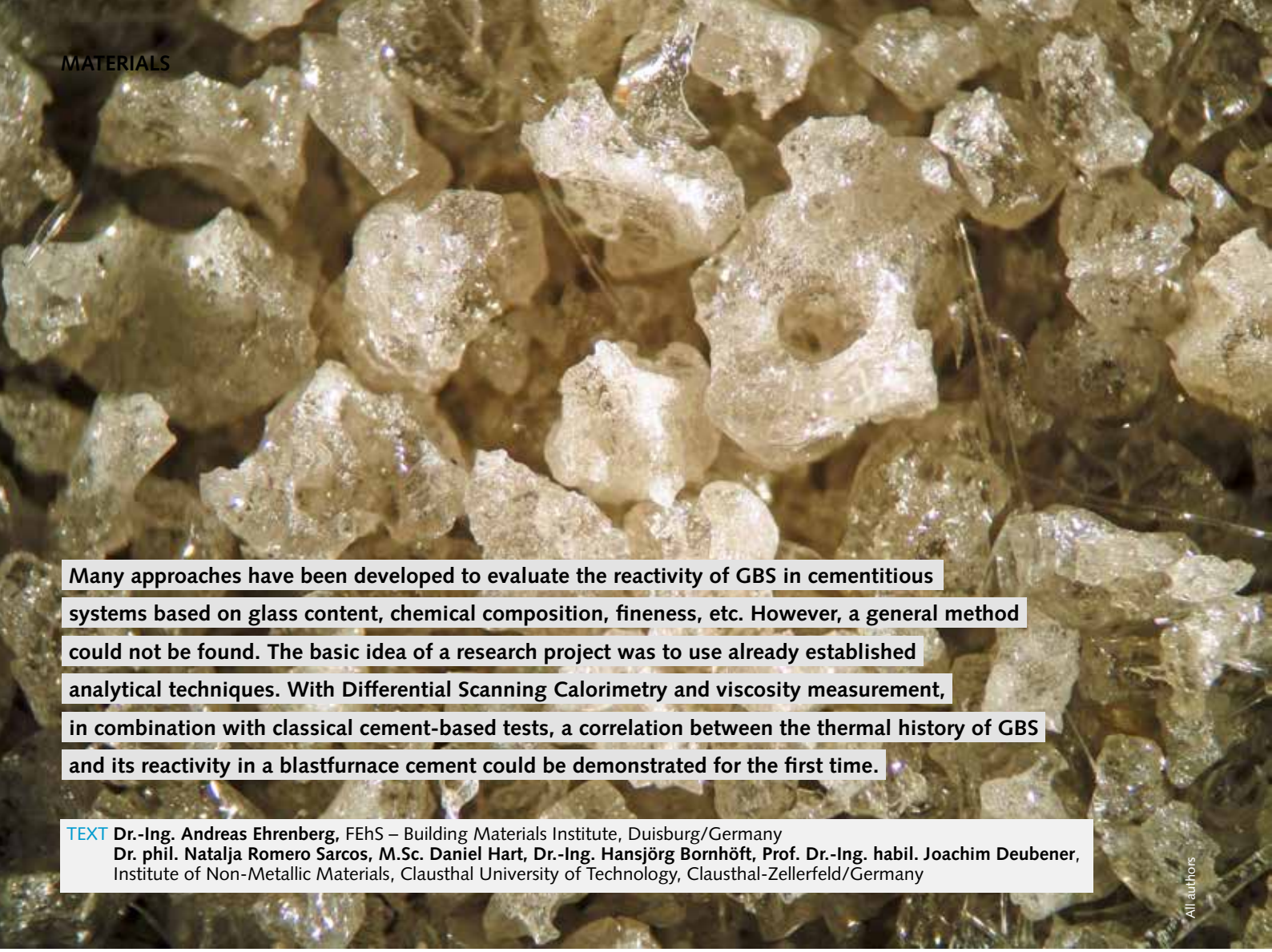

Many approaches have been developed to evaluate the reactivity of GBS in cementitious systems based on glass content, chemical composition, fineness, etc. However, a general method could not be found. The basic idea of a research project was to use already established analytical techniques. With Differential Scanning Calorimetry and viscosity measurement, in combination with classical cement-based tests, a correlation between the thermal history of GBS and its reactivity in a blastfurnace cement could be demonstrated for the first time.

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The thermal history of granulated blast furnace slag and its impact on reactivity

About 400 million t of blast furnace slag are produced annualy world-wide, together with about 1247 million t of hot metal (2018). About 300 million t are quenched forming the glassy granulated blast furnace slag (GBS). For more than 130 years the GBS has been used as a clinker substitute in cement and concrete due its latent hydraulic property. Many approaches were developed in order to evaluate GBS reactivity in cementitious systems based on glass content, chemical composition, fineness, etc. However, a general method could not be found. Only a rough differentiation might be possible. Compared to other parameters influencing GBS reactivity, the thermal history and the glass structure have mostly not been investigated so far. However, from thermodynamic and kinetic points of view it is obvious that the thermal history of the slag (= blast furnace and quenching processes) should have a significant influence on enthalpy content, glass structure and reactivity.

The basic idea of the research project was to use the analytical techniques already established for e.g. lime-soda-silica glasses. Differential Scanning Calorimetry and viscosity measurements have been combined for GBS characterisation in order to measure the fictive temperature T_f (glass transition temperature on cooling during the industrial quenching process) and to calculate retroactively the unknown cooling rates of liquid blast furnace slags. Using these methods in combination with classical cementitious tests it was possible to verify for the first time a correlation between the thermal history of GBS and its reactivity in a blast furnace cement.

1 Introduction

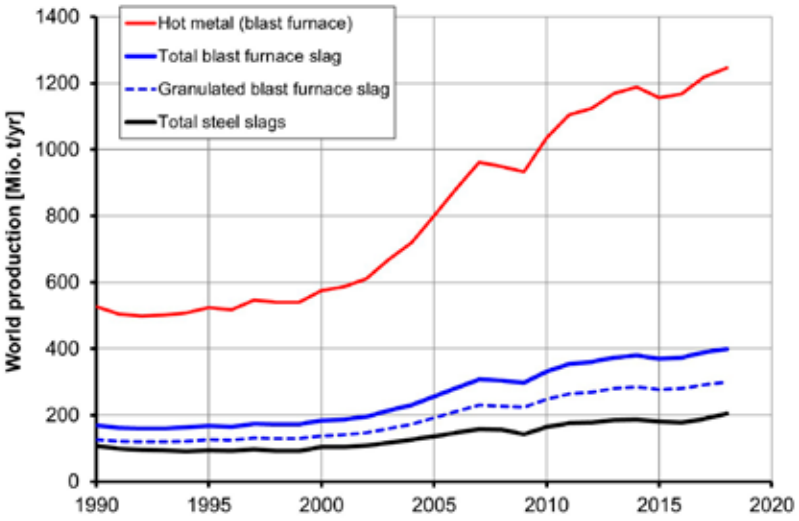
1.1 Current situation

In 2018 the world-wide hot metal production in blast furnaces was about 1247 million t [1]. Together with the hot metal also about 400 million t of blast furnace slag are generated annually. It can be assumed that about 300 million t are cooled rapidly resulting in glassy GBS with a latent hydraulic property (Figure 1). Thus, for more than 150 years GBS has been used as an important supplementary cementitious material due to its beneficial technical (concrete durability, workability, colour), economic (avoiding costs for Portland cement clinker) and ecological (use of a by-product saving natural resources and reducing CO_2 emissions) advantages. Other GBS applications, e.g. as concrete or road making aggregate, as liming agent or for brick production, are of less importance. GBS is ground separately or together with other cement constituents like Portland cement clinker, limestone and sulphates. In Europe Portland slag cements CEM II/S with a GBS content of 6–35 wt.-% and blast furnace cements CEM III/A with a GBS content of 36–65 wt.-% are the most important ones being defined in the standard EN 197-1 for common cement. In some countries also the use of ground GBS as a concrete addition (e.g. according to EN 15167-1) is well established.

GBS properties differ depending on individual raw materials input, blast furnace operation and granulation conditions [2]. Already the visual appearance differs a lot. Figure 2 shows three examples of the 16 different GBS being investigated in a research project of the FEhS Institute and Technical University of Clausthal [3].

The different colours result from the different chemical composition (see 2.1) whereas the different grain sizes result from the different viscosities of the liquid slags (see 2.3) in combination with different quenching ("granulation") conditions.

Since the late 19th century many approaches exist to evaluate the GBS reactivity in cementitious systems. Glass content, chemical composition, fineness, etc. are considered. But all approaches failed to define a suitable tool for judging an unknown GBS in a way that its strength contribution can be predicted [4]. Already in 1980 at the 7th International Congress on the Chemistry of Cement (ICCC) H.G. Smolczyk (FEhS Institute) stated: "It cannot be expected that the resulting strength development can be predeterminated by aid of a simple hydraulic factor" [5]. Only a rough differentiation might be possible, but at the end time-consuming cement tests have to be done [6]. An in-depth discussion of the problem was given at the 14th ICCC [7].

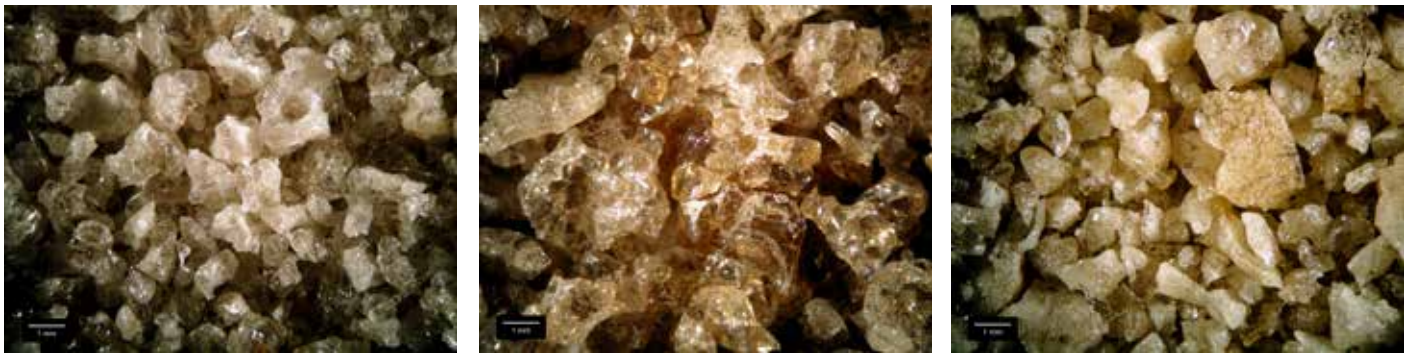


1 World-wide production of pig iron, blast furnace slag and steel slags

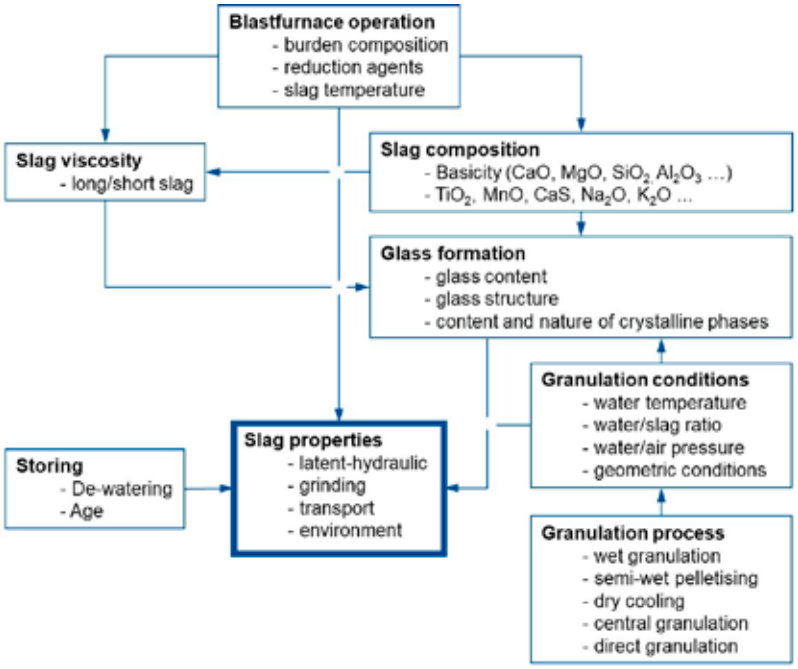
The reason for the failure to predict GBS reactivity only based on chemical composition and glass content is that several overlapping or unconsidered parameters exist which may have influence on the reactivity (Figure 3).

1.2 The new approach

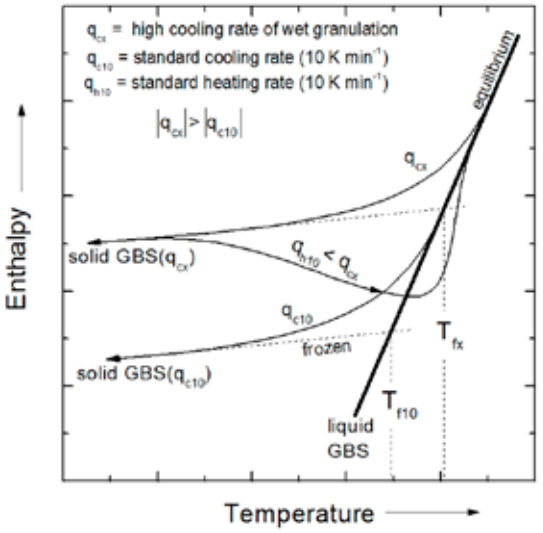
Compared to the different parameters mentioned above which were already thoroughly investigated, parameters such as thermal history have mostly not been considered so far. However, from thermodynamic and kinetic points of view it is obvious that the thermal history of the slag within the blast furnace process, in the slag runner and during the quenching process should also have a significant influence on the enthalpy content and the structure of GBS glass. Therefore, GBS glasses of the same chemistry and the same glass content may show a different resistance against corrosion at high pH values and therefore a different reactivity in



2 Several exemplary GBS, No. 3 (left), No. 13b (middle) and No. 14 (right)



3 Parameters with influence on the properties of granulated blast furnace slag [2]



4 Schematic dependence of glass enthalpy on cooling/heating conditions

a cementitious system. One of the questions to be answered in the research project was whether any correlation exists between thermal history of GBS and its technical properties – like heat of hydration or strength development of slag cements. However, it is not possible to measure e.g. the cooling speed in industrial granulation facilities. Therefore, it was obvious to use the established analytical methods for e.g. soda-lime-silica or other glasses in order to also be able to quantify the unknown thermal history of GBS.

A glassy material (GBS in this case) stores the information about prior cooling (so-called thermal history) in its structure and a common way to define it is based on the fictive temperature (T_f) determination. Figure 4 shows that on cooling of the slag melt from the equilibrium liquid state, the fictive temperature T_f is equal to the physical temperature ($T_f = T$) and the system is in equilibrium. With decreasing temperature, however, the molecular mobility decreases while the viscosity of the melt increases and at one point the time required for structural rearrangement (relaxation) becomes longer than the time that the melt spends at that exact temperature, the structure freezes in and the material deviates from the equilibrium and begins to form a solid glass ($T < T_f$). The fictive temperature depends on the cooling rate that the melt undergoes: a faster cooling leads to higher fictive temperature and higher enthalpy. Hence, the higher T_f, the stronger the system has departed from equilibrium. The glass transition temperature T_g, on the other hand, is a particular case of the fictive temperature T_f. It is determined for the de-

Table 1 Chemical composition and glass content of the original GBS

		GBS 3	GBS 13b	GBS 14
SiO_2	wt.-%	37.6	38.0	34.6
Al_2O_3		9.8	12.4	13.7
CaO		40.9	33.7	38.9
MgO		6.4	11.3	8.4
TiO_2		0.61	0.61	1.16
Na_2O equivalent		0.62	1.74	0.68
S^{2-}		0.58	0.61	0.79
SO_3		0.20	< 0.07	0.14
CO_2		0.14	0.05	0.11
H_2O		0.14	0.16	0.23
(CaO+MgO)/SiO_2	–	1.26	1.18	1.44
Glass	vol.-%	100	99	98

fined thermal history at a cooling rate of 10 K min^{-1} ($T_g = T_{f10}$ in Figure 4). Of course, T_g depends also strongly on the glass composition. Relaxation of the GBS glass is a spontaneous process that occurs constantly in nature. However, at room temperature relaxation takes billions of years so that annealing at temperature T_a below the glass transition temperature is necessary to decrease T_f towards its physical temperature in reasonable time scales.

For the investigations differential scanning calorimetry (DSC) and viscosity measurement were combined to measure the fictive temperature T_f and to calculate retroactively cooling rates. The enthalpy content of the GBS was modified by annealing tests below the glass transition temperature T_g. In addition, the characterisation of the cementitious properties was done (mortar strength, heat of hydration) in order to correlate GBS glass properties and GBS reactivity.

2 Experimental methods to characterize the granulated blast furnace slags

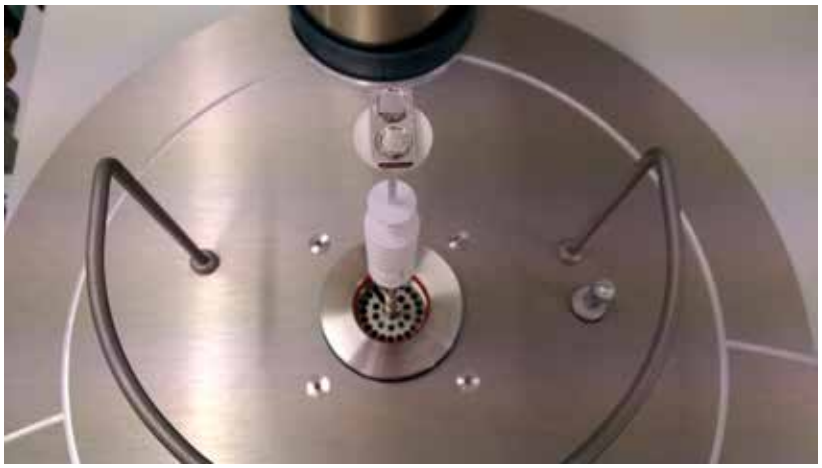
2.1 Chemical composition and glass content

For the tests described in this paper three industrial water-quenched GBS were investigated: GBS 3, GBS 13 b and GBS 14. Table 1 summarises their chemical composition and glass content. The chemical composition was measured by XRF (main and minor constituents), wet chemistry (S^{2-}, SO_3) and IR spectroscopy (CO_2, H_2O). The glass content was measured by optical transition light microscopy [8]. The chemical composition of the three slags covers the typical range for the GBS being produced in middle and western Europe. As measured for the three selected GBS in most cases the glass content of industrially-produced GBS is > 95 vol.-%.

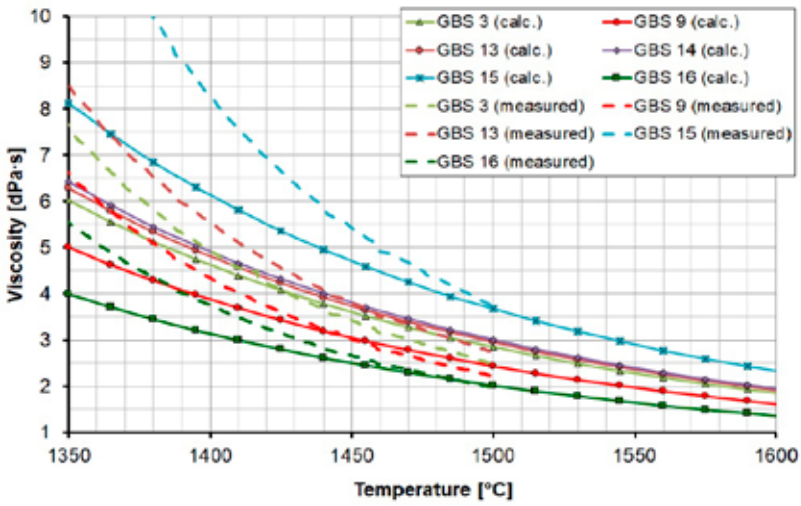
2.2 Differential Scanning Calorimetry

The selected industrial GBS were investigated as received (quenched) with original higher T_f and as annealed with lowered T_f. To anneal GBS, it was kept at 0.93 x T_g for 1.5, 24 and 96 h in air. This allows the relaxation of the glass and thus modifies the thermal history derived from quenching during granulation and lowers T_f. All samples were nearly totally glassy which is a mandatory requirement for the method being applied to determine T_f.

GBS were sieved to a fraction of 355–500 µm to minimize the grain size effect on the fictive temperature. The sieved samples were then analyzed calorimetrically and T_g and T_f were determined as described in detail in [9] with the only difference that the fictive temperature was calculated using a unified approach [10]. Figure 5 shows the small sample chamber for the Differential Scanning Calorimetry. The samples were subjected to the sequence of heating to 771 °C for the GBS 13 and 794 °C for the GBS 3 and 14, with subsequent cooling to 40 °C followed by reheating to 815 °C. The heating, cooling and reheating were performed with 10 K min^{-1}. The first upscan was carried out on the quenched sample (unknown thermal history derived from the slag granulation), whereas the second upscan was done on the sample with equilibrated thermal history created with standard cooling of 10 K min^{-1}. To compensate for possible inhomogeneities the samples were measured at least twice. Glass transition temperature T_g was evaluated graphically by determining the crossover temperature of two tangents aligned to the base and the decreasing flank of the endotherm of c_{p2}, respectively.



5 Small sample chamber for Differential Scanning Calorimetry

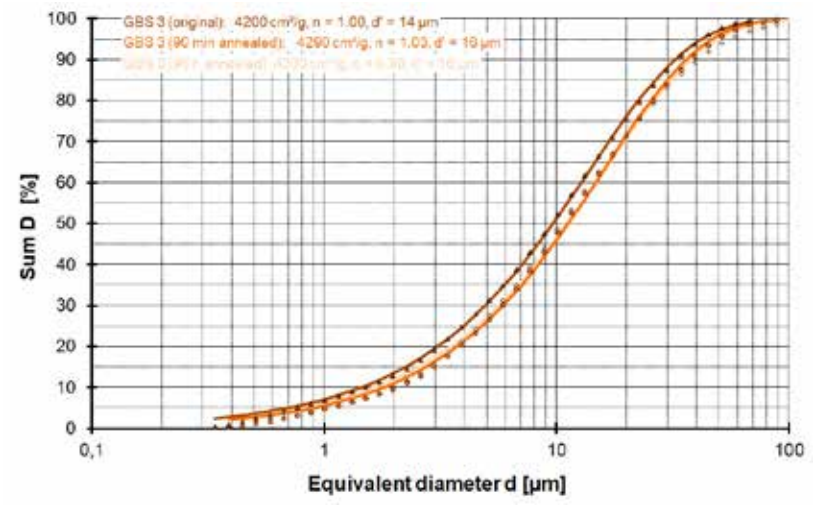


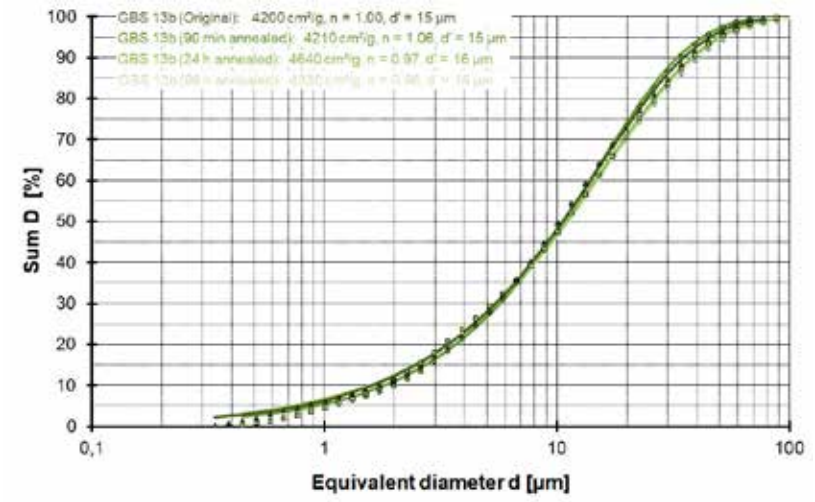
6 Measured and calculated dynamic viscosity of liquid blast furnace slags

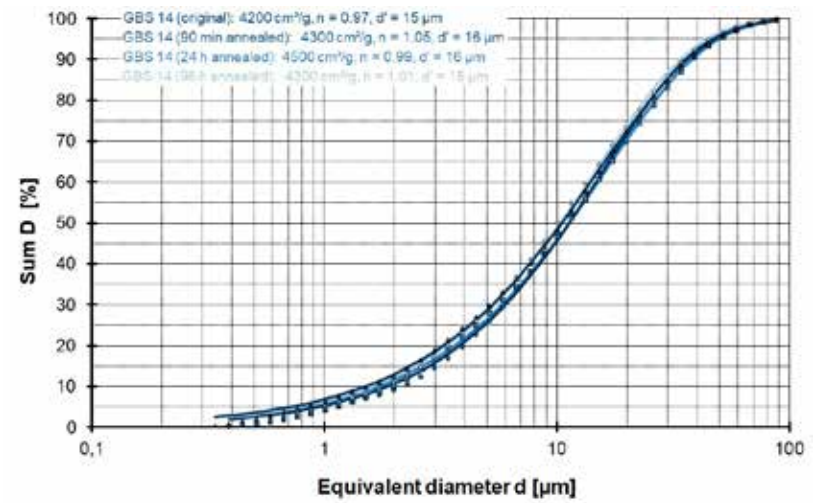
2.3 Viscometry

For calculation of the cooling rate q it is necessary to know the viscosity η of the slag at the fictive temperature T_f ($\log q_{cx} = K - \log \eta(T_f)$). However, it is not possible to measure the viscosity around T_f because of crystallization. Therefore, an appropriate interpolation model has to be used in combination with the measured viscosities around the glass transition temperature T_g and above the liquidus

7 Particle size distributions of ground GBS 3, 13b and 14 being used for heat of hydration and mortar strength tests







temperature T_l. It is possible to measure the latter with a rotating viscometer. In order to avoid the time-consuming test procedure years ago FEhS has modified the Urbain model [11] to calculate the viscosity above T_l. For this study some calculations were verified by measurements for which $\Delta\eta$ = 0.1 dPa·s is considered to be the error of measurement. The measurements were done by the Technical University of Freiberg. The results show for several slags a good matching between calculation and measurement for temperatures > T_l (Figure 6). With decreasing temperatures < T_l a very fast crystallisation occurs resulting in increasing differences between measured and calculated values. In general the viscosity of liquid blast furnace slags is much lower compared to other glasses, e.g. lime-soda-silica glasses.

The viscosity data in near T_g range was determined calorimetrically using cooling and heating rates of 10, 15, 20 and 30 K min^{-1} (q_h = q_c) and a shift factor of F = 11.35. For determination of the required viscosity at T_f the temperature dependence was interpolated using the MYEGA model as described in detail in [8].

2.4 Cementitious tests

To test the GBS reactivity, all samples were ground in a 10 kg laboratory ball mill. Figure 7 shows the particle size distributions of ground GBS 3, 13b and 14, measured by laser granulometry. All samples had a comparable fineness, as the RRSB parameters (DIN 66145) d' and n show.

Based on the database of the FEhS Institute blast furnace cements CEM III/B incorporating 75 wt.-% GBS and 25 wt.-% Portland cement clinker were mixed. A total SO_3 content of 4.5 wt.-% was adjusted. This cement type is not the most relevant one in the market, however, specific slag properties can be illustrated very well. The cementitious properties were tested according to EN 196-1 (mortar strength) and EN 196-11 (heat of hydration) at a water/cement ratio of 0.50.

3 Results and discussion

3.1 Differential Scanning Calorimetry

As an example Figure 8 (left) shows for GBS 14 the heat capacity curves as a function of the temperature obtained from DSC measurements of two upscans.

For the quenched GBS (Figure 8, left) sample the 1st upscan (red) reveals the broad exothermic effect that is attributed to the release of the potential energy enclosed in the GBS during the wet granulation process. The 2nd upscan (black) of the standard cooled GBS exhibits no exothermic effects (since the cooling rate equals the heating rate). In the case of the annealed GBS 14 (Figure 8, right) the 1st and the 2nd upscan match in the sub-T_g range (T_g equals 1007 K or 734° C), but a larger overshoot at T > T_g is

Table 2 Glass transition temperatures and cooling rates of original and annealed GBS

GBS	Status		T_g	T_f	log viscosity at T_f	Calculated cooling rate
			°C		Pa s	K s^{-1}
3	Original		469	826	7.22	12160
	annealed T_a = 668 °C	90 min		756	10.56	3
		24 h		728	12.38	$4x10^{-2}$
		96 h		716	13.26	$4x10^{-3}$
13b	Original		720	828	6.38	86439
	annealed T_a = 650° C	90 min		720	12.06	$2x10^{-1}$
		24 h		703	13.41	$8x10^{-3}$
		96 h		686	14.94	$2x10^{-4}$
14	Original		734	738	7.46	91300
	annealed T_a = 666° C	90 min		744	11.67	$6x10^{-1}$
		24 h		709	13.88	$5x10^{-4}$
		96 h		699	14.60	$4x10^{-5}$

evident, which indicates a somewhat lower T_f than that of the standard cooling. The determined T_g and T_f temperatures are summarized in Table 2.

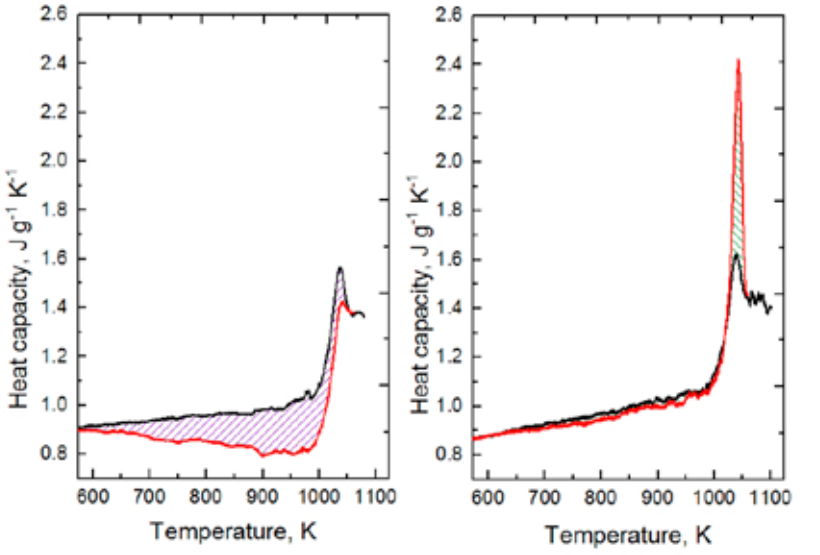
As can be observed from Table 2 the highest T_f was measured for the industrially quenched GBS. Further annealing was lowering the fictive temperature towards the annealing temperature T_a.

As Figure 9 demonstrates, the drop of T_f is strongest during the first 1.5 h of the annealing at 0.93 x T_g. Longer annealing time up to 96 h continues to lower the resulting T_f of the GBS and the time needed to lower the T_f by 1 K grows exponentially. It can also be suggested based on the obtained results for further investigation of the reactivity impact of composition and other properties that are not linked to T_f, to perform annealing to bring the glasses to the same structural state (anneal until T_f = T_g) which will eliminate the effect of thermal history and thus can make the comparison more precise.

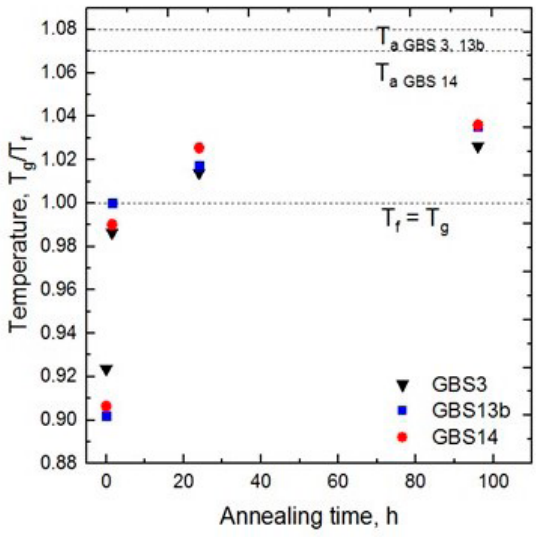
Figure 10 shows measured viscosity data for GBS 3 and the MYEGA model fitted through the data points. The viscosity values as well as estimated cooling rates are shown in Table 2. The cooling rates of the original GBS are very high. Moreover, while in case of quenched samples viscosities are obtained by interpolation leading to more or less precise data, the viscosity values for annealed samples are extrapolated and because of that are expected to have more errors. The cooling rates for the annealed samples are only theoretical values. If a real blast furnace slag were cooled down so slowly and if its glassy state were still guaranteed then it would have the same fictive temperature as the annealed GBS. In practice a blast furnace slag being not quenched is a crystalline material.

3.2 Annealing for cementitious tests

After the calorimetric tests in small scale (see 3.1)



8 Heat capacity curves of GBS 14 after wet granulation (left) and after annealing at 0.93 x T_g for 24 h (right). The red curve represents the heat capacity of the first upscan (c_{p1}), while the black line represents the heat capacity of the second upscan (c_{p2}) after standard cooling at 10 K min^{-1}



9 Dependence of the fictive temperature T_f on the annealing time

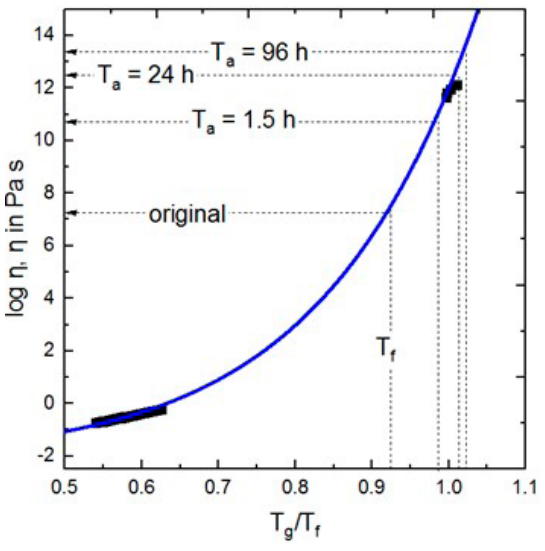
Table 3 Properties of original and annealed GBS

GBS	Status		CO_2	H_2O	Glass	True density	Vickers
			wt.-%		vol.-%	g/cm³	$HV_{0.1}$
3	Original		0.14	0.14	100	2.913	-
	annealed T_a = 668 °C	90 min	0.11	0.05	100	2.957	-
		24 h	-	-	-	-	-
		96 h	0.11	0.12	100	2.969	-
13b	Original		0.05	0.16	99	2.895	606
	annealed T_a = 650 °C	90 min	0.13	0.06	100	2.932	-
		24 h	0.09	0.18	95	2.943	648
		96 h	0.11	0.08	99	2.946	-
14	Original		0.11	0.23	98	2.910	599
	annealed T_a = 666 °C	90 min	0.14	0.09	99	2.952	-
		24 h	0.12	0.22	99	2.958	633
		96 h	0.13	0.11	99	2.949	-

a bigger volume of GBS 3, GBS 13b and GBS 14 have been annealed at 0.93 x T_g in a muffle furnace. After 24 hours at 650 °C/923 K (GBS 13b) and 666 °C/939 K (GBS 3 and 14), respectively, the slags cooled down for about 48 hours. DSC analyses confirmed that the thermal history of the annealed GBS was modified, as was intended.

In Table 3 data regarding different chemical and physical properties of the original and annealed slags GBS 3, 13b and 14 are summarised. The data show that there is no change in chemical composition, neither in chemically bound CO_2 and H_2O, as is typical for GBS being stored for a longer time in humid atmosphere, nor in sulphate, as it might occur if sulphide is transformed to sulphate. Also the glass content is nearly unchanged, if the repeatability of the analytical method (transition light microscopy, Figure 12), is considered. The increase in true density (measured by He pycnometry) with annealing indicates that the structure of the annealed slag glass is denser. Also the Vickers hardness increased. Both correspond as well with the thermodynamic expectation (lower enthalpy = lower volume = denser structure = higher density) as with experiences for other glass systems.

XRD and microscopical tests confirm that the annealing procedure < T_g did not create crystallisation, as Figure 11 exemplary shows for GBS 13b. Only after 96 h first traces of gehlenite may appear. Figure 12 illustrates the glassy appearance as well of the original as of the annealed slags. The sporadic coloured zones in some grains indicate few crystalline parts. From the macroscopic point of view there is no change in the appearance of the original and annealed samples, as Figure 13 exemplary shows for GBS 13b. All samples show the typical glassy brightness. However, annealed GBS is darker.



10 Temperature dependence of viscosity of GBS 3. Line is best fit through the data using MYEGA viscosity model [9]

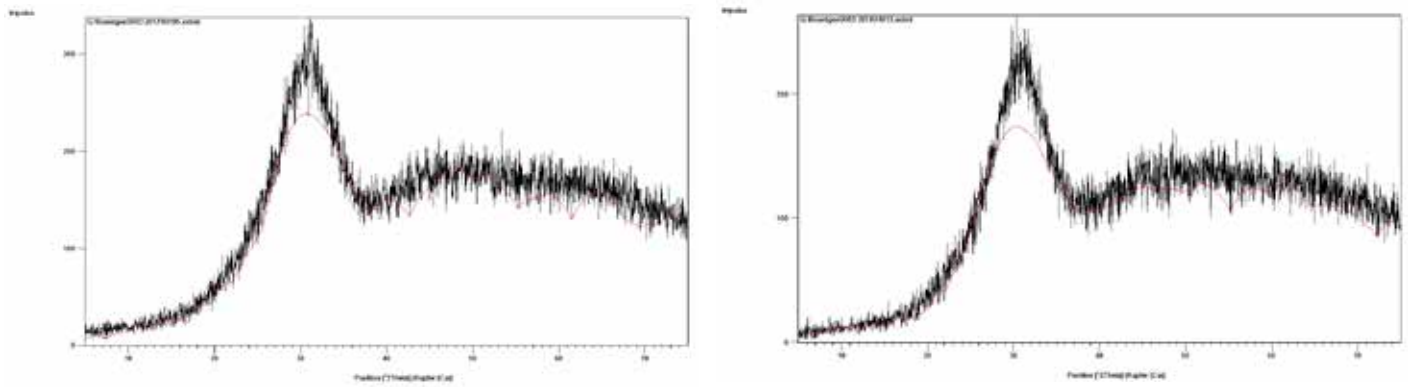
3.3 Cementitious properties

The mortar compressive strength between 2 and 28 days is shown in Figure 14. The black lines indicate the typical small standard deviation for each strength test. It is obvious that all cements with annealed GBS result in a significant lower compressive strength at all hydration ages being tested. For GBS 13b the effect after 2 days is very limited due to the general lower reactivity resulting from the lower basicity (see Table 1). But for GBS 14 which also has a higher basicity as well as a higher alumina content after 2 days the negative impact of the annealing procedure is considerable.

Figure 15 shows that the influence of the fictive temperature on the compressive strength is not linear within the investigated T_f range. The dependence is positive: higher fictive T_f results in higher compressive strength.

The results of heat of hydration measurements are shown in Figure 16.

In all cases the 2nd peak of the specific heat flow being typical for the GBS reaction is significantly lower for cements with annealed GBS whereas the 1st peak after about 12 hours resulting from the clinker reaction is nearly unchanged. The effect is



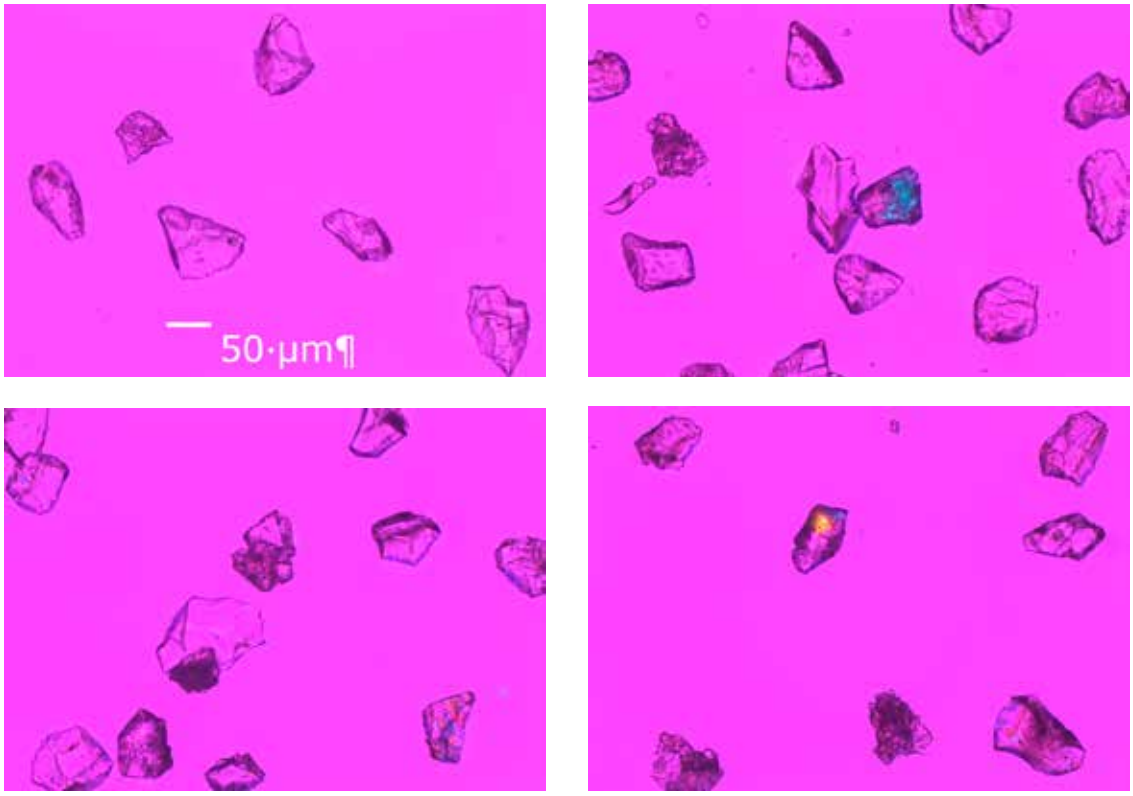
11 XRD of GBS 13b before (left) and after (right) 24 h annealing procedure

more distinctive if the annealing time was longer and therefore the enthalpy content was lower. It was shown that 90 min annealing time was already sufficient for a significant loss in reactivity. However, the total hydration heat after 7 days varied only in a limited scale. Moreover, the figures illustrate very well the general difference in reactivity between the three slags. E.g. for GBS 13b the heat flow level is generally very low and the 2nd peak arises at a later hydration time.

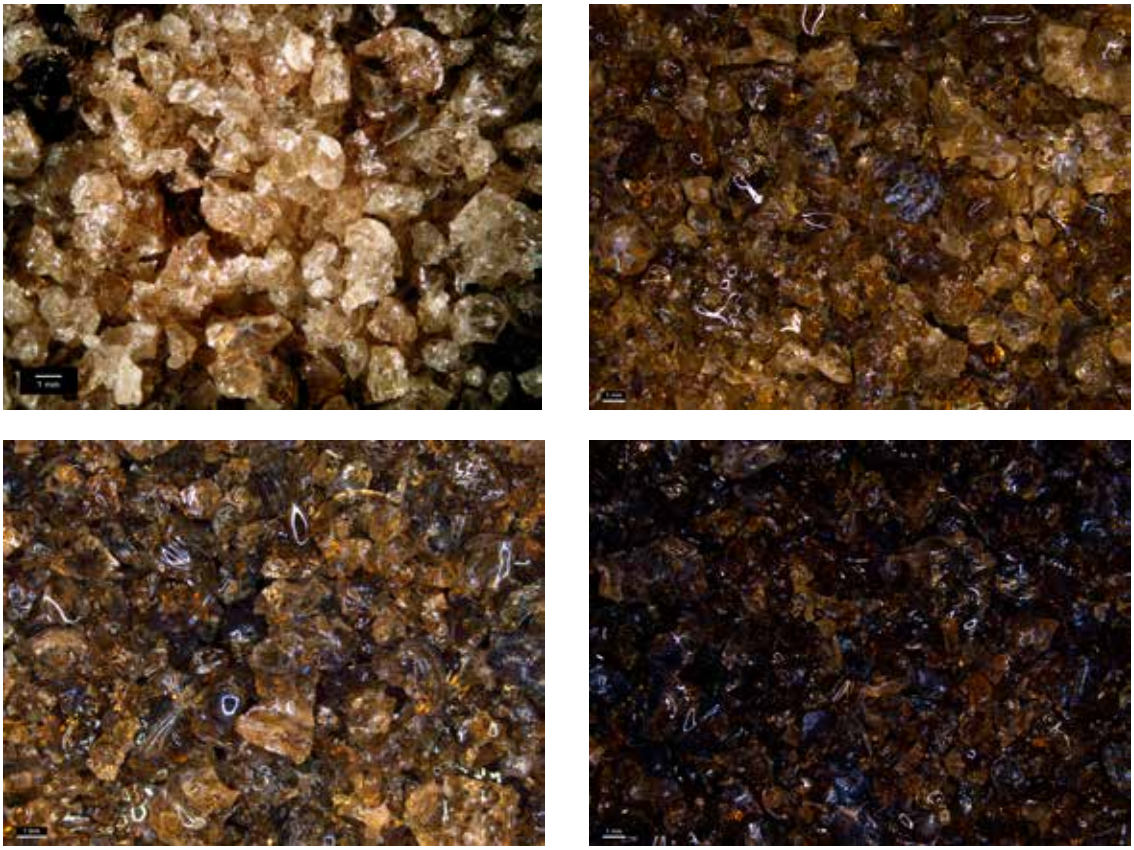
4 Conclusion and outlook

For the first time it was possible to verify that the thermal history of a granulated blast furnace slag resulting as well from the blast furnace process as the following quenching process of the liquid slag has a significant impact on the technical properties of cements containing slag. As can be expected from basic thermodynamic consideration an annealed GBS with a lower enthalpy (= lower fictive temperature T_f) has a lower reactivity in the sense of heat of hydration or strength development compared to an industrial GBS.

This relationship is of great importance. It explains very well why the attempts to explain the latent hydraulic property of GBS and to predict the strength development of slag cements only based on chemistry and glass content fail. Now it has to be investigated whether a standardised annealing procedure can be used to equalize each thermal history



12 Fraction 40-63 µm of crushed GBS 13b (above) and GBS 14 (below) before (left) and after (right) 24 h annealing procedure (transition light microscope)

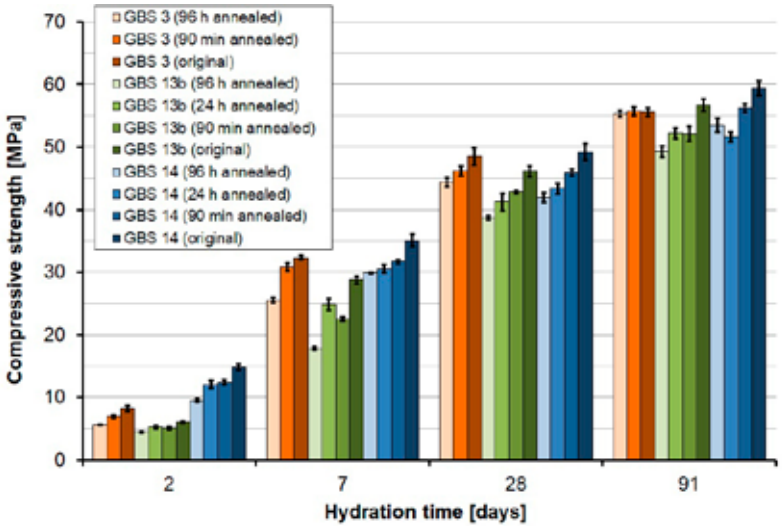


13 GBS 13b before (above, left), after 90 min (above, right), after 24 h (below, left) and after 96 h (below, right) annealing procedure (reflected-light microscope)

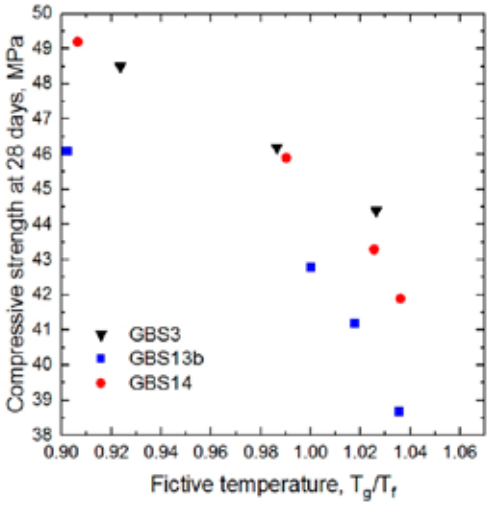
of a GBS in order to allow achievement of a better correlation between chemical composition and reactivity while all other frame conditions (glass content, fineness, mixture etc.) are kept constant. The relationship also allows the conclusion that a higher cooling rate during the wet or dry granulation process or a higher melting temperature of a liquid blast furnace slag should result in a higher GBS reactivity. It has still to be verified whether smaller GBS particles have a higher reactivity compared to larger particles due to their faster cooling.

The results also explain very well why the reactivity of dry granulated blast furnace slag was found to be lower compared to the same slag being quenched with water [12]. The cooling with enormous airflows is slower compared to the classical water granulation process also if it results in a glassy slag.

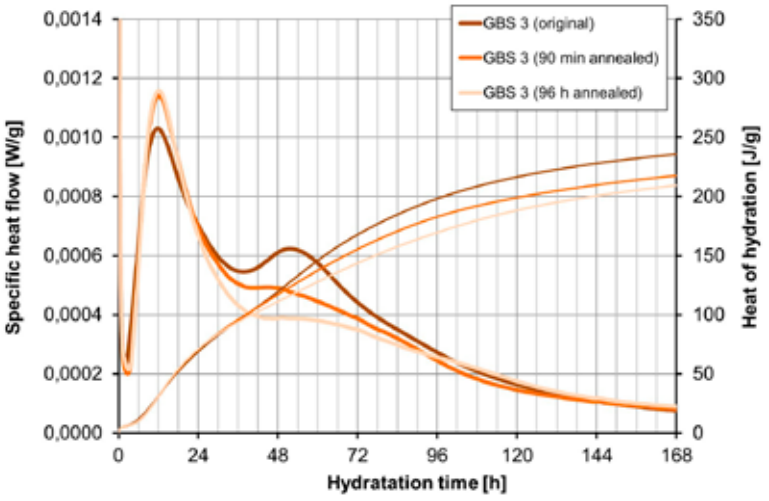
In addition, the findings are also important for the current scientific work on the possible use of other slags, e.g. BOF or Electric Arc Furnace slags, as cementitious materials. In the original state

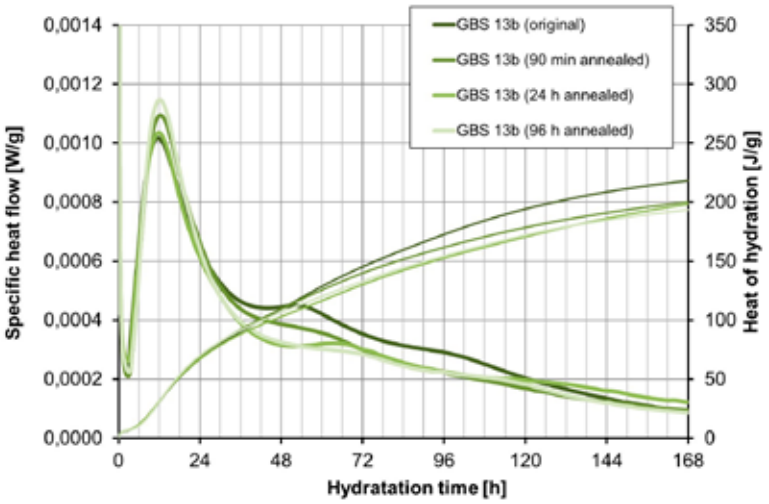


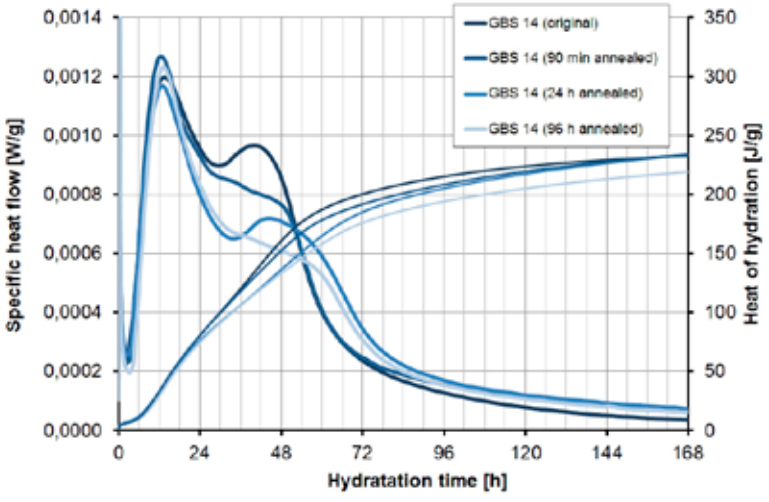
14 Slag cement mortar strength for original and annealed GBS



15 Dependence of the slag cement mortar strength on the fictive temperature of GBS







16 **Heat of hydration for original and annealed GBS 3, 13b and 14**

these slags do not show a (latent) hydraulic behaviour. However, after a chemical and/or thermal treatment and a fast cooling process also steel slags can be achieved as more or less glassy materials. Against the background of the world-wide discussion on an intensified use of supplementary cementitious materials instead of clinker in order to reduce the CO_2 footprint of cement production, such modified steel slags might be an additional contribution for a more sustainable cement and concrete production.

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