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Summary Report on IGM characteristics

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Document title

IGM: A porous free graphite matrix for a long term safe storage of irradiated graphite (i-graphite)

Executive summary

IGM is a development of FNAG as an alternative waste form for the management of irradiated graphite. It can be assumed as a vitrification of i-graphite. However the glass has only a volume of 20% which is close to the pore volume of bulk graphite. The manufacturing of IGM is based on a hot pressing of the glass-graphite mixture in vacuum. This leads to a material with densities better than 98% of theoretical density and has no significant open pores larger than 0.1 µm.

The glass corrosion has been investigated by weight changes and potassium release of IGM samples with different glass types immersed in water and magnesium rich salt brine. The glass 8800 of Schott has been identified as the most corrosion resistant glass type.

Therefore further investigations will be focused on this glass type for the qualification of IGM as waste embedding matrix

Revisions

Rev.	Date	Short description	Authors	Internal Review	Task Leader	WP Leader
01	26.03.2013	Final Report	J.Fachinger	M.Oppenländer	E. Visser-Tynova	B. Grambow

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1 Introduction

Graphite itself is a geologically stable material proven by its natural occurrence (1). However its porous structure enables the penetration of aqueous phases into the graphite (2) and therefore radionuclides can be leached (3). However i-graphite can be transformed into long term stable impermeable matrix (impermeable graphite matrix IGM) which inhibits the ingress of water. This allows a safe final disposal. Such a graphite composite material with glass as an inorganic binder has been developed by FNAG. The composite has densities better than 99 % of theoretical density and therefore a negligible porosity. Figure 1 shows such an IGM sample. The inner zone consists of virgin graphite material from UK Magnox reactors mixed with mineral and metallic scrap. The out zone is made of natural graphite which represents an inactive coating of the radioactive material in the proposed final product. This shows also the possibility to utilize raw crushed i-graphite inclusive scrap in the proposed process and the possibility to embed other radioactive waste stream in IGM

Figure 1: IGM sample with virgin Magnox graphite mixed with mineral and metallic scrap

2 Manufacturing of IGM

The following Figure 2 shows a general description for the management of i-graphite from a reactor core. First of all the graphite has to be retrieved from the core. This can be performed under water or in atmosphere. The graphite can be retrieved as block and will be milled after retrieval to an acceptable grain size. An alternative could be an in situ size reduction of the graphite inside the vessel as well as the following size reduction by nibbling, crushing, milling or electro-pulse segregation. The wet route requires an additional drying step for the further processing. The drying can be performed separately or simultaneously with the filling of the mould or HIP can.

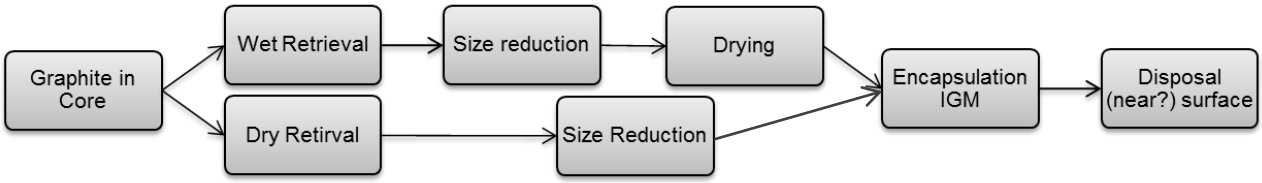


Figure 2: Optional process route for handling i-graphite

The Figure 3 shows a manufacturing of the IGM a little more detailed. The crushed i-graphite will be mixed with an inorganic binder e.g. borosilicate glass. The amount of the inorganic binder is in the same range of the pore volume of the former graphite parts which is about 20 vol%. An optional compaction and granulation step is eventually beneficial to increase the filling density in the mold which would reduce the size of the pressing equipment. Finally the mixture is pressed under vacuum at elevated temperatures. The temperature is chosen with respect to the viscosity of the glass which should be in the range of 10^4 dPa s. The vacuum is required to remove any gases which would form pores during the pressing step.

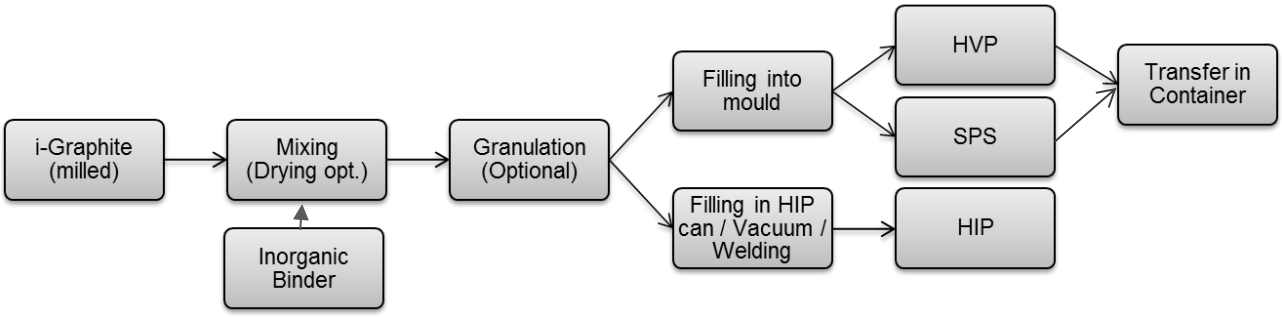


Figure 3: Process scheme for manufacturing IGM

The pressing of the IGM can be performed by three different methods:

- Hot vacuum pressing (HVP)
- Spark Plasma Sintering (SPS)
- Hot Isostatic Pressing (HIP)

HVP and SPS are axial vacuum presses whereas the mould of a HVP is heated by external heating elements and the SPS heats the material to be pressed by a current directly through the material. The advantage of the SPS is a faster process because the IGM will be heated up directly in opposite to the HVP where the mould is heated. This enables a faster cooling.

The third method, HIP, has larger differences to the two methods mentioned above. The pressing will not be performed by a punch. The can filled with IGM granulate is placed in a pressure vessel (Figure 6) which will be heated up. The pre-pressure of the gas plus the thermal pressure increase are adjusted to the final required pressure. The can is pressed isostatic from all sides and form an anisotropic product.

Figure 4: HVP pressing mould and external graphite heating elements

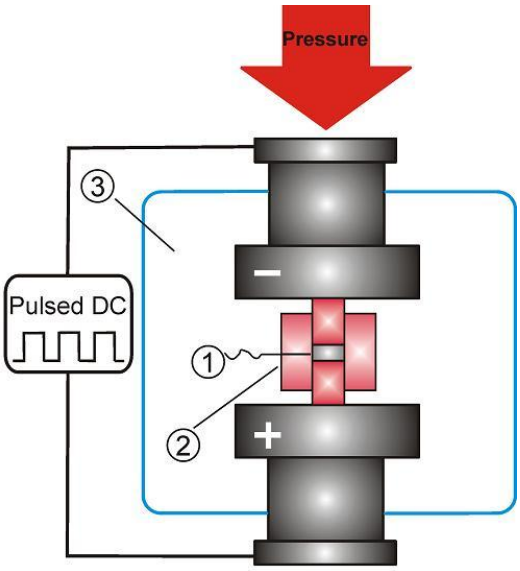


Figure 5: Schematic drawing of SPS pressing device

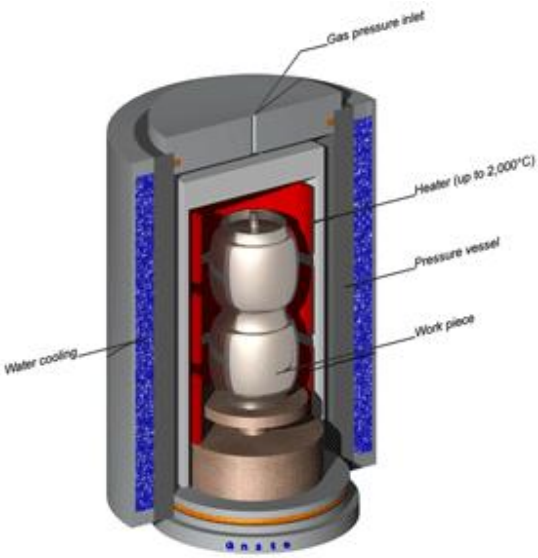


Figure 6: Scheme of a HIP

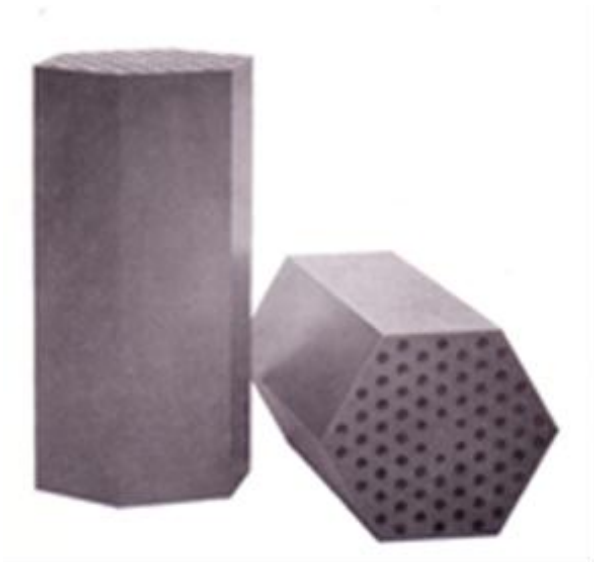


Figure 7: IGM block with holes

The pressed canister can be removed from the vessel at elevated temperatures (~400 to 500°C) and can cool down separately whereas the HIP can be used already for the next pressing. This reduces the process time. A further advantage is the canister itself. It provides an outer shell free from contamination.

Additional the IGM waste block can be surrounded by a shell of radioactive waste free IGM. Therefore the IGM can be prepared with several small holes (Figure 7) or one large hole as green body. This space can be filled with pure waste or a mixture of waste and IGM powder. Finally a top cap of IGM powder is added before final pressing.

An alternative is the direct filling of the mould or a HIP canister by different filling nozzles one for the centre with the waste/IGM mixture and one for the outside with uncontaminated IGM mixture. In lab scale the filling of the outer and inner zone can be achieved by separation of the zone with a paperboard cylinder.

3 Material properties

3.1 Sample composition and manufacturing parameters

Table 1: Manufacturing parameter

Number	Mixture[1]	Mass [g]	Temperature [°C]	Force [kN]
F_001	V1G	164	1270	200
F_002	V2G	164	1270	200
F_003	V3G	164	1055	200
F_004	V3G	700	1055	200
F_005	V3G	730	1055	200
F_006	V3G	580	1055	200
F_007	V17G	4.225	1270	700
F_008	V17G	1.438,5	1270	302
H_001	V3G	250	1065	113
H_002	V3G	273	1065	113
H_003	V3P	251,5	1065	113
H_004	V3P	215	1065	81,2
H_005	V3P	215	1065	81,2
H_006	V3P	216	1065	81,2
H_007	V5P	215	1300	81,2
H_008	V5P	215	1300	81,2
H_009	V5P	215	1300	81,2
H_010	V5P	215	1300	81,2
H_011	V6P	410	1300	154
H_012	V6P	410	1300	154
H_013	V11P	329	1020	78,5
H_014	V13P	406	1300	154
H_015	V12P	404	1300	154
H_016	V12G	420	1300	154
H_017	V15P	405	1300	154
H_018	V16P	405	1300	154
H_019	V19P	410	1200	154
H_020	V19P	405	1300	77
H_021	V7P	405	1300	154
H_022	V19P	406	1100	154
H_023	V21P	405	1300	154
H_024	V22P	409	1100	154

[1] See Table 2

Number	Mixture[1]	Mass [g]	Temperature [°C]	Force [kN]
H_025	V23P	416	1100	154
H_026	V24P	409	1100	154
H_027	V25P	408	1000	154
H_028	V26P	410	1000	154
H_029	V27P	411	1000	154
H_030	V28P	404	1150	154
H_031	V29P	408	1100	154
H_032	V30P	407	1100	154
H_033	V31P	408	1100	154
H_034	V32P	407	1000	154
H_035	V33P	407	1000	154
H_036	V34P	407	1000	154
H_037	V36P	413	1200	154
H_038		435	1300	154
H_039		410	1300	154
H_040		420	1300	154
H_041	V17G	425	1100	154
H_042	V17G + Graphit-kugeln	410	1300	154
H_043	V41P + Graphit-kugeln	480	1300	154
H_044	V17G	412	1300	154
H_045	V42P	410	1100	154
H_046	V17G	420	1300	154
H_047	V17G	415	1300	154
H_048	V17G + Graphit-kugeln	521,21	1300	154
H_049	V17G	420	1300	154
H_050	V44P	420	1150	154
H_051	V20G	420	1300	154
H_052	V44P	700	1000	154
H_053	V45P	740	770	135
H_054	V46P	675	770	135
H_055	P47P	400	1700	154
H_056	V48P	400	1600	154
H_057	V47P	450	1700	154
H_058	C/SiO_2 P	420	1100	154

Number	Mixture[1]	Mass [g]	Temperature [°C]	Force [kN]
H_059	N3/G4.22	550	1200	154
H_060	N3/S5/G4	490	1200	154
H_061	S5/G4.22	475	1200	154
H_062	N3/G4.22	420	1200	154
H_063	S5/G4.22	420	1200	154
H_064	V4/G4.22	435	1200	154
H_065	V4/G4.22	460	1200	154
H_066	V17G	420	1300	154
H_067	V17G	420	1300	154
H_068	V17G	420	1300	154
H_069	V35	400	1300	154
H_070	V35	420	1100	154
H_071	V35	~ 500	1300	154
H_072	V17	460	1300	154
H_073	V60	440	1300	154
H_074	V59	435	1300	154
H_075	N3/G1.20	430	1100	154
H_076	N3/ G23.25	460	1100	154
H_077	V17	468	1300	154
H_078	V17	483	1300	154
H_079	V24	420	1100	154
H_080	V36	420	1200	154
H_081	V28	420	1150	154
H_082	V75	420	1300	154
H_083	V78	425	1200	154
H_084	V78	420	1200	154
H_085	V75	420	1300	154

Table 2: Composition of IGM mixtures

Mixture	Graphite				Glass	
	Natural Graphite		Synt. Graphite			
	Type	Amount	Type	Amount	Type	Amount
V1	WGB	64,00%	CS-Grade	16,00%	8250	20,00%
V2	WGB	80,00%			8250	20,00%
V3	WGB	80,00%			8330	20,00%
V6			KBR2000	80,00%	8330	20,00%
V7			KBR2000	85,00%	8330	15,00%
V9	WGB	80,00%			8330	20,00%
V10			Magnox-Graphit fein	80,00%	8330	20,00%
V12			Magnox-Graphit fein	80,00%	8330	20,00%
V13			Magnox-Graphit fein	80,00%	8330	20,00%
V17	SGB25L/99,9	80,00%			8330	20,00%
V18			Magnox-Graphit Fließbett	80,00%	8330	20,00%
V19			KBR2000	80,00%	8330	20,00%
V20	SGB25L/99,9	80,00%			8330	20,00%
V21			KBR2000	90,00%	8330	10,00%
V22	SGB25L/99,9	78,40%			A0	21,60%
V23	SGB25L/99,9	78,40%			A1	21,60%
V24	SGB25L/99,9	78,40%			A2	21,60%
V25	SGB25L/99,9	77,90%			C0	22,10%
V26	SGB25L/99,9	77,90%			C1	22,10%
V27	SGB25L/99,9	77,90%			C2	22,10%
V28	SGB25L/99,9	77,90%			E0	22,10%
V29	SGB25L/99,9	78,50%			F0	21,50%
V30	SGB25L/99,9	78,50%			F1	21,50%
V31	SGB25L/99,9	78,50%			F2	21,50%
V32	SGB25L/99,9	75,90%			P0	24,10%
V33	SGB25L/99,9	75,90%			P1	24,10%
V34	SGB25L/99,9	75,90%			P2	24,10%
V35	SGB25L/99,9	80,00%			8330	20,00%
V36	SGB25L/99,9	80,00%			8800	20,00%
V37	SGB25L/99,9	80,00%			8330	20,00%
V38	SGB25L/99,9	80,00%			8330	20,00%
V39	SGB25L/99,9	64,00%	KBR2000	16,00%	8330	20,00%
V40	SGB25L/99,9	80,00%			8330	20,00%
V41	SGB25L/99,9	80,00%			8330	20,00%
V42	SGB25L/99,9	80,00%			8250	20,00%
V43			KBR2000	80,00%	8330	20,00%

V49	SGB25L/99,9	75,00%			8330	25,00%
V50	SGB25L/99,9	75,00%			8250	25,00%
V51	SGB25L/99,9	80,00%			8250	20,00%
V52			Magnox-Graphit fein	80,00%	8250	20,00%
V53			Magnox-Graphit grob	80,00%	8250	20,00%
V56	FP M99,5LCL	78,00%			8800	22,00%
V57	FP M99,5LCL	62,40%	Magnox-Graphit Fließbett	15,60%	8800	22,00%
V58			Magnox-Graphit Fließbett	78,00%	8800	22,00%

3.2 Influence of temperature and pressure on density

The negligible porosity of IGM is one of the main parameters for the safe enclosure of the radioactive waste. The porosity is directly related to the density of the IGM material. The density depends on the pressing parameters temperature corresponding to the viscosity of the glass and pressing force. Therefore the influences of these parameters have been studied.

First the influence of temperature was investigated with a constant force of 2 kN/cm². Therefore 10 samples were prepared for each temperature out of the pure natural graphite and the glass 8250. The results are given as mean values in Figure 8. A maximum density was achieved at temperatures above 1050°C. This temperature corresponds to glass viscosities of 10^4 dPas or less. Similar tendencies have been observed for other glasses however no statistical evaluations have been performed.

The influence of the pressing force has been investigated with samples out of natural graphite and the glass 8330. The chosen temperature was 1250°C which corresponds to a viscosity of 10^4 dPas. Ten samples had been prepared for each pressing force. The results revealed that a pressure of 5.6 kN/cm² is sufficient to obtain the required minimum density of 2.2 g/cm³. The maximum density 2.23 g/cm² has been obtained with a force of 20 kN/cm². However this last data point has been evaluated with only 3 samples. Therefore further investigations will be performed to evaluate the statistical significance of the pressing with 20 kN/cm^2 as well as study of the further pressing forces between 7 and 20 kN/cm² to evaluate whether the density of 2.23 g/cm³ can be achieved at lower pressures than 20 kN/cm².

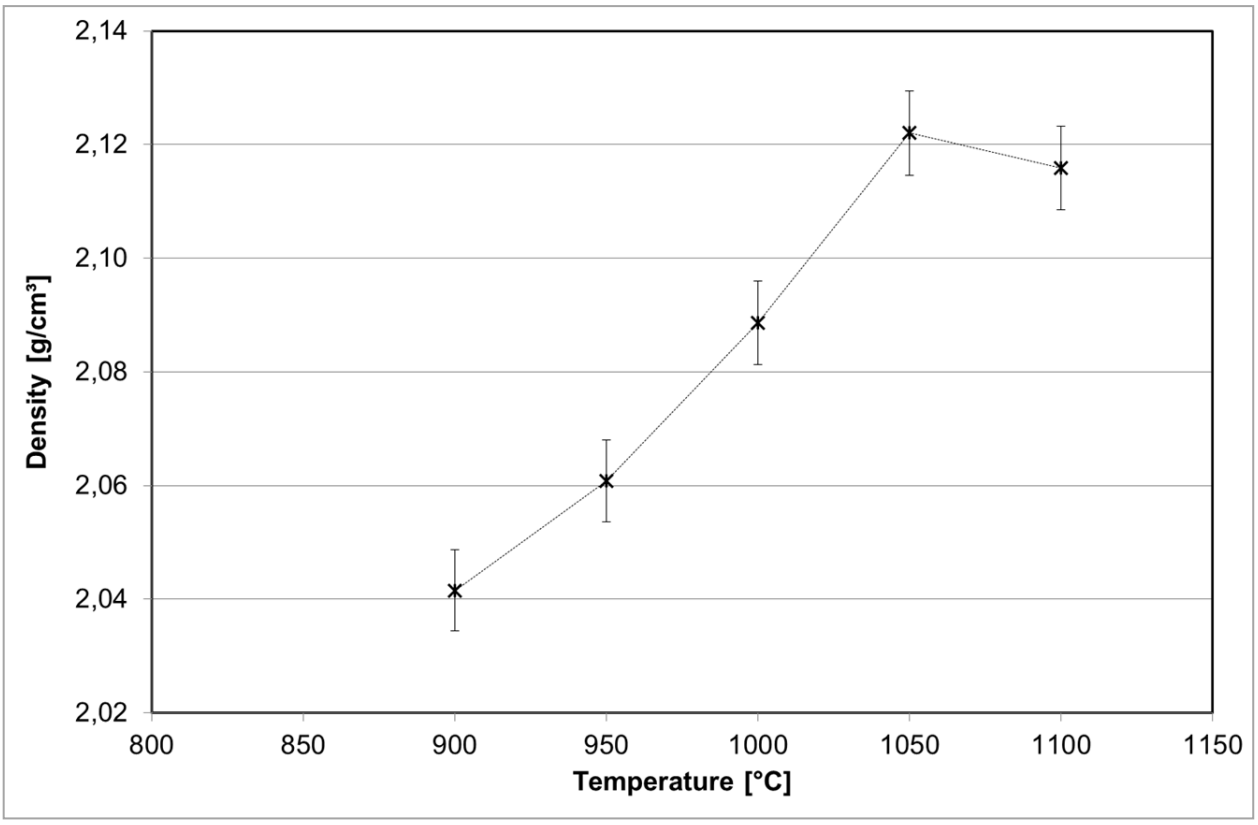


Figure 8: Dependence of density from temperature

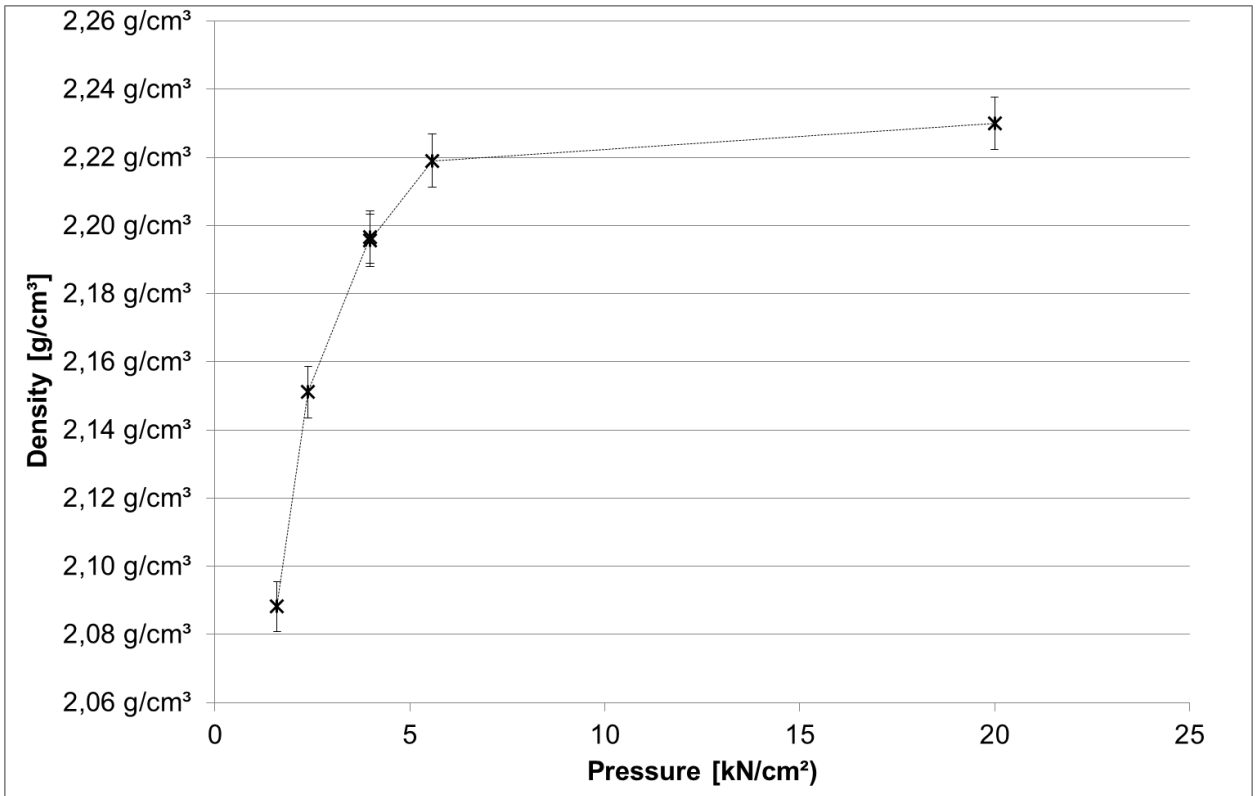


Figure 9: Dependence of density from pressing force.

3.3 Mechanical Strength

The determination of the strength was performed with samples of 5 x 5 x45 mm for the bending strength and sample 10 x 10 x 10 mm for the compression strength. The samples were cut from blocks with a diameter of 50 to 70 mm and a high of about 60 mm. The final samples were precisely shaped by a moulding cutter after a raw cut with a band saw as shown in Figure 10. It was carefully distinguished between samples parallel and vertical to the direction of the pressing. The number of samples for the bending strength could be doubled if no samples for the determination of the compressing strength were required.

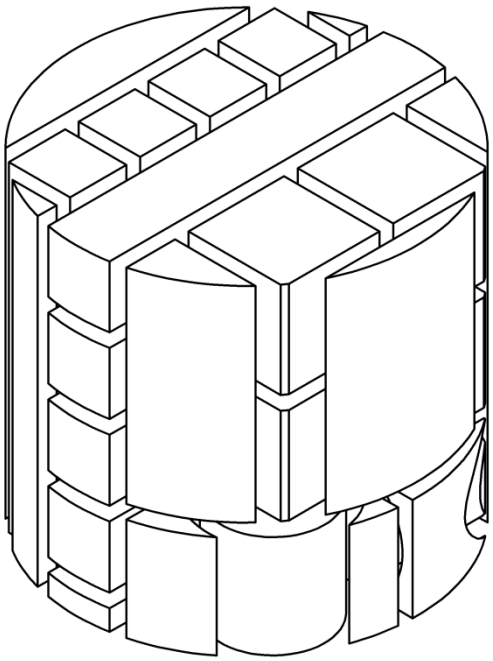


Figure 10: Cut scheme for samples to measure the mechanical strength

Bending strength

The bending strength depends clearly on the sample direction. Samples cut parallel to the press direction have a mean bending strength of 21 MPa and perpendicular samples 47 MPa. These are average values from 102 samples with different materials compositions, temperatures from 1100°C to 1300°C and pressures from 2 to 4 kN/cm².

Figure 11 shows the statically variation of the bending strength cut from one IGM bloc out of synthetic graphite KRB2000 and glass Schott 8330. The large deviations form ~ 50 to ~ 60 MPa can be explained by small defects at the edges of the samples which lead to a lower bending strength. A significant increase of the bending strength can be obtained by the addition of carbon fibres.

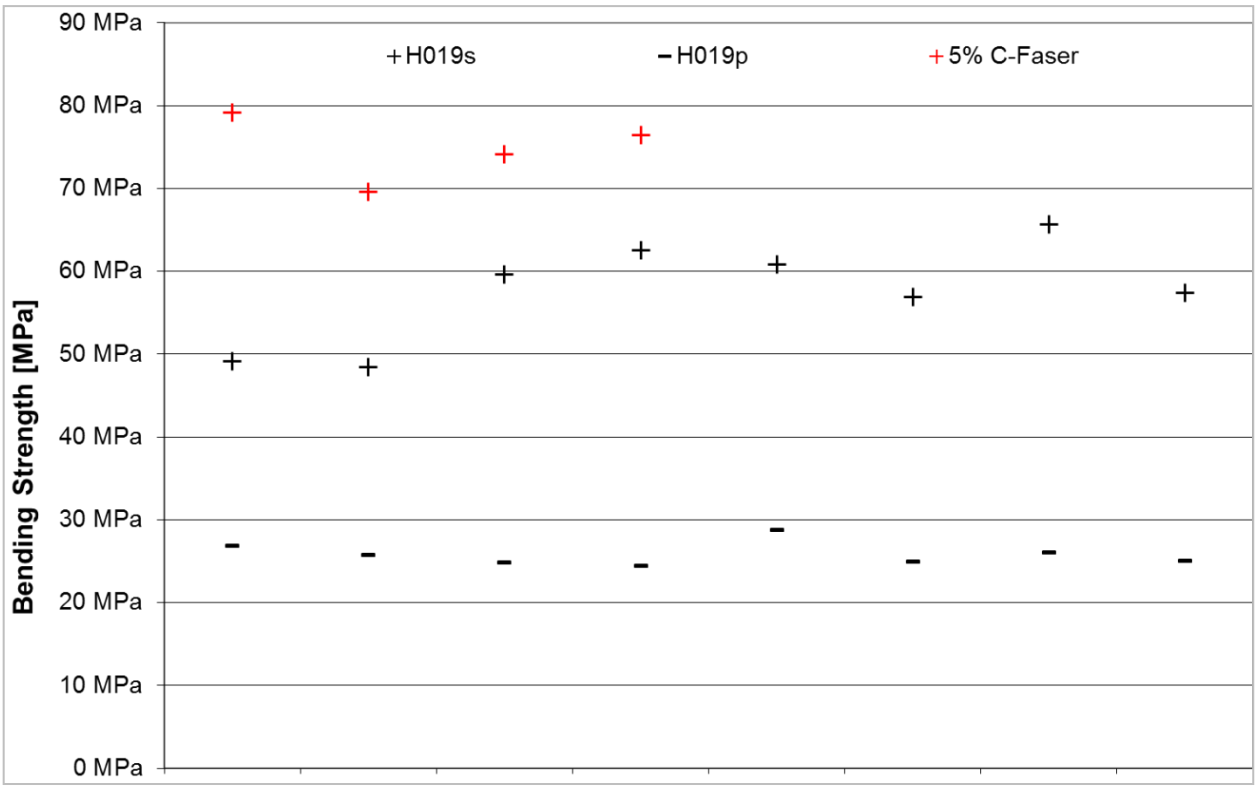


Figure 11: Bending strength of sample H019 and H019 plus carbon fibers

The compressive strength has only been determined for the two IGM blocks F004 and F006. The obtained strength was 51 and 67 MPa respectively. The related bending strength was in the range of 31 MPa for perpendicular samples and 19 MPa for parallel samples.

Anyhow the obtained results prove a sufficient strength of the IGM material e.g. the required compressive strength of 10 MPa for waste packages for the repository side Konrad in Germany is given for all samples.

3.4 Physical properties

3.4.1 Thermal properties

The heat capacity, the thermal diffusivity and the heat conductivity have been determined for the samples F006, H008 and H009.

The results measurements are shown in Figure 12, Figure 13 and Figure 14. It can be clearly distinguished between the thermal diffusivity and heat conductivity perpendicular and parallel to the direction of the pressing. This anisotropic behaviour is expected due the anisotropic properties of the graphite itself.

The thermal diffusivity and the heat conductivity of graphite decrease with temperature. The same behaviour has been observed for the IGM samples. The heat conductivity at 100°C is approximately 90 to 95% of the heat conductivity at room temperature (1). The estimated heat conductivity at 20°C is in the range of 110 W/(m K) for the F-samples and approximately ~ 100 W/(m K) for the H-samples. Typical values for graphite are in the range of 100 to 170 W/(m K).

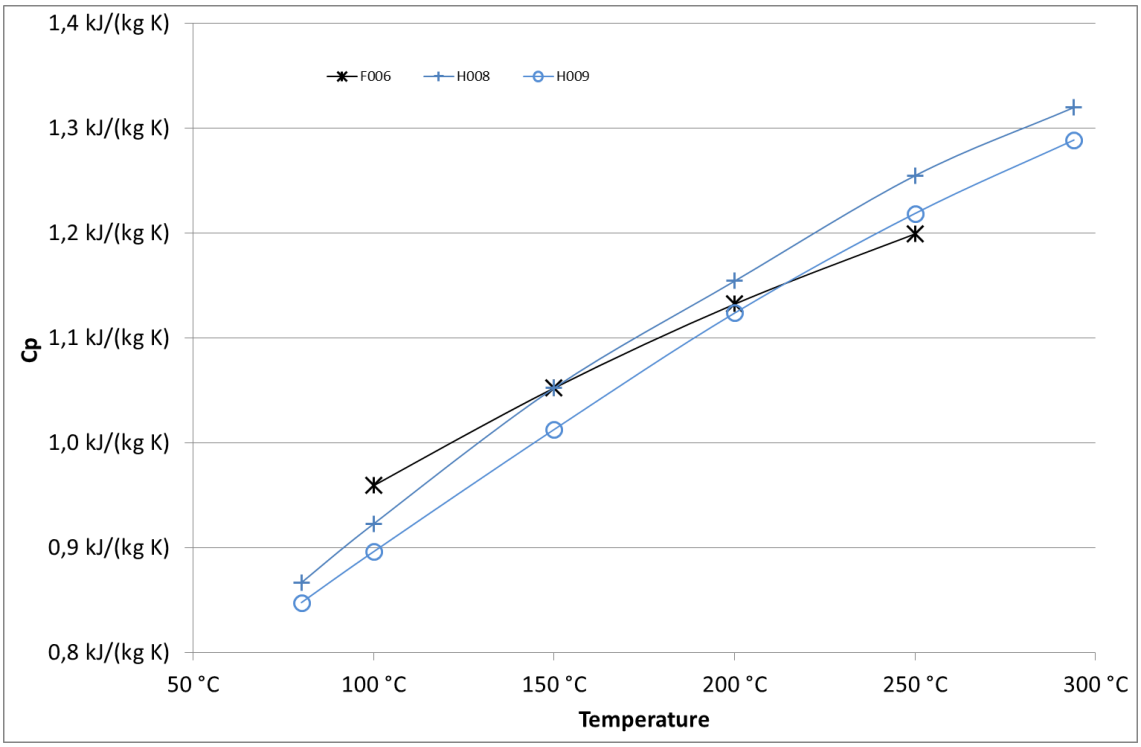


Figure 12: Cp measurement of IGM

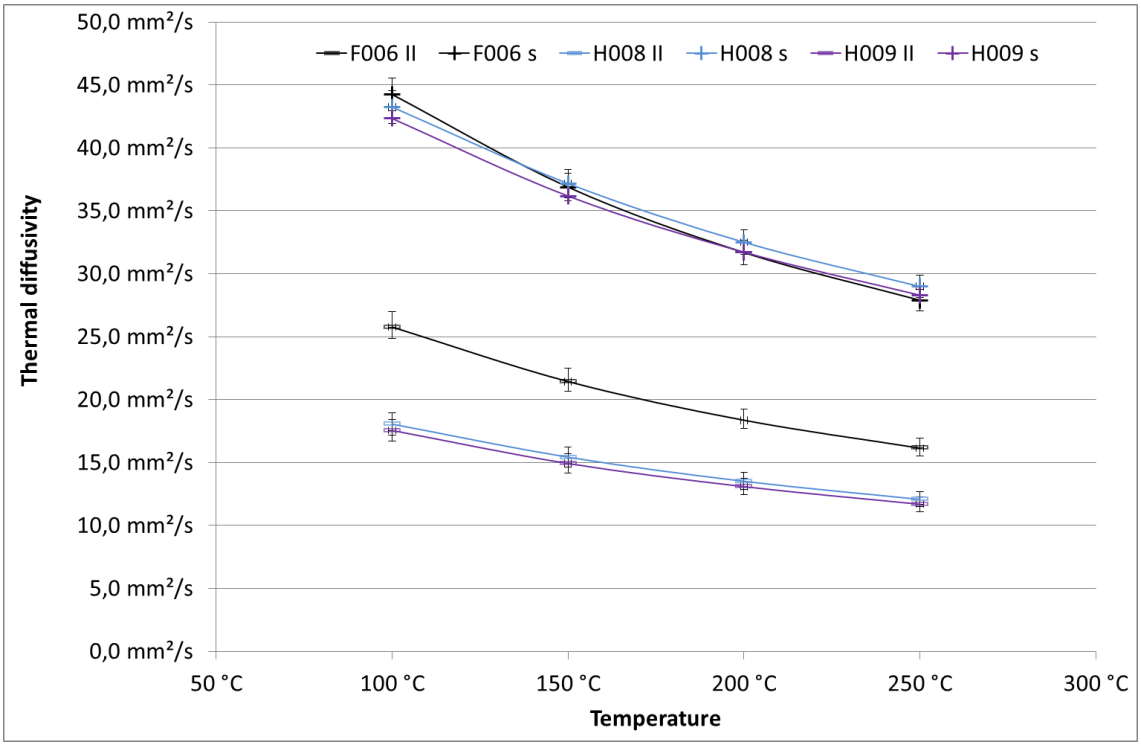


Figure 13: Temperature Diffusivity

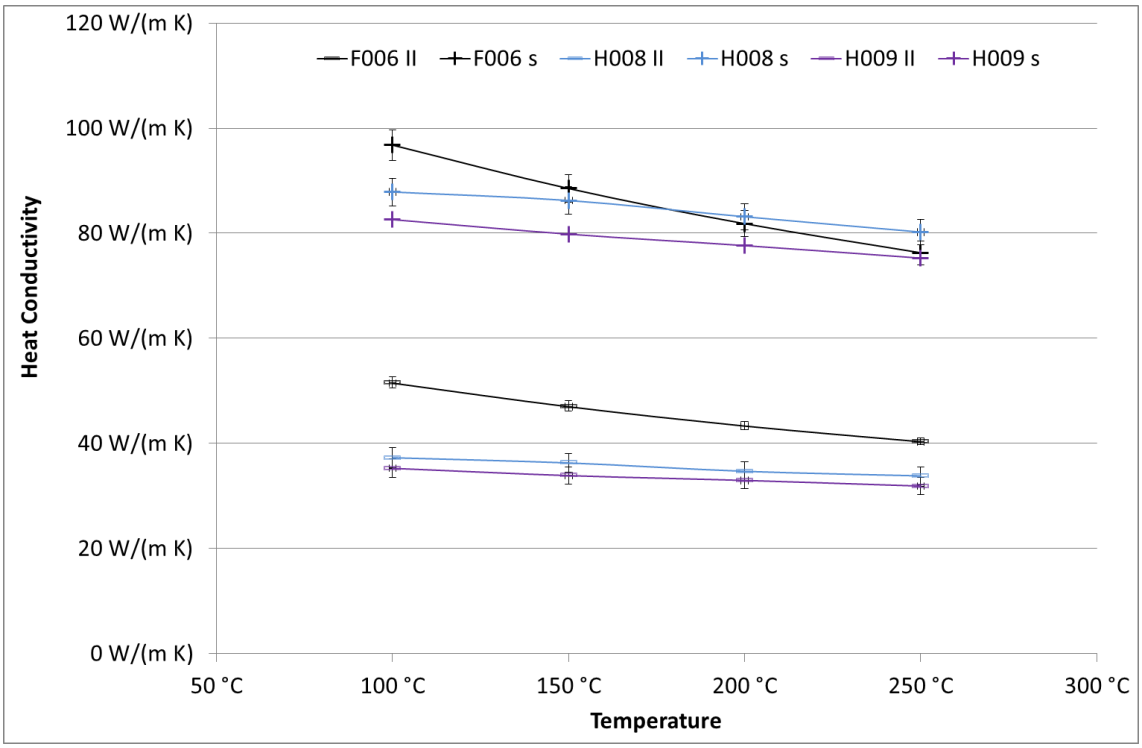


Figure 14: Heat conductivity of IGM

The difference between the F- and H-samples shows the influence of the used graphite. The F-sample has been made from natural graphite whereas the H-samples are out of synthetic graphite. A mixing of these materials would probably allow an adjustment of the thermal properties. Such investigations are proposed for end 2013.

Anyhow it shows that IGM has two orders of magnitudes higher heat conductivity than glass and concrete (0.5 to 3 W/(m K) (2). This will allow a higher load with heat developing waste without local overheating effects. Therefore the inclusion of activated material e.g. metal springs will be possible.

The thermal expansions have been measured for the samples F004 and F006. The material shows a significant difference perpendicular and parallel to the pressing direction. The perpendicular expansion is about a factor of 3 less in comparison to the expansion parallel to the pressing direction. A residual expansion of 1% perpendicular and 4% parallel remains after cooling down to room temperature. This can be explained by an annealing of internal forces due to compaction of the graphite plains at temperatures above 500°C. This is also indicated by the peak of the extension coefficient between 500°C and 600°C.

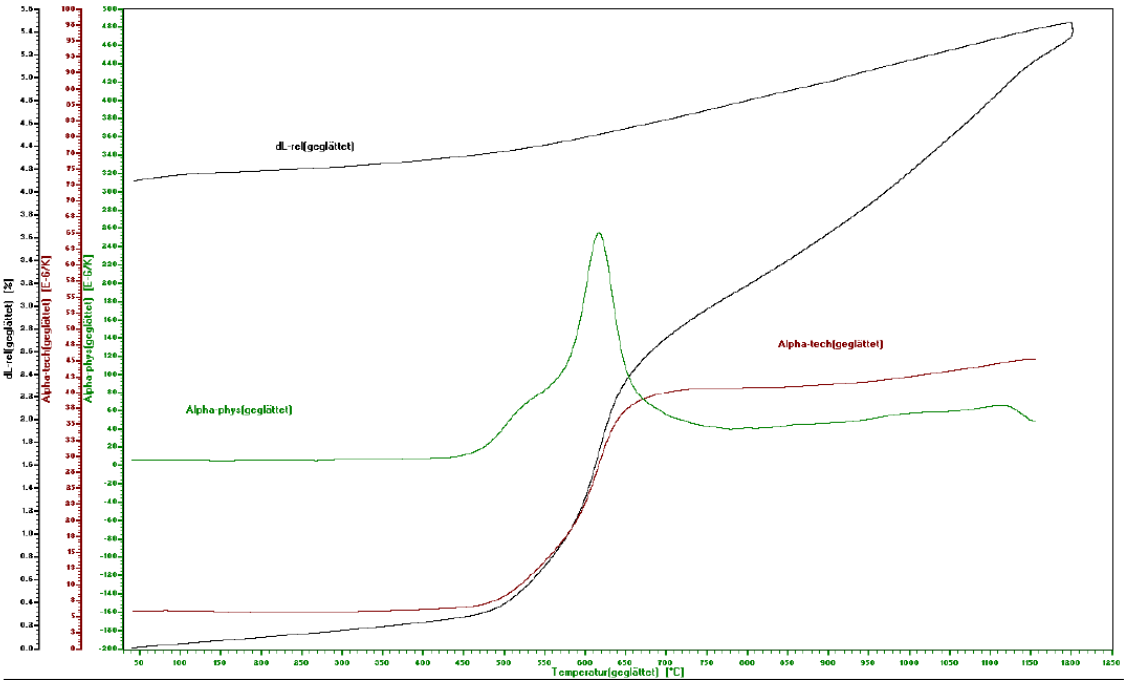


Figure 15: Thermal expansion of sample F004 (parallel)

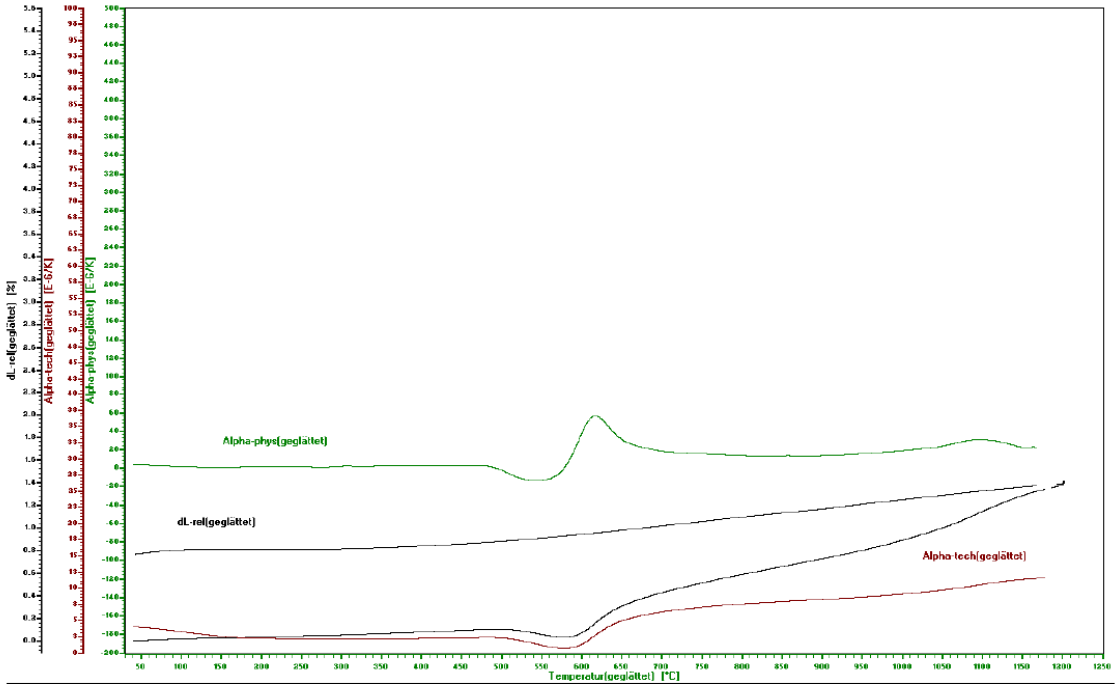


Figure 16: Thermal expansion of sample F004 (perpendicular)

3.4.2 Electrical conductivity

The electrical conductivity has been measured for 3 samples cut form F004 and F006 each. The same expected anisotropy has been observed as for the thermal properties. The specific resistance of the samples F004 perpendicular (F004+) is similar to the specific resistance of A3-3 (~ 17 Ohm mm²/m) whereas A3-3 is more or less isotropic due to its manufacturing process.

Sample	Spec. Resistance
F004 +	16,6 Ohm mm²/m
F004 +	17,4 Ohm mm²/m
F004 +	19,1 Ohm mm²/m
F006 II	32,0 Ohm mm²/m
F006 II	30,8 Ohm mm²/m
F006 II	29,9 Ohm mm²/m

3.5 Structural properties

3.5.1 Open Porosity

The open porosity has been investigated by mercury porosity measurement. Figure 17 shows the pore volume distribution of IGM in comparison to an A3-3 graphite. The red line represents the detection limit of the measurement as a straight line parallel to the x-axis. This does not represent the increase of the detection limit for small pores and therefore it is a conservative assumption. The main open pore volume of the A3-3 material is related to a pore size of about 2 µm down to 0.8 µm. No open pores

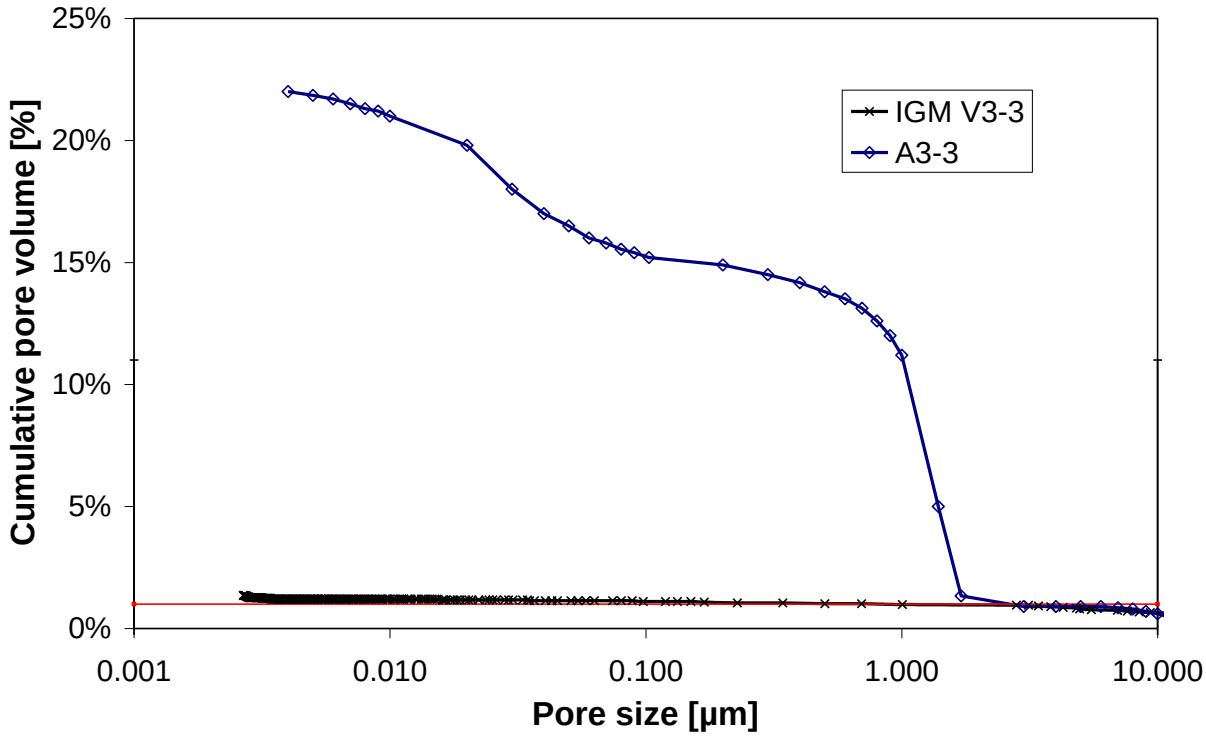


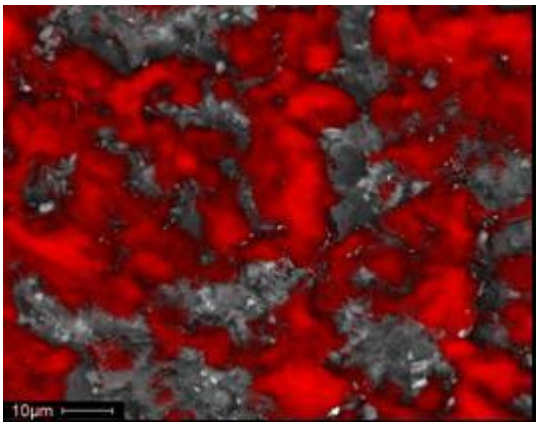
Figure 17: Mercury porosity of IGM and A3-3 graphite

have been observed for IGM in this pore size range. Water ingression experiments showed that open pores of this size can be accessed by water whereas smaller pores will not be penetrated. May be this will be shifted towards smaller pore sizes under high pressure but a penetration of micro-pores seems to be not probable. In case of IGM a small pore volume of pores < 0.2 µm cannot be excluded by this measurement. This is probably related to the conservative assumption of the detection limit. But anyhow it is unlikely that pores of this small size will contribute to a water penetration of the IGM.

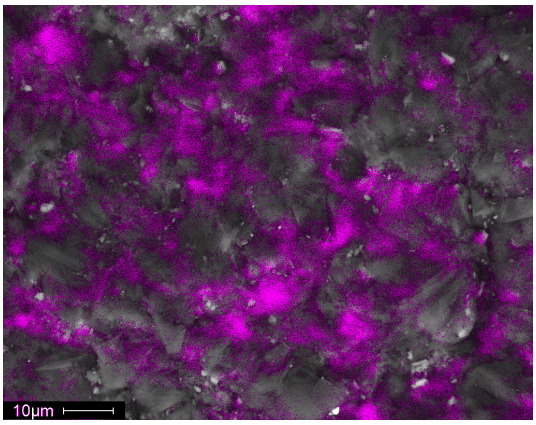
3.5.2 REM images of IGM with EDX maps

Raster electron microscopy (REM) investigations and energy dispersive x-ray examinations (EDX) has been performed. Figure 18 A and B shows the graphite in red and the glass in purple. The overlaying of these two figures (Figure 18 C) shows that the glass is percolated between graphite particles and confirmed the pore free structure already obtained by the mercury porosity measurement.

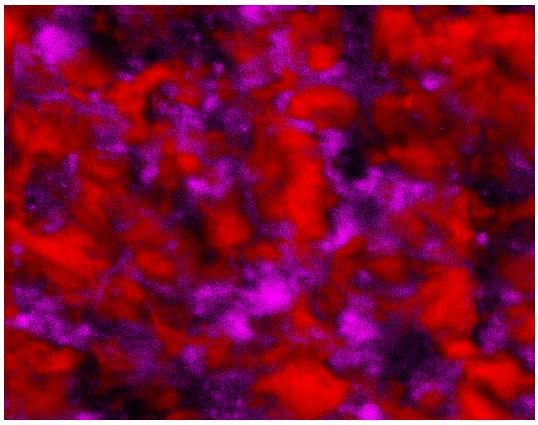
Another interesting observation was the percolation of glass in fractures of a larger graphite particle (Figure 19). This proves that glass with viscosities in the range of 10^4 dPas can penetrate small cracks in graphite particles and close them.



A



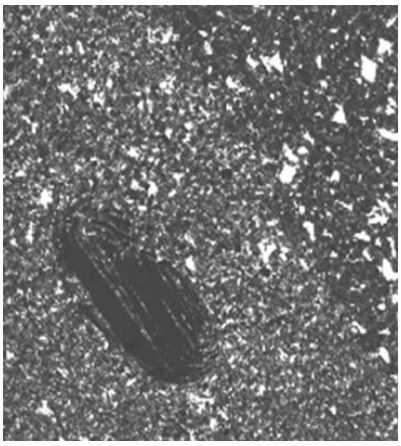
B

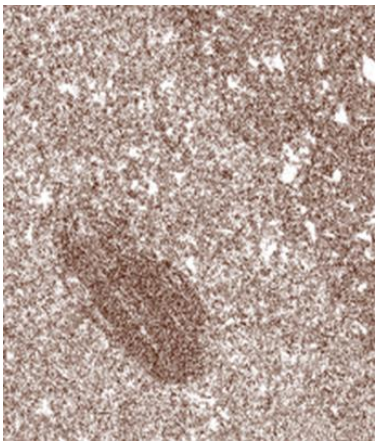


C

Figure 18: and Silicon distribution in an IGM sample

A: Red Carbon
B: Purple Silicon
C: Overlay





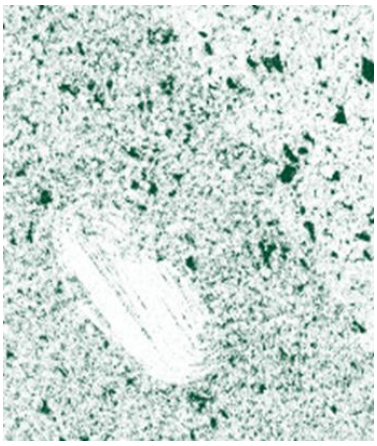


Figure 19: REM and EDX of larger graphite particle

a) REM image b) EDX carbon map c) EDX silicon map

4 Corrosion behaviour

4.1 Description of experiments

Graphite is a stable material under disposal conditions and therefore no leaching of graphite is expected in aqueous phases. However the glass may interact with aqueous phases and will be dissolved.

Therefore a leaching resistant glass is required for the IGM in order to obtain a long term stable material. Therefore leaching experiments of inactive IGM samples will be performed with 15 different glass types used as inorganic binder material. Samples of 10x10x50 mm have been cut form the pressed IGM blocks and immersed into deionised water and magnesium chloride rich salt brine at 90°C. Experiments with alkaline solutions are planned in 2013.

Table 3: Identification table for leaching samples

Sample	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	P
Mixture	V35	V42	V36	V22	V24	V24	V25	V26	V27	V28	V29	V30	V31	V32	V33	V34

4.2 Development of sample weight

The glass corrosion behaviour of IGM was compared for the glasses 8330 and 8250 for Schott in deionised water and magnesium chloride brine (pH ~ 4). Figure 20 show the results for the glass 8250. It can be clear seen, that the corrosion in deionised water is much faster than in the salt brine. Similar results have been obtained for the glass 8330 but the differences were smaller because the glass 8830 has a better corrosion resistivity. Therefore further experiments were performed with deionised water.

Figure 21 shows the weight change of the IGM samples in deionised water. It can be assumed that the weight change mainly depends on the interaction of the aqueous phase with the glass material. The initial weight increase can be explained by reactions of the glass with water. This could ad- and absorption of water at the glass surface followed by formation of hydrolysed species. The on-going hydrolysis reaction leads to a dissolution of glass components which explains the weight loss.

Figure 22 shows the weight change per square meter and day. The first 20 days have been skipped from the diagram because the huge mass changes are not representative for the long term behaviour of the material. It is assumed that the weight change is only related to the glass interaction with water. The obtained results show that all weight changes decrease in time. The lowest weight change rate has been obtained for the glass 8800 with 0,008 g/m²d. This would be equal to the corrosion rate if only glass corrosion is assumed for weight change

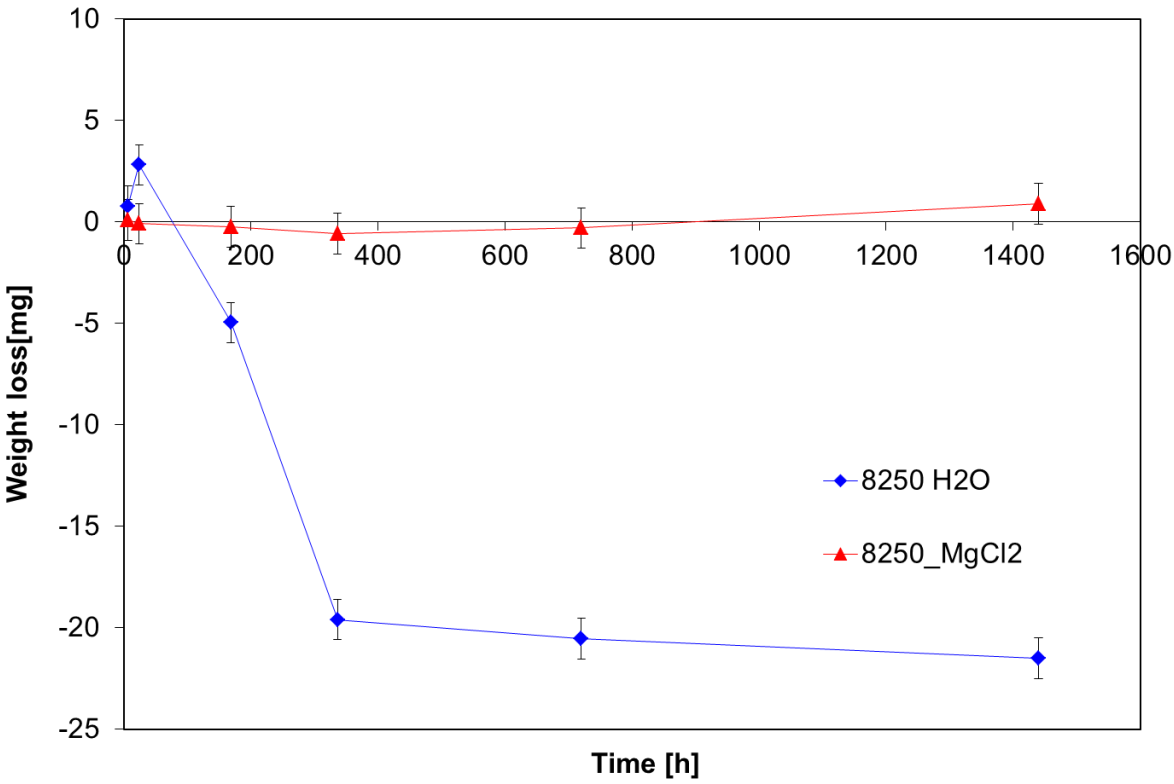


Figure 20: Comparison of weight change in deionised water and magnesium chloride rich brine.

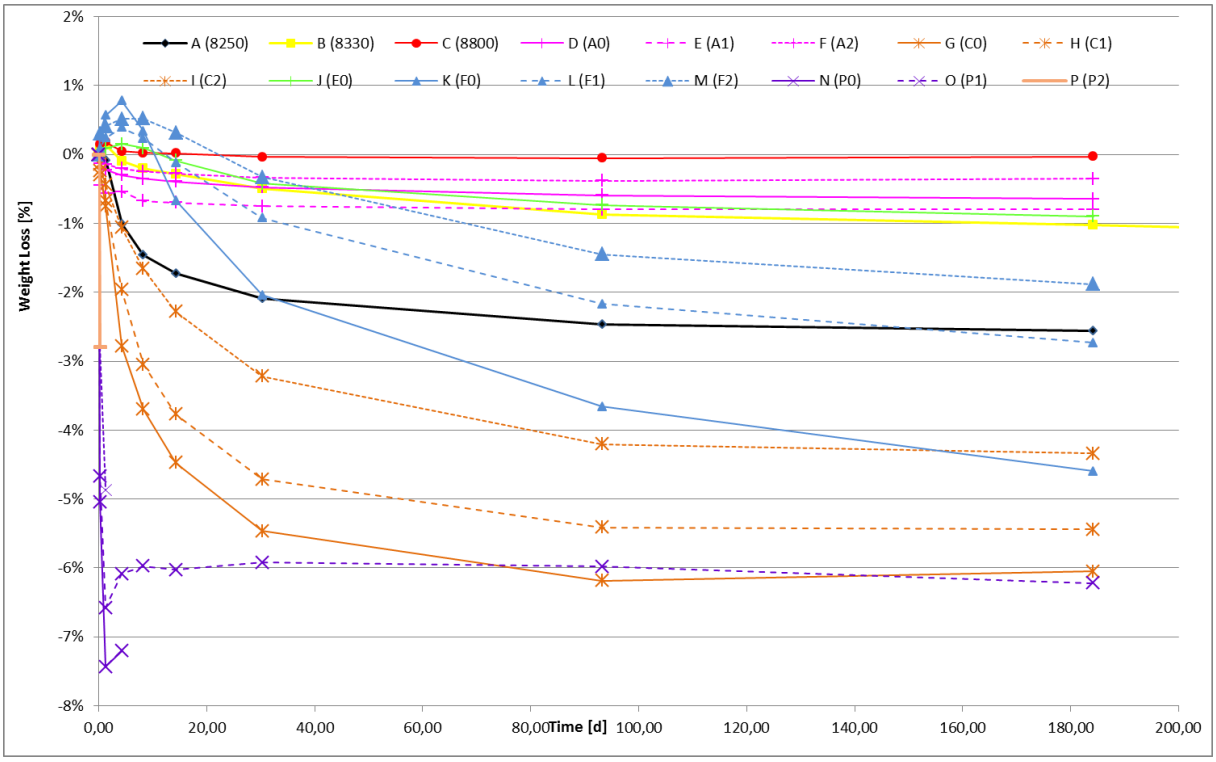


Figure 21: Weight loss of IGM by leaching in de-ionised water at 90°C

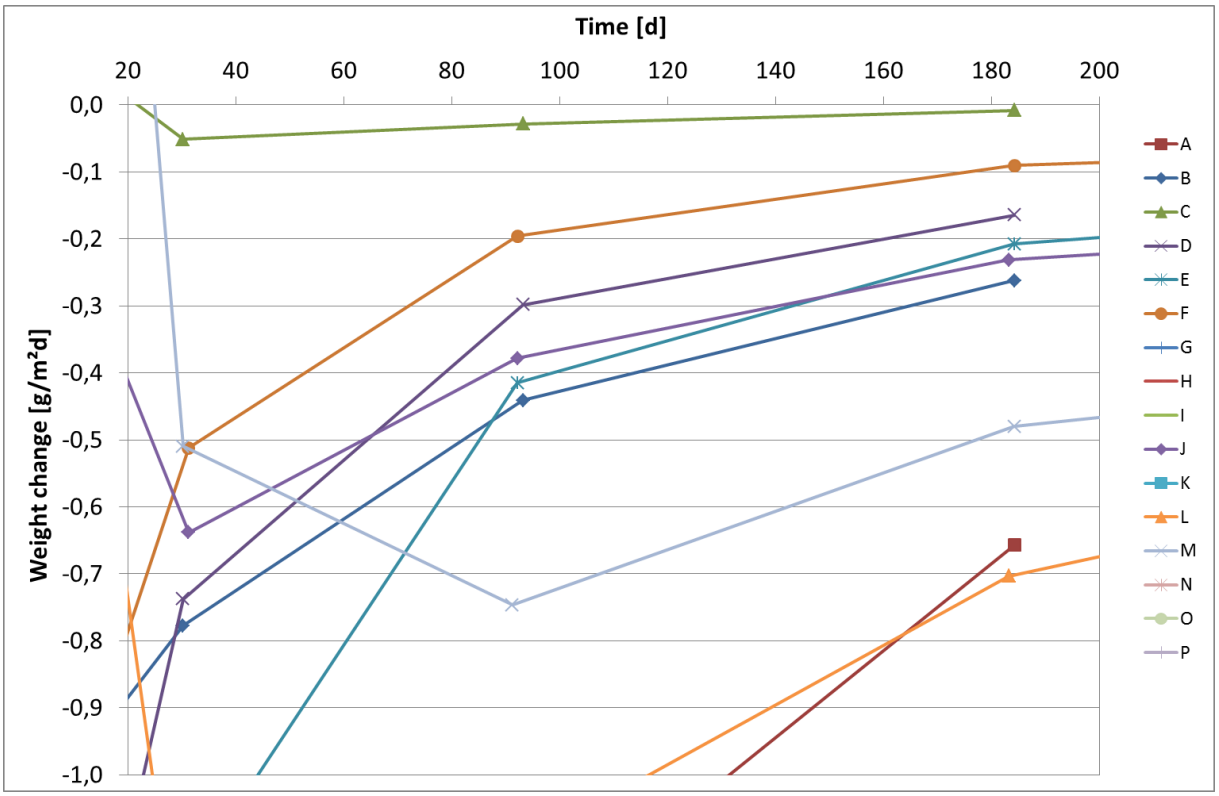


Figure 22: Weight change per square meter and day related to the start mass

4.3 Leaching of glass components

The IGM samples were not manufactured from nuclear grade graphite and contain sodium, boron and silicon as impurities. Only the potassium concentration in the graphite was in an acceptable range in comparison to the potassium concentration in the glass. Therefore potassium has been measured as indicator for the corrosion progress of the glass. Figure 23 shows the release of potassium from IGM prepared with the glass 8800. The corresponding corrosions rate for the glass is in the range of 0.002 g/m²d which is comparable with the assumed corrosion rate by weight change. Other glasses shower higher release rates corresponding to the related higher weight loss.

Based on this results, the weight change and the release of potassium, further investigations will be focused on the glass 8800.

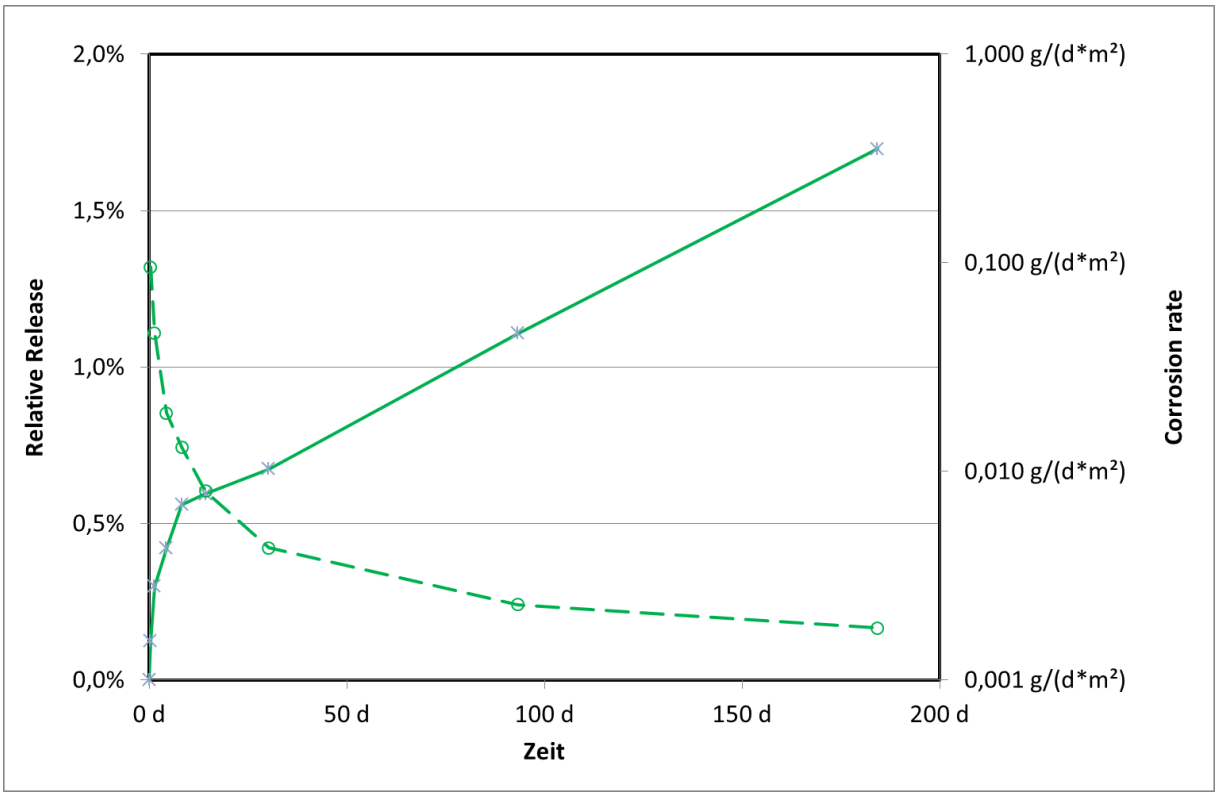


Figure 23: Leaching of potassium from IGM with glass 8800

5 Reference

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