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Atmosphere monitoring in a continuous sintering belt furnace

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ABSTRACT

The atmosphere composition in a continuous belt furnace during sintering of powder metallurgical aluminium components with ethylenbisstearamide (EBS) pressing agent was determined by Fourier Transform Infrared Spectroscopy (FTIR). The spatial distribution of the decomposition products was measured at four positions in the dewaxing zone. It is shown that the major decomposition products of the dewaxing process are CH groups, which mainly occur between 310 and 410 °C. As the powder compacts pass the dewaxing zone, each part drags along its own bell-shaped gas atmosphere. In the vertical direction, a flow profile with two separated concentration zones is observed, leading to large quantities of unused process gas passing the furnace. The amount of unused gas is mainly controlled by the gas flow and the belt speed of the furnace. In particular, high belt speeds and low gas flow parameters may lead to the introduction of harmful carbon into the sintering zone.

1. Introduction

Powder metallurgy represents an efficient method for the production of structurally complex metal components. For cost efficient production, continuous furnaces with high output represent the gold standard. Such furnaces are typically comprised of various components: an inlet zone, followed by the dewaxing zone, where organic pressing agents are pyrolized. Sintering is then carried out in the hot zone, and the final zone serves as a fast cooling zone. Continuous furnaces are complex systems since various parameters like belt speed, furnace load, flow parameters and geometry influence the temperature and atmosphere profiles. Thus, exact parameterization is not trivial. As an example, complex heat transfer and heat transmission model based controllers have been developed in order to improve the temperature stability (Bitschnau and Kozek, 2009).

The stabilization of the atmosphere is essential in order to ensure appropriate reproducibility. The positive effects of atmosphere injectors, flame curtains on the atmosphere distribution and thus on the maintenance of the furnace has been described by Silva et al. (2012) in their review on the developments over the past 30 years. In a recent approach, Hryha et al. (2012) have monitored the reducing and carburizing/decarburizing processes of PM steel components by using CO/CO_2 sensors in order to minimize oxidation effects in a batch furnace. In a continuous furnace, Malas (2011) has shown that oxygen probe and CO sensors, which are situated in external chambers, allow monitoring of further lubricant decomposition and carbon control of PM steels. It has been shown by Palermo and Malas (2014), that such systems can be applied to optimise the use of trim gases in order to improve the part quality. However, the data based knowledge on the composition and distribution of process atmospheres in continuous furnaces is rather scarce.

In continuous furnaces, the process gas has various functions, depending on the material to be sintered. PM steels need carburizing/decarburizing/neutral gases in the sintering zone, and reducing atmosphere in the heating zone. In the dewaxing zone, lubricant is removed by decomposition in dry atmospheres. As Hryha et al. (2013) state, typically used lubricants may lower the atmosphere purity significantly and may form stable oxides (as it is the case with aluminium), therefore, after rapid removal of the lubricant, the transport of decomposition products into the sintering zone has to be prevented.

In sintering of aluminium the function of the process atmosphere is somewhat different. Aluminium needs Mg-containing liquid phases during sintering since its surface oxide cannot be reduced in practically achievable atmospheres. In any case, too high of an oxygen partial pressure in the process atmosphere leads to stable oxide layers and hamper the sintering process locally, resulting in porous surface layers (Schubert et al., 2012). Dry nitrogen

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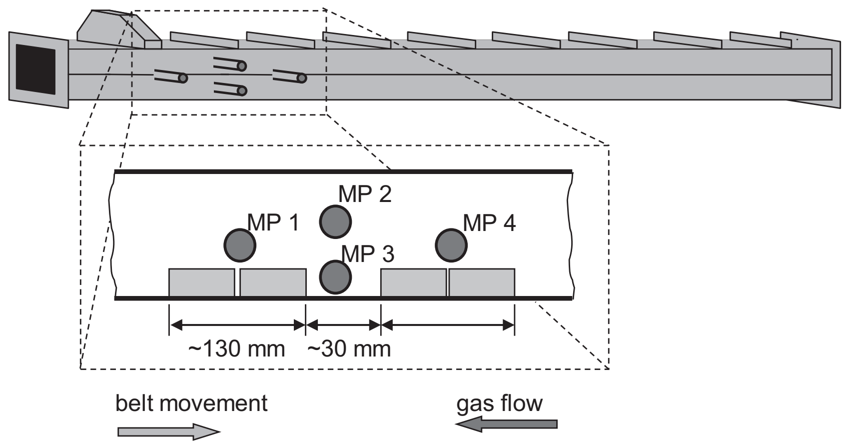

Fig. 1. Position of the four measuring flanges (indicated as measuring point MP 1–4) at the continuous aluminum sintering furnace and position of the powder compacts (grey rectangles) with respect to the flanges.

is the most efficient atmosphere for sintering of aluminium. The known self-gettering effect at the surface of pressed samples can reduce the oxygen partial pressure within the bulk, thus supporting the sintering performance (Schaffer and Hall, 2002). However, the careful control of the oxygen impurities in the furnace atmosphere is important during sintering of hypereutectic Al–Si alloy (Schubert et al., 2012). The detrimental effect of such oxygen residues in the process atmosphere on the shrinkage behavior also has been reported for sintering of AlZnMgCu alloys (Yuan et al., 2014).

A quality-determining step prior to sintering is the delubrication process, where pressing agents and further additives are partially pyrolized. The fundamentals of thermal decomposition of such polymers were investigated in earlier studies. As an example, Grassie and Scott (1985) have stated that polymers undergo a variety of intrinsic degradation processes during thermolysis, including depolymerization, random scission, and side group elimination. Higgins (1990) has shown that polymers that initially decompose by side group elimination or by random scission processes produce appreciable amounts of involatile carbonaceous residue, whereas polymers that decompose via depolymerization show much "cleaner" delubrication properties.

A typical pressing agent that is used in powder metallurgy is ethylenbisstearamide (EBS, $C_{38}H_{76}N_2O_2$). This polymer consists of a dimer with two long-chain aliphatic hydrocarbons and two functional carboxylic groups, which are connected by an ethyl group. EBS decomposes at temperatures between 270 and 410 °C (Karamchedu et al., 2011). Proskrebyshev et al. (2007) state, that the decomposition of EBS shows a bi-modal time dependence, where a fast process was attributed to the formation of CO_2, and the formation of CO, CH_4 and C_2H_2 could be attributed to the decomposition of the secondary products of the EBS-decomposition. The analysis of the decomposition of ethylenebisstearamide in Fe/C-compacts reveals a competition between hydrolysis and *cis*-elemination via γ-H abstraction (Baum et al., 2004). As shown by Jellinek (1978), stearic acid is one of two decomposition products of hydrolysis, and for the further pyrolysis, Baum et al. (2004) suggest decomposition via four pathways. The most likely pathways involve either decarboxylation, resulting in heptadecane and CO_2, or dehydration of carboxylic acids to ketene. On the other hand, *N*-vinyl amides and stearamides are expected upon *cis*-elemination.

Although the single mechanisms of delubrication and sintering of PM aluminium are largely understood, the parameterization and layout of the continuous heat treatment has always been done empirically. Only little is known about the real composition of process atmospheres and their spatial distribution in continuous furnaces. Hence, in the present work the real-time developing composition of the process gas atmosphere in the dewaxing zone of a continuous aluminium belt furnace was studied with an in-situ Fourier-Transform-Infrared spectroscopy (FTIR) process gas analysis method.

2. Experimental

The initial characterization of the gas evolution of PM aluminium compacts was carried out in a quartz tube furnace in a pure N_2 atmosphere. The temperature was raised from room temperature up to 530 °C with a heating rate of 3 K/min, and the gas flow was 6 L/min. The observation of the gas composition during the process was carried out by FTIR spectroscopy, using a ThermoFisher Scientific Antaris IGS. The infrared beam from the broadband IR source first passed through a capsuled mirror system, then entered the tube furnace via KBr windows. On the opposite side of the tube, the signal again passed a KBr window and was finally detected by an external Mercury–Cadmium–Telluride photodetector. In order to identify single species, reference spectra (CO, CO_2, CH_4) were taken with the help of gas filled reference cells. Spectra were taken in temperature steps of 5 K.

Further measurements were taken using a Quantum Cascade Laser Measurement and Control System (Q-MACS, neoplas control GmbH, Germany), which operates with a narrow-band source in the mid infrared spectral range.

In order to characterize the gas composition in a belt furnace, the muffle of sinter belt furnace for the production of PM aluminum parts with height 120 mm and width 300 mm (Sarnes, Ostfildern, Germany) was equipped with four flanges and KBr windows on both sides within the dewaxing zone. As depicted in Fig. 1 the flanges were positioned at the inlet of the dewaxing zone, in the center at two height levels and at the end of the dewaxing zone at the transition to the sintering zone. The temperature profile in the furnace and the position of the flanges are shown in Fig. 2.

The analysis of the process gas composition was carried out as mentioned above with a FTIR spectroscope (see above). Analogous to the batch furnace the signal passed the furnace via the KBr windows and was detected by an external Mercury–Cadmium–Telluride photodetector on the opposite furnace side. Each spectrum was analysed with regard to the detected species. Every species exhibits a characteristic band, whose shape changes with the temperature due to Doppler broadening. The relative concentration of CH groups (which are the main decomposition products) was determined by drawing a baseline between 2990.61 and 2834.85 cm^{-1} and integrating the area between the baseline and the measured absorbance. This method may be defective to