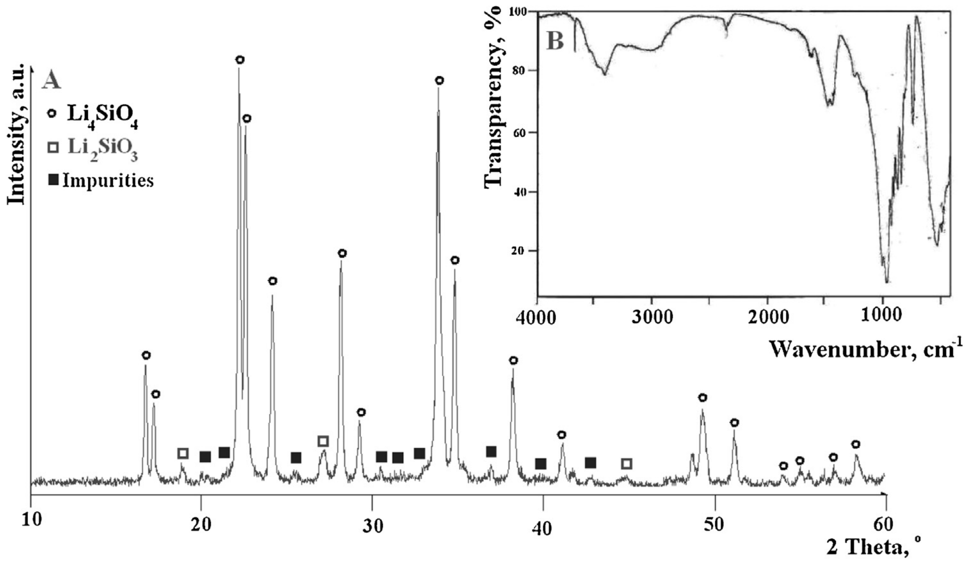

Fig. 2. p-XRD pattern (A) and FT-IR spectrum (B) of the “pure” lithium orthosilicate powder before irradiation.

the FT-IR spectrum bond vibrations of C—O (1400–1500 cm^{-1}) and O—H (2800–3500 cm^{-1}) were detected (Fig. 2B). By TGA a mass loss up to 2 wt% was observed during heating to 1073 K, which was unambiguously assigned to desorption of water (T = 420–570 K) and carbon dioxide (T = 670–870 K) [8]. The formation of the chemisorption products most likely occur during the fabrication or storage stage.

After irradiation up to 4 h (D = 56 kGy) at room temperature ($T \approx 300$ K) in air, major changes in the TGA curves, p-XRD patterns and FT-IR spectrum were not observed. Yet, due to the short irradiation time and the small absorbed dose, significant changes in the “pure” lithium orthosilicate powder were not expected [17].

However, in the ESR spectrum of the powder, seven signals were detected after irradiation (Fig. 3). Five of them are identical to the signals which were observed in the ESR spectrum of the pebbles (Fig. 1) and were identified as signals of atomic hydrogen, HC_2 and E′ centres, respectively. The formation of atomic hydrogen was attributed to the radiolysis of the lithium hydroxide. Whereas the interpretation of the two remaining signals with *g*-factors 2.026 and 2.036 is more complicated.

In the ESR spectrum of the pebbles the formation of these two signals were not observed, most likely due to high absorbed dose. In irradiated “pure” silicates these signals were often attributed to peroxide radicals ($\equiv Si-O-O^{\bullet}$) or HC_2 centres [13,17]. Yet in the ESR spectra of irradiated lithium hydroxide and carbonate powders signals with similar g-factors were observed [19]. This suggests that the origins of these signals could not be only peroxide radicals or HC_2 centres, but also paramagnetic RD of the chemisorption products. Due to that, both of them were marked as un-identified.

Other ESR signals, which could be related to RD of the chemisorption products, like, ion-radicals CO_2^- (g = 2.0006), CO_3^- (g = 2.0036) and CO_3^{3-} (g = 2.00415) [19], in the spectrum were not observed, most likely due to overlapping with signals of the E′ and HC_2 centres.

3.4. *Influence of excess of silica on radiolysis of lithium orthosilicate powder at room temperature in air*

After adding 3 mol% (2 wt%) excess of silica and subsequent homogenization by milling, the resulting lithium orthosilicate powder shows a rapid decrease of the RD concentration – up to 40% (Fig. 4). The silica practically does not influence the surface area and the grain size, thus the decrease of RD concentration could be related to the chemical properties of silica. In the homogenization process silica most likely accumulate on the surface of the

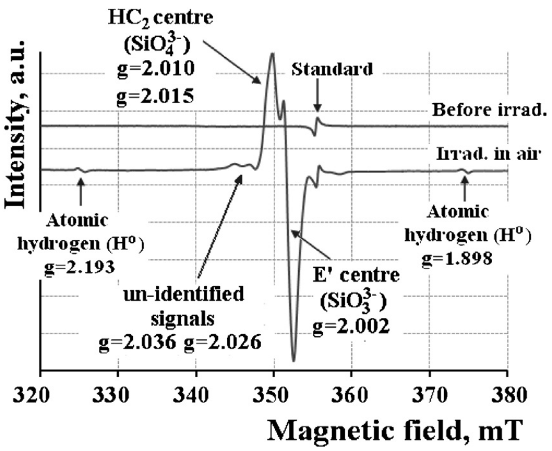

Fig. 3. ESR spectra of the “pure” lithium orthosilicate powder before and after irradiation (D = 56 kGy, $T \approx 300$ K) in air.

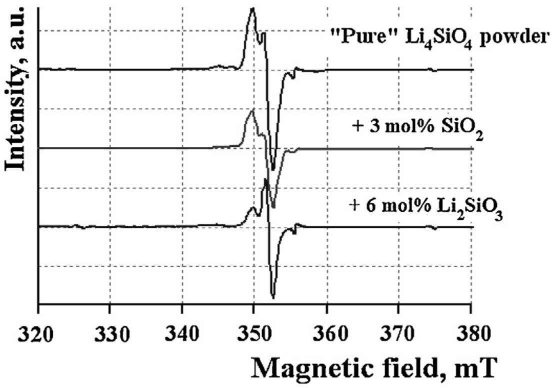

Fig. 4. ESR spectra of the “pure” lithium orthosilicate powder, with 3 mol% of silica and with 6 mol% of lithium metasilicate phase after irradiation (D = 56 kGy, $T \approx 300$ K) in air.

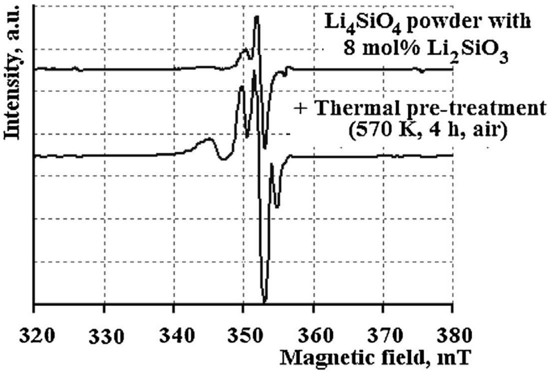

Fig. 5. ESR spectra of the irradiated (D = 56 kGy, $T \approx 300$ K, air) lithium orthosilicate powder with 6 mol% lithium metasilicate before and after thermal pre-treatment (T = 570 K, t = 4 h) in air.

lithium orthosilicate grains and so possibly disturb the formation of chemisorption products.

After annealing up to 920 K in air, silica on the surface of the grains reacts with the lithium orthosilicate (Eq. (12)) [20,21] and the surface area decreases from 22 to 17 m^2 g^{-1}, probably due to particle agglomeration. The formed 6 mol% lithium metasilicate phase also decrease the RD concentration – up to 25% (Fig. 4). The decrease of RD concentration was attributed both to a change of the contact surface area with air and the properties of lithium metasilicate [17,22].

$$Li_4 SiO_4 + SiO_2 \xrightarrow{CO_2 \text{ and } H_2O} 2Li_2SiO_3 \quad (12)$$

3.5. *Influence of irradiation temperature on radiolysis of lithium orthosilicate powder in air*

To simulate the processes, which may occur on the surface of the pebbles during one of the irradiation cycles, the nanosized lithium orthosilicate powder with 6 mol% lithium metasilicate phase was used for further experiments.

The MSC and ESR results suggest that at elevated temperature the atomic hydrogen recombines at 320 K, HC$_2$ centres at 400–550 K, un-identified RD at 420–520 K and E′ centres at 450–650 K. Besides that, investigations by TGA in air, suggest that simultaneously with thermally stimulated recombination of RD, the formation of the chemisorption of water vapour and carbon dioxide may occur up to 420 K [21]. At higher temperature both gases are released in two steps: first occurs desorption of water (T = 420–573 K) and then carbon dioxide (T = 670–920 K) [21]. On basis of these results it has been sugested that at 550–590 K in air, besides recombination of RD, on the surface of the pebbles mainly the formation and radiolysis of the chemisorption products of carbon dioxide occurs.

After thermal pre-treatment at 570 K for 4 h in air, using TGA, it has been determined that the lithium orthosilicate powder contained up to 12 wt% of water vapour and carbon dioxide. Due to the radiolysis of the chemisorption products, an increase of RD concentration – up to 50%, was detected after irradiation (Fig. 5). The obtained results confirm that the chemisorption products increase not only the concentration of E′ centres, but also the amount of HC$_2$ centres and un-identified RD.

The obtained results clearly correlate with the data of the lithium orthosilicate pebbles, which were irradiated up to 11 GGy at 550–590 K in air (Fig. 1). These results also confirm the suggestion that the chemisorption products, due to the formation up to 420 K and radiolysis, can increase the concentration of the hole and electron type RD and the decomposition degree of the pebbles.

4. Conclusions

In this study the influence of the chemisorption products of water vapour and carbon dioxide (lithium hydroxide and carbonate) on the radiolysis of the lithium orthosilicate pebbles was investigated. It was concluded that the radiolysis of the chemisorption products can significantly affect the formation of the hole and electron type RD on the surface of the pebbles. It was found that the chemisorption products can increase the concentration of RD and the decomposition degree of the pebbles. Due to the radiolysis of the chemisorption products on the surface of the pebbles and increase of RD concentration, tritium diffusion could be affected.

Processes were described, which may occur on the surface of the pebbles during irradiation in air. It was determined that the formation and radiolysis of the chemisorption products on the pebbles depend on the chemical composition of the surface and the irradiation temperature. The excess of silica, due to the chemical properties, can disturb the formation of the chemisorption products and so could decrease the concentration of RD. Whereas at elevated irradiation temperature, due to the formation of the chemisorption products up to 420 K from air, the concentration of RD can significantly increase.

Acknowledgments

This study was carried out in cooperation with Institute of Inorganic Chemistry (Riga Technical University) and Faculty of Chemistry (University of Latvia). Research was financed by Ministry of Education and Science (Republic of Latvia), project EURATOM No. 11-11/ES12.

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