Experimental and Theoretical Comparison of a Systematic Variety of Thermite Compositions

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Abstract

Thermites enable the release of large amounts of heat accompanied with low gas production. This offers a high potential to divers pyrotechnic applications like incendiaries, flares, igniter or delay compositions. Thermite type reactions are characterized by a redox reaction of a metal with a metallic oxide. Nearly all solid metals or even half-metals may be used for this type of reaction. So a comprehensive thermodynamic Ellingham-analysis is performed calculating the heat of reaction of more than 10000 possible thermite type reactions. From this, micro-sized particles of Al, Mg, B, Si, Ti and Zr in a matrix-combination with 18 promising oxides were selected for experimental investigation in order to compare reaction velocity (characterized as progression rate) and reaction temperature in nitrogen and air. The latter one aims in additional performance using post-combustion of the produced metal with atmospheric oxygen. Depending on the combined substances progression rate varied from 2 mm/s to 20 m/s. Measured temperatures in nitrogen ranked from 1200 to 3000 K, which were significantly low as the combustion temperatures in air ranking from 1550 to 4500 K.

Introduction

The thermite reaction is known for a long time and was first patented in 1895 by H. Goldschmidt. Originally it was a new method to obtain pure metals from the metal ores without the use of carbon [1][2].

This most famous thermite type reaction is also called aluminothermic reaction and works with aluminum and iron (III) oxide. Still the term THERMIT™ is a protected trademark of Goldschmidt Thermit Group [3] but the name is used more commonly today. Thus thermites are powdered mixtures of a metal and an oxide of another nobler metal, reacting after ignition in a very violent reaction accompanied by low gas production and high heat-release to form the metal previously bound in the metal oxide. Here the term metal also includes semi-metals such as boron and silicon [2].

Generally thermite reactions can be described as exothermic chemical reactions where the metal (M^1) is oxidized by a metal-oxide ($M^2_{x2}O_{x3}$) to form the more stable metal-oxide ($M^1_{x4}O_{x5}$) and resulting metal (M^2) or alloy. The overall reaction equation referring to 1 mol of metal fuel is given as [4][5]:

$$M^1 + \frac{x_5}{x_3 x_4} M^2_{x2} O_{x3} \rightarrow \frac{1}{x_4} M^1_{x4} O_{x5} + \frac{x_2 x_5}{x_3 x_4} M^2 \quad (1)$$

Possible combination of reactants for M^1 and M^2-oxide can be selected on the basis of Gibbs-energy or chemical potential retrieved from the Ellingham diagram showing the oxidation of the elements with elemental oxygen as proposed by Ellingham **Fehler! Verweisquelle konnte nicht gefunden werden.**].

In addition to aluminum, boron, magnesium and titanium are often used as fuel metals M^1. Typical oxides are barium (IV) oxide, bismuth (III) oxide, copper (II) oxide, chromium (III) oxide, iron (III) oxide, manganese (IV) oxide and tungsten (VI) oxide [7].

Actually, this type of reaction is of great interest and found many applications, because most mixtures are insensitive concerning shock and friction and rapidly produce great amounts of heat on high temperature level [4][8]. So the thermite welding process is still the most frequently used method for welding railroad tracks [9][10] or another welding process is using copper thermite to create electric joints. Thermite reactions are also used to purify ores of some metals (e.g. uranium). Recently, the preparation of ceramics becomes more important [11][12]. Due to the large heat release and its self-sustaining nature, thermites are used in warheads as incendiary [13] for destructive devices [14].

Caused by the availability of metallic and oxidic nanoparticles, thermites recently get in the focus of interest and research. Subsequently, thermite compositions with microparticles gain in importance for many applications [15].

Another aspect is the potential post-combustion with atmospheric oxygen of the metal formed in the previous thermite reaction. In this exothermic process depending on the type of metal a similar amount of heat can be released which can also be used.

Depending on their components thermites show different combustion properties. Some mainly react in the gas phase, others in the melting. Others in turn tend to strong particle expulsion. Some are extremely fast and hot, while others react slower and much colder. Additionally, some thermite reactions show distinct post-combustion with oxygen. So every single thermite reaction shows a specific behavior, which determines its possible application. The reported study aims in a comprehensive screening of a large number of possible thermite reactions performing a theoretical thermodynamic analysis. Then a broad matrix of different metal/oxide pairings with promising energy balance was selected for experimental investigations to characterize reactivity (progression rate) and heat release (temperature). Another focus lies in the possible post-combustion with atmospheric oxygen, which was studied by comparative measurements in inert gas (nitrogen).

Thermodynamic Considerations

Fischer and Grubelich [8] have calculated the thermo-chemical properties of thermite combustion for about 200 metal-oxidizer reactions. Their selection of reactants covers most prominent substances but is far away from completeness. The aim was to study a more comprehensive number of solid element/oxide combinations (more than 10000) on feasibility and effectiveness and to find out the maximum possible heat release. Therefore, a small computer code fed with the thermodynamic data of about 250 oxides taken from the database of Binnewies [16] was used to estimate the possibility of reaction of nearly most elements of the periodic system in combination with most thinkable stable oxides based on the idea of Ellingham [6]. He proposed to calculate Gibbs energy from enthalpy and entropy

for the oxidation of an element with ½O_2. The results of such calculation can be used to create a ranking for the oxidation of each element with regard to Gibbs energy. Combining two oxidation reactions of metals M^1 and M^2 (Eq. (2) and Eq. (3)) results in a thermite reaction Eq. (1).

$$\frac{x_4}{x_5} M^1 + \frac{1}{2} O_2 \rightarrow \frac{1}{x_5} M^1_{x4} O_{x5} + \Delta_R H^1 \tag{2}$$

$$\frac{x_2}{x_3} M^2 + \frac{1}{2} O_2 \rightarrow \frac{1}{x_3} M^2_{x2} O_{x3} + \Delta_R H^2 \tag{3}$$

If the Gibbs energy of Eq. (2) is lower than that of Eq. (3) the combined reaction described by Eq. (1) is the self-sustaining reaction. This way, couples of M^1 and M^2-oxides can be chosen on the basis of Gibbs energy from the ranking of the oxidation of elements with pure oxygen [6]. The enthalpy of the combined reaction is the difference between the reaction enthalpies of Eq. (2) and Eq. (3): $\Delta_R H = \Delta_R H^1 - \Delta_R H^2$. Then the reaction enthalpy can be used for a new ranking of the thermite reactions. In respect of the most application, the reaction enthalpy per reactant mass is of critical interest.

Figure 1 summarizes the general results of $\Delta_R H$ for the first 10000 reactions sorted by the *ranking number of decreasing* $\Delta_R H$ in logarithmic scale (solid dots). The highest heat of reaction is achieved by

$$Be + \frac{1}{2} NaO_2 \rightarrow BeO + \frac{1}{2} Na \tag{5}$$

with a value of -13.9 MJ/kg. This is a factor of 3.5 higher than the standard thermite reaction of Al/Fe_2O_3 of about 4 MJ/kg. The 10 elements Be > Li > Al > Mg > Sc > B > Si > Ca > Ti > Y > Zr appear first in the resulting ranking. Most prominent oxides which produce highest heat release are various oxides of sodium, potassium, selenium, lithium, chromium, technetium, phosphorus, arsenic, osmium, rubidium, manganese, vanadium, molybdenum, calcium, boron, iron, …

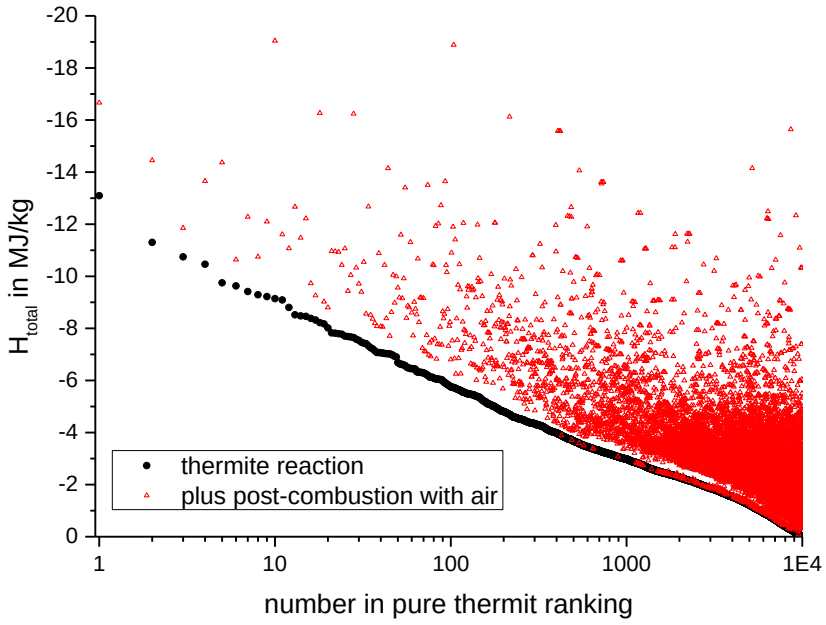


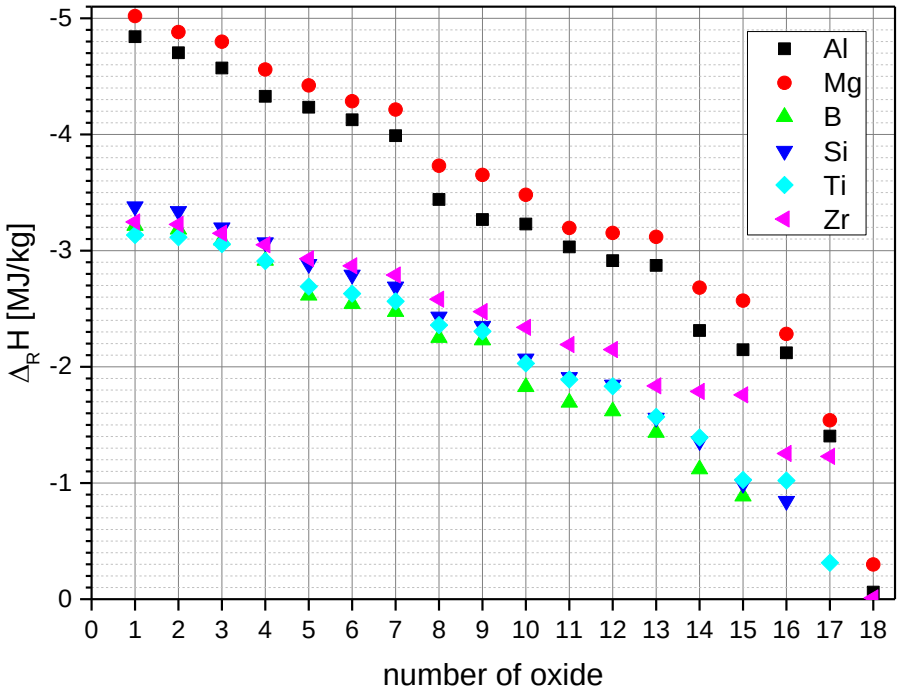
Figure 1 Calculated heat release of all considered thermite type reactions as function of the ranking number.

For many applications as incendiary or flare operating in ambient air, a second important aspect is the heat of combustion of the produced metal. This was also considered in this thermodynamic study. Figure 1 also plots the total reaction enthalpy of the thermite reaction plus the post-combustion with oxygen of the produced element M^2 (open triangles) using the same ranking number as the corresponding thermite reaction. This clearly demonstrates the large potential of an additional use of ambient oxygen. The largest heat release of more than 19 MJ/kg is provided by Be/LiO_2 followed by Be/B_2O_3 with 18.8 MJ/kg. It is obvious that also medium and even low energetic reactions with high ranking numbers achieve similar theoretical heat releases as the thermite combination Be/NaO_2.

Unfortunatelly many elements and oxides are inconvenient due to toxicity, stability, costs, availability, hygroscopicity or radioactivity. By exclusion of such species only thermite reactions remain with reaction enthalpies of below 6 MJ/kg.

For technical application the elements Al, Mg, B, Si, Ti and Zr appear to be most preferable and were selected also for the below described experimental studies. Selected oxides were: B_2O_3, BaO, BaO_2, Bi_2O_3, Co_3O_4, CrO_2, CuO, Fe_2O_3, MnO_2, MoO_2, MoO_3, NiO, PbO, PbO_2, SiO_2, SnO_2, V_2O_5, WO_3.

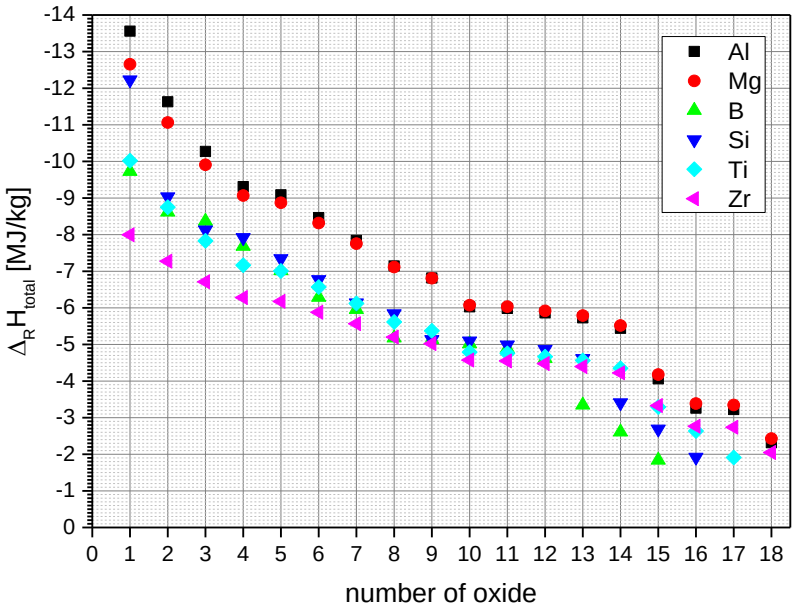
Figure 2 compares the calculated heats of reaction of this selection of the stoichiometric thermite reactions and in Figure 3 with additional post-combustion with oxygen. The values are plotted in decreasing order as function of the ranking number of the oxide. The individual correlation to the oxides is listed below the graphs.



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No.	Al	Mg	B	Si	Ti	Zr
1	MnO2	MnO2	MnO2	MnO2	MnO2	MnO2
2	MoO3	MoO3	MoO3	MoO3	MoO3	MoO3
3	O5V2	O5V2	CuO	CuO	CuO	CuO
4	CrO2	CrO2	Co3O4	Co3O4	Co3O4	Co3O4
5	Co3O4	Co3O4	O5V2	O5V2	O5V2	O5V2
6	CuO	CuO	CrO2	CrO2	CrO2	CrO2
7	Fe2O3	Fe2O3	Fe2O3	Fe2O3	Fe2O3	Fe2O3
8	NiO	B2O3	NiO	NiO	NiO	NiO
9	B2O3	NiO	O2Pb	O2Pb	O2Pb	O2Pb
10	MoO2	MoO2	MoO2	MoO2	MoO2	MoO2
11	O2Pb	O2Pb	O3W	O3W	O3W	O3W
12	O3W	O3W	O2Sn	O2Sn	O2Sn	O2Sn
13	O2Sn	O2Sn	Bi2O3	Bi2O3	Bi2O3	B2O3
14	BaO2	O2Si(Quartz)	BaO2	BaO2	BaO2	Bi2O3
15	O2Si(Quartz)	BaO2	OPb	OPb	B2O3	BaO2
16	Bi2O3	Bi2O3	--	B2O3	OPb	O2Si(Quartz)
17	OPb	OPb	--	--	O2Si(Quartz)	OPb
18	BaO	BaO	--	--	--	BaO

Figure 2 **Heat of reaction of metal/oxide combinations of selected thermites as function of oxide number in decreasing order.**



No.	Al	Mg	B	Si	Ti	Zr
1	B2O3	B2O3	O5V2	B2O3	B2O3	B2O3
2	O2Si(Quartz)	O2Si(Quartz)	CrO2	O5V2	O2Si(Quartz)	O2Si(Quartz)
3	O5V2	O5V2	MnO2	CrO2	O5V2	O5V2
4	CrO2	CrO2	MoO3	MnO2	CrO2	CrO2
5	MnO2	MnO2	Fe2O3	MoO3	MnO2	MnO2
6	MoO3	MoO3	Co3O4	Fe2O3	MoO3	MoO3
7	Fe2O3	Fe2O3	MoO2	Co3O4	Fe2O3	Fe2O3
8	Co3O4	Co3O4	NiO	MoO2	Co3O4	Co3O4
9	MoO2	MoO2	O2Sn	NiO	MoO2	MoO2
10	NiO	NiO	O3W	O2Sn	NiO	NiO
11	O2Sn	O2Sn	CuO	O3W	O2Sn	O2Sn
12	O3W	O3W	BaO2	CuO	O3W	O3W
13	CuO	CuO	O2Pb	BaO2	CuO	CuO
14	BaO2	BaO2	Bi2O3	O2Pb	BaO2	BaO2
15	O2Pb	O2Pb	OPb	Bi2O3	O2Pb	O2Pb
16	BaO	BaO	--	OPb	Bi2O3	BaO
17	Bi2O3	Bi2O3	--	--	OPb	Bi2O3
18	OPb	OPb	--	--	--	OPb

Figure 3 **Heat of reaction of metal/oxide combinations of selected thermites as function of oxide number in decreasing order including additional post-combustion with oxygen.**

Experimental Investigations

Samples

The selected species were stoichiometrically mixed according to the nominal reaction equation as thermite (Eq. (1)). Table 1 lists all compositions of the investigated combinations:

Oxid	Al	B	Mg	Si	Ti	Zr
B_2O_3	56,3%	--	48,8%	--	49,2%	33,7%
BaO	89,6%	--	86,3%	--	--	--
BaO_2	90,4%	92,2%	77,7%	85,8%	78,0%	65,0%
Bi_2O_3	89,6%	--	86,5%	--	86,7%	77,3%
Co_3O_4	77,0%	89,3%	71,2%	81,1%	71,6%	56,9%
CrO_2	70,0%	85,4%	63,3%	74,9%	63,7%	47,9%
CuO	81,6%	91,7%	76,6%	85,0%	76,9%	63,6%
Fe_2O_3	74,0%	88,1%	68,7%	79,1%	69,0%	53,9%
MnO_2	70,7%	85,8%	64,1%	75,6%	64,1%	48,8%
MoO_2	--	--	64,1%	--	72,8%	--
MoO_3	72,7%	86,9%	66,4%	77,4%	66,7%	51,3%
NiO	80,6%	77,6%	75,5%	--	--	--
PbO	92,5%	--	90,2%	--	--	--
PbO_2	86,9%	--	83,1%	--	83,3%	72,4%
SiO_2	62,6%	--	55,3%	--	--	--
SnO_2	80,7%	91,3%	75,6%	84,3%	75,9%	62,3%
V_2O_5	66,9%	83,5%	60,0%	72,2%	60,3%	44,4%
WO_3	81,1%	91,5%	76,1%	84,6%	76,3%	62,9%

Table 1 Compositions of investigated thermite mixtures.

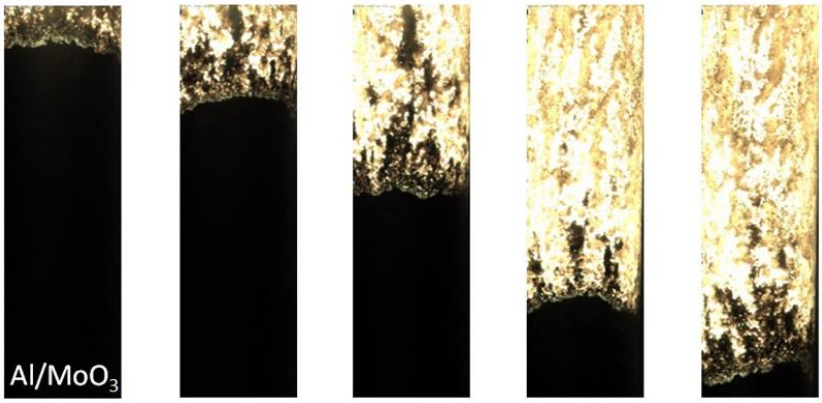
All components had a micro-size particle diameter between 2 to 60 µm. No nanoparticles were investigated in this study.

The experimental tests concentrated on the reaction rate of the pure thermite mixtures, characterizing the propagation velocity inside a test tube and determining the reaction temperature in nitrogen and air.

Propagation Rates

The mixtures were filled into test tubes (diameter 7 mm, length 70 mm) and compacted by stamping to realize the same bulk density for each thermite mixture. The regression rate was measured in analogy to the burning rate of solid or gelled propellants [17][18][19] using a high-speed camera inside a chimney-type optical bomb at constant atmospheric pressure provided by a low nitrogen flow. The samples were ignited using a melting wire. Some samples of certain mixtures could not be ignited at all.

In case of ignitability the mixtures burnt top-down with a plane reaction front and a constant propagation rate at the very most. Figure 4 shows a characteristic example of an Al/MoO_3 mixture and the corresponding regression picture whose slope was used to determine the progression rate.

a) 

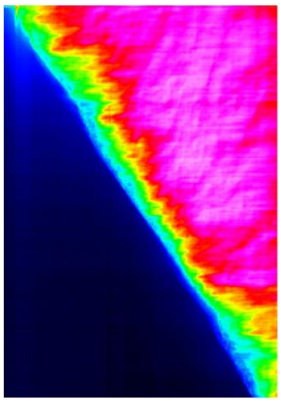
b) 

Figure 4 **a) Progression of Al/MoO_3 thermite reaction at 0.1 MPa inside a test tube; b) corresponding regression picture**

Every sample was tested 3 to 5 times. Abnormal tests were neglected and repeated. This method to characterize the total reaction velocity of the investigated thermite results in a suitable reproducibility that strictly depends on the thermite couple as to be seen in Table 2. This table summarized the spread of measured values of each thermite combination. In Figure 5, a bar chart is shown allowing a better comparision of the total range of mean values (in logarithmic scale), sorted by oxides in alphabetical order. The metal fuels are marked by order and colour.

Depending on the thermite composition the absolute propagation rates vary from about 2 mm/s (Si/CrO_2) up to 20 m/s (Al/SnO_2). But measured values of most thermite combinations rank between 10 und 100 mm/s. The mixture Zr/Bi_2O_3 reacted also extremely fast but without constant progression rate and could not be analysed.

In many cases the progression rates of aluminium and magnesium are very fast. In general mixtures with silicon react most slowly followed by boron. With reference to the oxides no trend is obvious.

	Al	**B**	**Mg**	**Si**	**Ti**	**Zr**
	mm/s	*mm/s*	*mm/s*	*mm/s*	*mm/s*	*mm/s*
B_2O_3	no go	--	no go	--	no go	no go
BaO	no go	--	no go	--	--	--
BaO_2	32,8-40,3	38,1-45,9	53,3-55,8	15,0-16,3	19,9-20,1	38,7-39,6
Bi_2O_3	3930-5210	--	166	--	46,3-54,0	n.a.
Co_3O_4	32,5-33,1	5,33-5,58	34,7-36,8	3,14	10,5-10,8	17,9-20,1
CrO_2	15,1-29,7	2,79-3,17	31,6-62,8	2,18-2,40	9,72-35,4	42,1-90,4
CuO	39,6-146,75	6,0-6,1	61,8-78,0	4,24-4,28	84,5-96,1	85,1-90,7
Fe_2O_3	52,4-75,5	no go	11,0-51,8	no go	16,2-47,5	71,2-72,4
MnO_2	30,4-56,6	5,2-5,3	5,5-27,3	4,29-4,89	20,1-22,1	86,3-91,2
MoO_2	--	--	20,0-22,9	--	10,1-11,0	--
MoO_3	409,5-745,3	6,0-37,0	39,5-52,4	no go	24,6-37,9	35,6-48,2
NiO	48,6-91,1	--	27,3-29,2	--	--	--
PbO	24,3-25,2	--	67,1-89,7	--	--	--
PbO_2	6,31-13,4	--	3340-4610	--	3,83-4,24	476,1
SiO_2	no go	--	12,5-18,9	--	--	--
SnO_2	17700-20100	14,1-15,1	30,6-34,4	4,40-4,50	17,7-19,7	44,4-45,7
V_2O_5	30,3-33,6	7,62-7,85	65,7-87,0	no go	17,4-17,7	54,1-55,4
WO_3	53,1-64,1	no go	16,2-17,4	no go	18,8-20,4	22,2-22,7

Table 2 **Spread of progression rates of all investigated mixtures under inert gas conditions (n.a. no analysis possible, -- not investigated).**

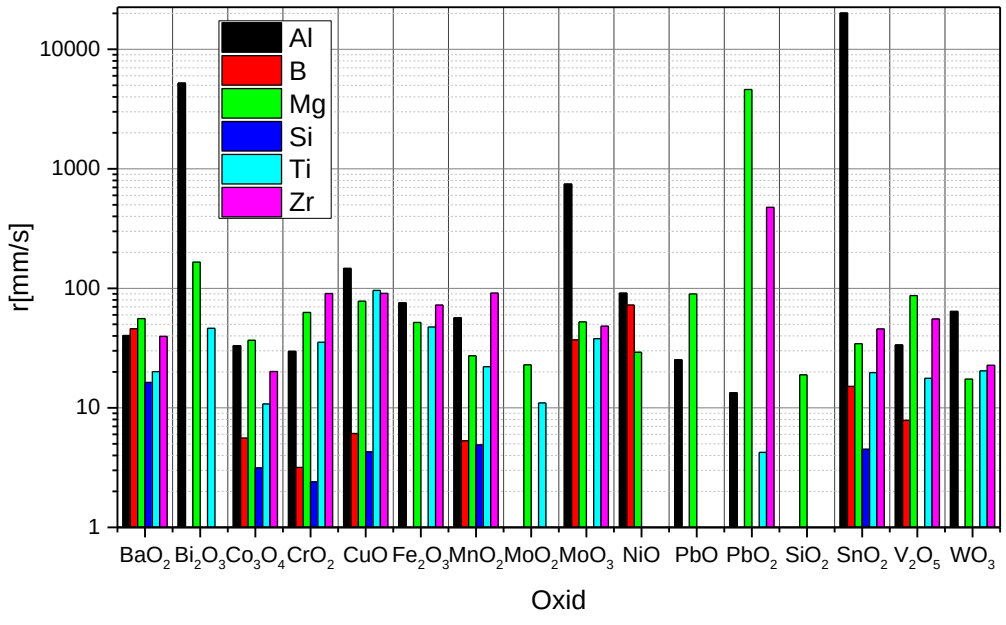


Figure 5 **Mean progression rates of all investigated mixtures under inert gas conditions**

Reaction Temperature in Nitrogen

The absorption and emissivity characteristics of the used test tubes did not allow a simultaneous measurement of reaction temperatures and progression rates. So all mixtures were additionally burnt in the optical bomb as bulk piles (of about 25x10x10 mm^3) at 0.1 MPa nitrogen also ignited with a melting wire. A possible reactive influence of nitrogen was neglected. The reaction was observed with a high-speed camera and a UV to NIR emission spectrograph. Temperature was measured by analyzing the NIR-range of the continuum emission assuming spectral grey body distribution which described the measured spectra with high agreement. The method is described in Ref. [17].

The test method proved to be reproducible for each thermite combination to characterize especially the measured temperature history. But not all mixtures could be ignited. In this contribution, the absolute maximum temperatures were chosen to characterize the heat release of the mixture. It should be stated, that this procedure mainly characterizes only a part of thermal reaction properties as e.g. radiation or heat transfer but do not specify the total heat release because some samples reacted very hot over a short time period of some hundred milliseconds while others burnt several seconds on a lower temperature level (refer Figure 7b).

Figure 6 summarizes these values in a similar bar chart as Figure 5. Absolute maxiumum temperatures vary from 1200 K (Mg/BaO) to >3000 K (Al/MoO_3). In general, mixtures containing aluminium achieve the highest maximum temperatures followed by thermite combinations including zirconium. All mixtures containing magnesium could be ignited and burnt also with high maximum temperatures. Most samples containing boron burnt with lower temperatures. Again no trend with respect to the type of oxide is obvious.

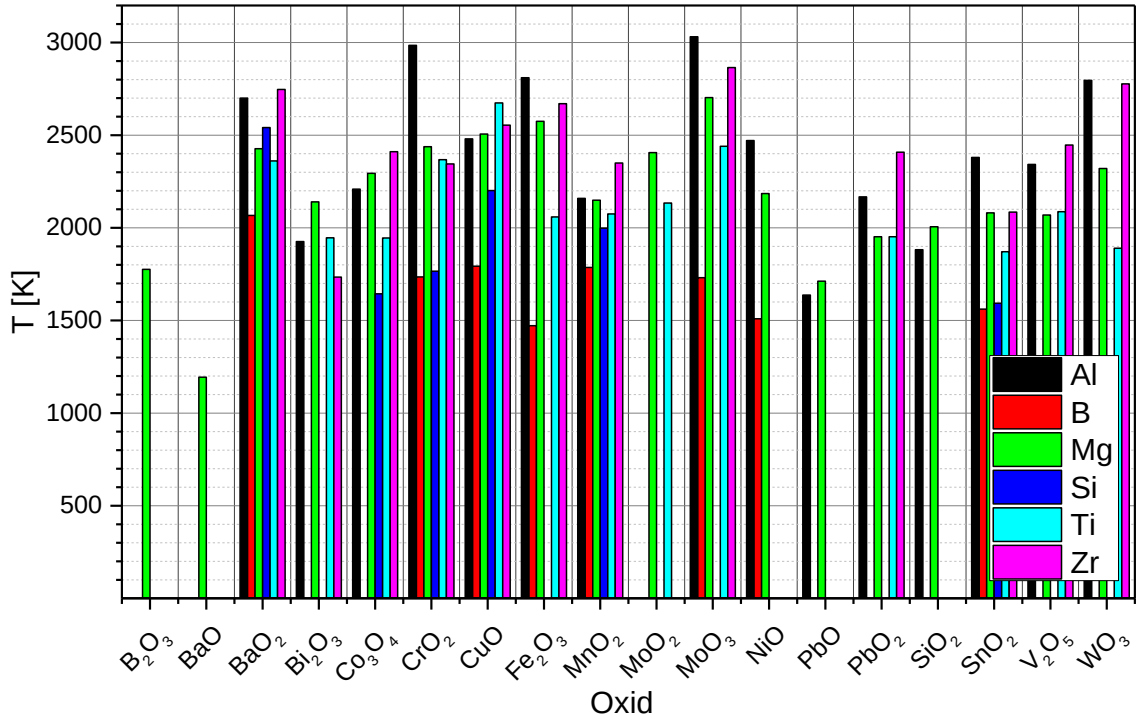
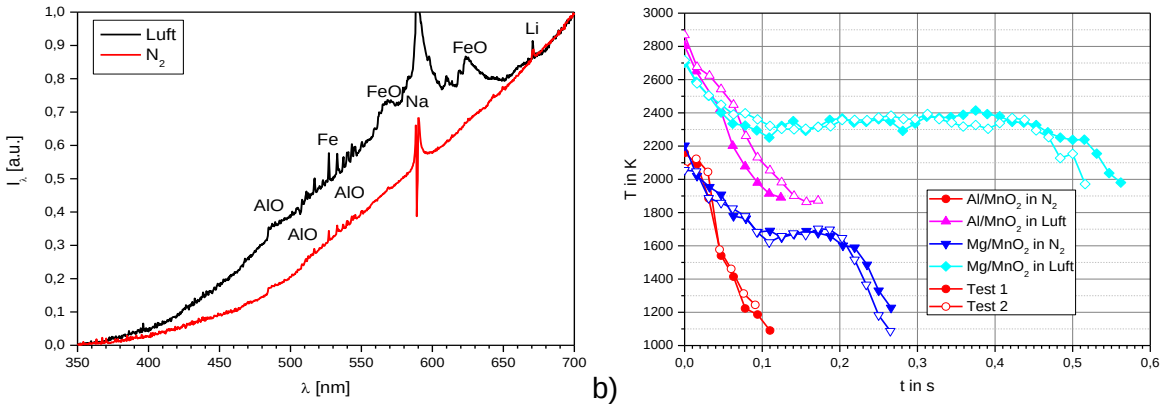


Figure 6 Maximum temperatures measured for all investigated thermite pairings in nitrogen.

Temperature of Thermite Reaction in Air

To investigate the reactive interaction or post-combustion of thermite reaction products with air the samples were also ignited and analysed in free atmosphere as bulk piles on a Ceran plate in similar manner as described before. With these experimental settings more compositions could be ignited as in nitrogen atmosphere.

As known from earlier investigations most thermite type reactions run different in air as in nitrogen [5]. A desired effect would be that the metal reduced by the thermite reaction will exothermically re-oxidise with ambient oxygen to increase the total heat release as discussed above. This is clearly demonstrated in Figure 7a by comparing emission spectra taken from the Al/Fe_2O_3 reaction in nitrogen (red) and in air (black). Beside lines of elemental iron and di-atomic systems of gaseous AlO, the spectra of the reaction in air additionally features FeO systems of iron oxidation. So the re-oxidation of iron is definitely proven. The exothermic afterburning with air is also evident comparing the temperature histories of thermite reactions taking place either in nitrogen or air. Figure 7b presents such results for the reaction of Al and Mg with MnO_2. In each case two tests were performed resulting in an excellent reproducibility. The radiation temperatures measured in air are at least 600 K higher than those in nitrogen. The reaction runs for a significantly longer time.



a) b)

Figure 7 a) UV/Vis emission spectra of Al+Fe2O3 reacting in nitrogen and air; b) temperature history of Al+MnO_2 and Mg+MnO_2 reacting in nitrogen and in air.

Beside the metallic emission lines, the spectra are dominated by spectral continuum emission that again allows to determine the radiation temperature in NIR range assuming grey body emission. Figure 8 compares the maximum temperatures measured for each thermite pairing in a bar chart explained above. These maximum temperatures rank from 1550 to 4500 K and are several hundred Kelvin higher as those measured in nitrogen. Again lower temperatures correspond to silicon and boron, highest temperatures correspond to aluminium, magnesium and zirconium, but the temperature differences for each groupe of the same oxide are lower compared to the reaction in nitrogen. In general a certain influence of the type of oxide is observable. So the highest temperatures correspond to the compositions containing MoO_3 followed by PbO_2.

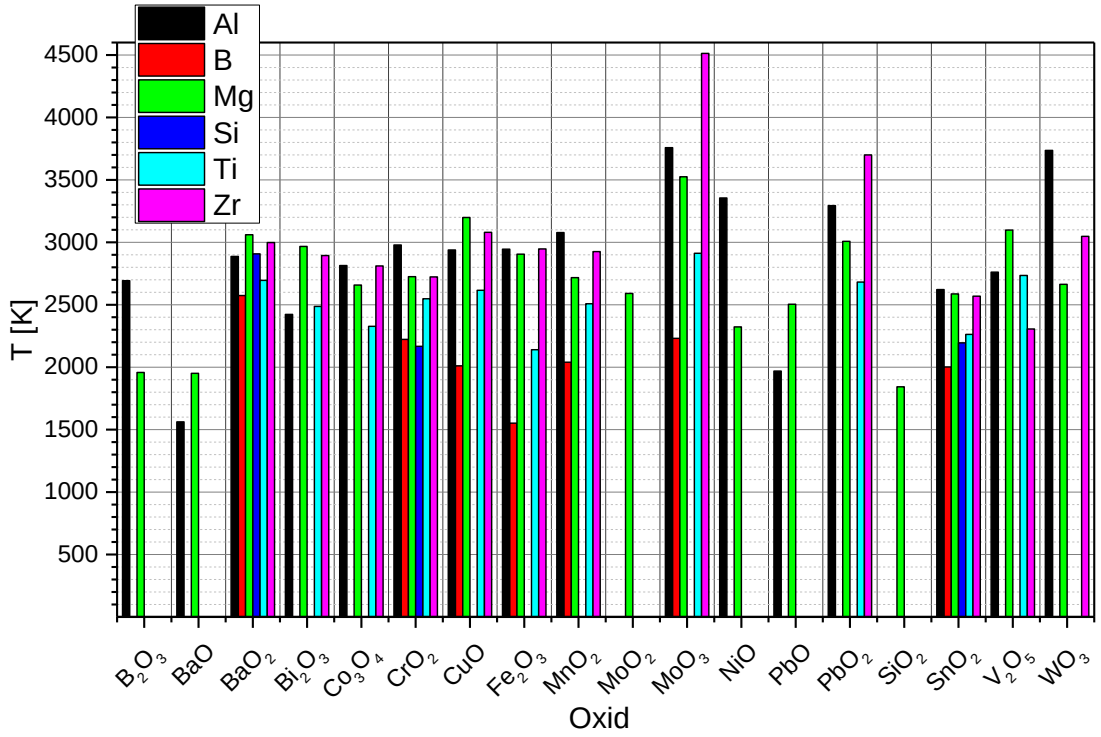


Figure 8 **Maximum temperatures measured for all investigated thermite pairings in air.**

Conclusions

Thermite type reactions offer promising properties as main ingredient for incendiaries, flares, igniter mixtures or delay compositions. These so-called solid state reactions appear particularly preferable concerning their large mass specific heat release, high reaction temperatures, strong emissivity and the ability to eject hot metal particles that are able to react exothermically with ambient air.

The possibility to combine many different metal or semi-metal particles with an even higher number of oxide particles offers the potential to tailor pyrotechnic compositions with adapted heat release rates. This potential drastically increases by the possibility of combining two or more thermite compositions with different properties.

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