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Characterisation of the activation and reaction behaviour and determination of the emissivity of reactive nickel-aluminium particles with regenerated fibre Bragg gratings

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Abstract. Reactive particles represent a customisable heat source for joining applications as each reactive particle is able to undergo an exothermic, self-sustaining reaction. Microwaves are used to homogeneously initiate this reaction and allow the resulting temperature-time profiles to be influenced. The possibility to derive cause-effect relationships between the different particle structures, the activation energy, and the resulting maximum temperatures is essential for developing a resource-efficient, tailored heat source in production engineering. The reaction of reactive nickel and aluminium particles generates heat and thus temperatures up to 1500 K within milliseconds. A promising temperature measurement method despite an unknown emissivity are optical fibre sensors. These feature high frame rates, a compact design, and resistance to high temperatures as well as to electromagnetic waves. In this paper, the investigation of the use of regenerated fibre Bragg gratings (RFBG) as a new, innovative approach to characterise reactive particles is described. Experimental studies with an infrared camera and RFBG demonstrated that RFBG retain their functionality while being exposed to microwaves and high temperatures. The good accordance of the recorded RFBG-based temperature evolutions with infrared thermography data confirmed the suitability of RFBG as a sophisticated characterisation method and for determining the emissivity of reactive metal particles.

1. Introduction

Industrially established joining technologies like welding, soldering, and adhesive bonding, require a sophisticated heat source, which guarantees an efficient and controllable energy input. Reactive particles meet these requirements, since they are able to provide energy by undergoing a self-sustaining, exothermic reaction. Particularly, when joining materials with different thermo-physical properties, a customisable energy input is essential to avoid thermal damage of the components in the heat-affected zones and to promote the bond formation. Measuring temperature profiles is a standard procedure used in academia and industry to quantify the impact of the heat source on the material. However, the characterisation of reactive particles is a demanding challenge. High-temperature measurement with frame rates in the range of kilohertz and an unknown emissivity is not yet state of the art. Optical fibre sensors, such as regenerated fibre Bragg gratings (RFBG), represent a promising solution for investigating reactive particles. They enable recording high-resolution temperature-time

evolutions and thereby can contribute to a profound understanding of the activation and reaction behaviour, which is necessary for the use of reactive particles in mechanical engineering.

1.1. Activation and characterisation of reactive particles

In the field of combustion synthesis, which is also known as self-propagating high temperature synthesis (SHS), powder materials or green compacts consisting of homogeneously mixed educts are used for the industrial manufacturing of advanced materials [1]. Moreover, reactive systems in the form of foils, wires, and bimetallic particles are investigated in scientific research. Figure 1 illustrates the different types of reactive particles and various intrinsic structures of particles with two reactants.
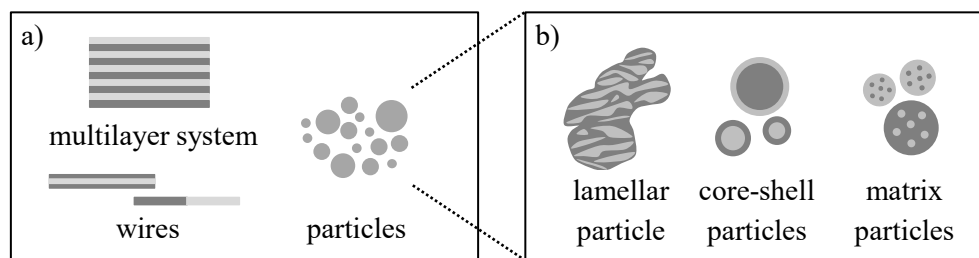
Figure 1. Scheme of a) different types of reactive systems and b) intrinsic structures of bimetallic reactive particles according to [2].

Bimetallic reactive particles can be divided into three categories according to their intrinsic structure: *core-shell particles* are obtained through electroless [3] or galvanic [4] plating processes, *lamellar particles* are commonly synthesised via high energy ball milling [5], and *matrix particles* are produced by either ultrasonic powder consolidation [2] or by planetary ball milling. Reactive particles represent an innovative heat source for thermal joining applications as each particle is able to release a defined amount of energy depending on the stoichiometric ratio of the contained reactants. Furthermore, they are a stable and compact alternative to brittle or fragile multilayer systems and wires, which are not or only to a limited extent applicable onto freeform surfaces.

A prominent example of a reactive system, which has induced increased interest in academia and industry, is nickel and aluminium. Nickel aluminides are high performance materials that form five intermetallic phases: $NiAl$, $NiAl_3$, Ni_2Al_3, Ni_3Al, and Ni_5Al_3. Due to their advantageous characteristics like excellent mechanical properties, low densities, high melting points, and good oxidation as well as chemical resistance, $NiAl$ and Ni_3Al have received the most attention [6; 7]. The combustion reaction of reactive systems with an equimolar ratio of nickel and aluminium exhibits the highest enthalpy of reaction of -62 ± 2 kJ/mol at 298 K [8]. Furthermore, these nickel aluminides feature the highest melting points of 1911 K [7]. Sophisticated investigations of the cause-effect relationships between the intrinsic structure and the resulting activation and reaction behaviour provided important findings about the reaction mechanisms in nickel and aluminium systems and demonstrated the importance of a liquid aluminium phase for the start of the chemical reaction [9–11].

A further, significant option to influence the characteristics of the combustion reaction is the ignition technique, which directly correlates with the heating rate. Reactive systems can generally be activated through a locally confined or a homogeneous energy input, which triggers either the propagating or the simultaneous combustion mode [12]. It is important to note when studying reactive particles, that the available contact area between the single particles may promote, limit, or inhibit the formation of a self-propagating reaction front. A widely used approach in combustion synthesis to ensure sufficient contact is to manufacture compacted pellets. However, varying densities of the green compacts affect thermal conduction processes and lead to deviations of the maximum combustion temperature T_{max} as well as to differences in the reaction kinetics.

Analysing loose reactive particles in order to avoid these unintended effects on the activation and reaction behaviour requires a homogeneous energy input. Typical activation methods to initiate the

simultaneous combustion mode include induction heating [13], a modified hot isostatic pressing (HIP) process [14], and heating in an oven, which might simultaneously be used for dynamic scanning calorimetry (DSC) [15] or time-resolved X-ray diffraction (TRXRD) [16] experiments. As heat transfer is often accompanied by temperature gradients that can affect the uniformity of the reaction, energy transfer into the reactive particles is a valuable alternative.

In this context, electromagnetic waves represent an efficient energy transfer method and are especially suitable for the volumetric heating of particles [17; 18]. Microwaves at a frequency of 2.45 GHz have been successfully used to initiate the combustion reaction of nickel and aluminium particles [19; 20]. Major advantages of utilising microwaves include the possibility to realise high heating rates, adjust them in a relatively wide range, and influence exothermic phase transformations. Furthermore, microwaves allow to manipulate the reaction even after the initiation, which is a unique option considering the high reaction rates in combustion reactions of nickel and aluminium. However, discrepancies in the experimental data in scientific publications of the ignition temperature T_{ig} and the maximum combustion temperature T_{max} can often be attributed to different experimental set-ups and only partially comparable temperature measurement techniques.

The ability of microwave-activated reactive particles to reach high temperatures within milliseconds to seconds is quite advantageous for reducing cycle times, but extremely challenging for measuring temperature profiles. Measurement methods must guarantee high frame rates to capture changes in temperatures, which might indicate intermetallic phase transformations, and should not interact with the electromagnetic field. Even though thermocouples are an elaborated and established measurement method, they are not suitable in this case as they reflect a certain amount of microwave radiation and might also act as transmission line or antenna.

Contactless temperature measurement methods, which detect emitted electromagnetic radiation, such as infrared thermography, are compatible with microwaves, but require profound knowledge of the emissivity of the material. In contrast to emissivity values for bulk materials, which are often documented in table books, reference values for particles are not reliable since powder beds feature non-reproducible cavities. These, in turn, depend on the particle shape, the size distribution, and the degree of compaction. Sophisticated mathematical equations [21; 22] are valuable approaches, but are not able to consider the phase transitions during the combustion reaction.

A promising technique for the desired temperature measurement are optical fibres, which have already been successfully applied in the form of sapphire fibres in combustion synthesis [20]. Fibre Bragg gratings represent an innovative high-temperature measurement method for reactive particles and enable the determination of the emissivity of powder beds when combined with an infrared camera system.

1.2. Regenerated fibre Bragg gratings
Optical fibre sensors based on Bragg gratings have been successfully used in a wide range of applications, like measuring the temperature gradient during the casting process of an aluminium alloy [23], in gas turbines [24], or in chemical and nuclear reactors [25]. Particularly noteworthy are their immunity to electromagnetic fields [26] as well as their small size [27]. Typical optical fibre diameters of 125 µm and sensor lengths from 0.5 mm up to a few millimetres enable their integration into even small-sized structures and thus a compact design of the experimental set-up. In addition, fibre Bragg gratings offer the possibility of wavelength multiplexing, which means that multiple measurement locations can be included in a single optical fibre [28; 29].

Fibre Bragg gratings (FBG) consist of refractive index modulations with a period length Λ of about 0.5 µm inside the fibre's core. If broadband waves propagate through the optical fibre and experience the refractive index modulation, the waves will be partially reflected at each refractive index step. The reflected waves interfere constructively if their wavelength is equal to the Bragg wavelength and destructively for all other wavelengths. The Bragg wavelength λ_B [30] is given by

$$\lambda_B = 2\, n_{eff} \Lambda \, , \qquad\qquad (1)$$

where η_{eff} is the effective refractive index. If heat is applied, the Bragg wavelength increases due to the thermo-optic effect and the thermal expansion of the silica fibre. For large temperature differences, a nonlinear relationship between the temperature und the Bragg wavelength occurs [23; 31; 32]. Fibre Bragg gratings, which are inscribed in optical fibres by using the photosensitivity of the germanium doped core, are referred to as Typ*I*-FBG [33]. If a Typ*I*-FBG is exposed to temperatures above approximately 600 K, the grating will degrade until it vanishes completely [34]. Temperature stable Bragg gratings, which have encountered a specific temperature treatment, like regenerated fibre Bragg gratings (RFBG), are resistant up to 1550 K [35]. With these RFBG, high-temperature measurements are possible.

2. Experimental set-up and materials

In order to evaluate the potential of RFBG sensors as an innovative temperature measurement method, specific RFBG and suitable sensor packages have been developed. Furthermore, an elaborated experimental set-up is required which allows both the integration of the fabricated sensors and the simultaneous observation with an infrared camera as a reference and additional source of information for the activation and reaction behaviour.

2.1. Fabrication of RFBG and sensors

The RFBG were produced employing a custom-built inscription set-up and a thermal annealing process. The FBG had a length of 3 mm and a Bragg wavelength of 1550 nm. The optical fibre sensors were mounted inside of glass capillaries with outer diameters of 360 μm as depicted in Figure 2.
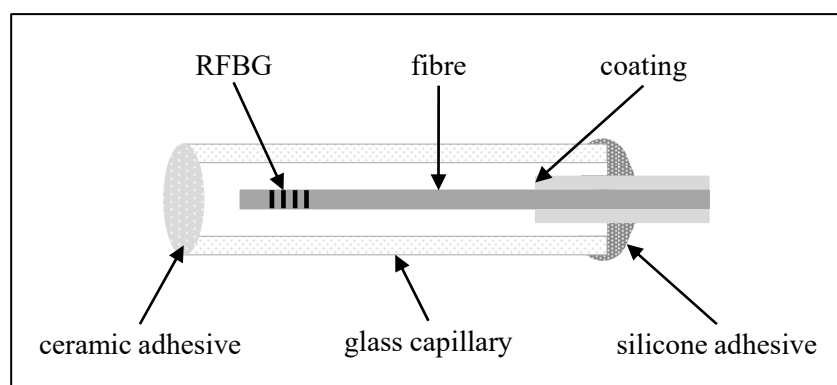
**Figure 2.** Scheme of the RFBG sensor package.

The end of each capillary was sealed with a ceramic adhesive to avoid unintended contamination and direct contact with the reactive particles. The Bragg wavelength was measured with a FBG Interrogator (I4, FAZ Technology Ltd., Dublin, Ireland). Its data sampling rate was 1 kHz.

The FBG were calibrated from room temperature up to 1073 K and the temperatures were determined on the basis of a fifth order polynomial according to [23]. Above 1073 K, the polynomial was linearly extrapolated.

2.2. Experimental set-up and temperature measurement

The investigated reactive systems consisted of reactive particles with fine lamellar structures of nickel and aluminium (Indium Corporation, Clinton, USA). Circular disk-shaped powder samples with identical masses were prepared with the help of a template with a diameter of 18 mm and a height of 1 mm. A groove within the template ensured a reproducible positioning of the fibre sensor within the sample. Boron nitride sheets (HeBoSint, Henze Boron Nitride Products AG, Lauben, Germany) served

as temperature resistant base material. Furthermore, boron nitride interacts with microwaves only to a limited extent, which is of major importance for the experimental set-up shown in Figure 3.
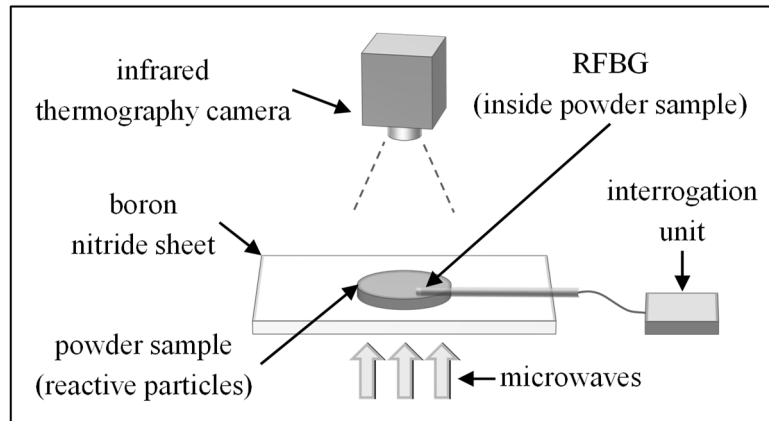

Figure 3. Scheme of the experimental and measurement set-up for the microwave activation of reactive particles.

A magnetron operating at a frequency of 2.45 GHz (MH2000S-215BB, Muegge GmbH, Reichelsheim, Germany) was used for the microwave activation of the prepared samples. An infrared (IR) camera (ImageIR 8300, InfraTec GmbH, Dresden, Germany) captured two-dimensional information of the reaction process at a frame rate of 1 kHz. As the emissivity ε of the reactive particles is not known, a value of $\varepsilon = 1$ was assumed, which allowed an initial estimation of the observed temperature distribution.

3. Results and discussion

Initial experiments aimed at investigating the influence of the microwaves on the optical fibre sensors and the RFBG. Therefore, a fabricated sensor was placed on the boron nitride sheet without any reactive particles and the microwave power was increased to 450 W at 50 W intervals. Neither the infrared camera nor the interrogation unit detected changes of the sensor's behaviour.

For the following experiments, the microwave power was set to 200 W. Three samples of reactive particles were activated as depicted in Figure 3. Scanning electron micrograph images of unreacted and microwave-activated lamellar particles are shown in Figure 4.
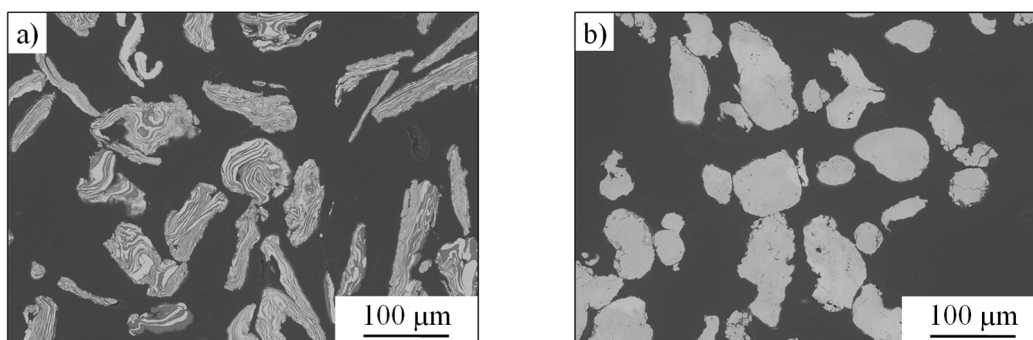

Figure 4. Scanning electron micrograph images of a) unreacted particles consisting of nickel (light grey) and aluminium (dark grey) and b) reacted particles.

The scanning electron micrograph images illustrate that both the shape and the size distribution of the unreacted particles deviated. Spherically and needle-shaped particles with fine lamellar structures

consisting of nickel and aluminium were found. Microwave-activated particles featured nickel aluminides, which was also indicated by an energy dispersive X-ray spectroscopy analysis.

During the activation, which was performed three times, reactive particles were monitored with an RFBG-based sensor and the infrared camera. Figure 5 shows selected results.
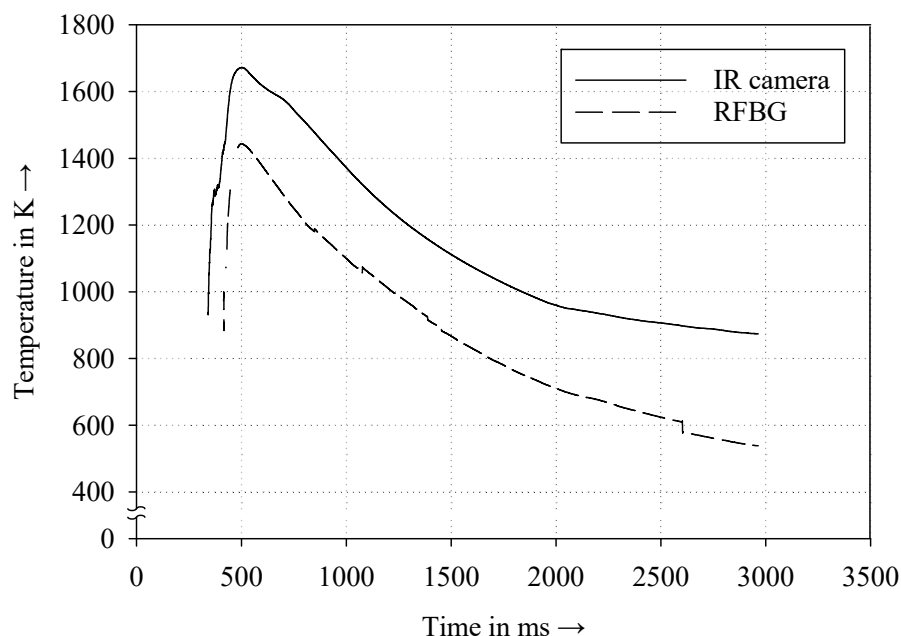
Figure 5. Temperatures as a function of time measured by a fibre sensor (RFBG) and with the infrared camera (IR camera).

Important conclusions can be drawn from the measured temperature-time profiles. The qualitative course of the recorded RFBG-based temperature evolutions show good accordance with the infrared thermography data. Expectedly, due to the overestimated emissivity, higher temperature values were measured with the infrared camera. Maximum temperatures were generated through the exothermic reaction of the reactive particles within 500 ms after the activation with the microwaves. A comparison of the measured values of the three different RFBG sensors (not shown for reasons of clear arrangement) indicated that the qualitative courses of the curves were identical, while maximum temperatures differed (1500 K ± 51 K). Similar observations for the data of the infrared camera suggested that deviations of the particle shapes and the size distribution of the prepared samples might have influenced the uniformity of the analysed combustion reactions and thus led to minor variations of the measured temperatures.

The great advantage of measuring with two independent systems is that the results can be used to derive the emissivity of the reactive particles during the reaction. For the example illustrated in Figure 5, an emissivity of 0.86 was calculated for the maximum temperature. This possibility represents a highly valuable approach not only for the field of combustions synthesis, but also for other metal-powder-based processes, like for example laser beam melting.

Moreover, the measured data formed the basis to evaluate the activation and reaction behaviour of the reactive particles. It can be stated that the obtained temperature-time profiles are comparable with other findings in literature. According to [36], it is assumed that interactions between a liquid aluminium and a solid nickel phase took place as the temperature-time profiles featured single peaks. As mentioned before, microwaves enable an efficient energy transfer and high heating rates, which promote solid-liquid or liquid-liquid interactions instead of solid state diffusion processes.

It also becomes evident that there are missing values for the RFBG-based fibre sensor. This probably resulted from predominant temperature gradients along the optical fibre sensor, which

distorted the spectrum of the RFBG and prevented the calculation of the Bragg wavelength. However, this evaluation is essential for deriving temperature values. Nevertheless, informative temperature-time profiles were obtained.

A further important outcome is the suitability of the fibre sensors for continuous recording regardless of the current temperature. In contrast to the fibre sensors, the infrared camera required a pre-selection of a specific temperature range. Consequently, several samples had to be activated in order to obtain a complete temperature-time profile starting from room temperature. As mentioned before, even similarly prepared powder beds feature deviating emissivity values which affects the comparability and complicates matching of the data. Therefore, the results of the infrared camera in Figure 5 are limited to temperatures ranging from 873 to 1773 K. Complete temperature-time profiles without missing values or limited temperature ranges allow the evaluation of the ignition temperature T_{ig} and the enthalpy of reaction, which in turn is essential for analyses of the reaction characteristics.

Finally, in order to evaluate a possible sensor drift of the RFBG during the exposure to microwaves and high temperatures, additional RFBG spectra were determined before and after the reaction in a temperature-stabilised room temperature furnace. The shape of the spectra before and after the use of the RFBG were almost identical. The differences of the Bragg wavelengths were 10.3 pm ± 8.7 pm, which demonstrated the resistance to electromagnetic waves as well as to the reaction conditions of reactive particles. Furthermore, this highlights the potential of using optical fibre sensors with RFBG as an innovative characterisation method in combustion synthesis.

4. Conclusions

Reactive particles, which were originally used in the field of combustion synthesis, represent a promising heat source for joining applications, such as welding, soldering, and adhesive bonding. Depending on the stoichiometric ratio of the reactants, a customisable amount of energy can be released by each particle through an exothermic reaction. As heat transfer is often accompanied by temperature gradients, which influence the resulting activation and reaction behaviour, the use of electromagnetic waves at a frequency of 2.45 GHz as an efficient energy transfer is a valuable ignition method. However, high temperatures, electromagnetic waves, high reaction rates, and unknown emissivity values of powder beds represent demanding challenges for conventional temperature measurement methods. A sophisticated approach are optical fibre sensors based on regenerated fibre Bragg gratings (RFBG). Their successful deployment for the characterisation of reactive nickel and aluminium particles with a lamellar structure has been demonstrated in this paper. Temperature-time profiles, recorded with RFBG-based sensors and an infrared camera, showed good accordance. The additional possibility to determine the emissivity of metallic particles demonstrated the potential of RFBG for a profound evaluation of reactive particles in mechanical engineering.

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