

Evolved gas analysis of PEP-SET sand by TG and FTIR



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ABSTRACT

Thermogravimetric analysis coupled with Fourier transform infrared spectroscopy (TG-FTIR) and on-line FTIR were employed in this study, aiming at investigating the pyrolysis characteristic of Polybenzyl Ether Phenolic resin sand mould (PEP-SET sand) and emission gases generated during casting. The non-isothermal pyrolysis of PEP-SET sand can be subdivided into three stages: emission of inherent moisture (below 215 °C), thermal decomposition at low temperature (215–315 °C) and further cracking process at high temperature (315–432 °C). The main emission gaseous products in pyrolysis processes were H₂O, CO₂, NH₃, CH₄, C₂H₆, NO and a little amount of CO, according to the FTIR spectrum analyses. The amount of N-containing pyrolysis products generated from cracking of the C–N bond reflects the collapse extent of the binder. The thermal shock tests at different temperatures were performed. Similar to non-isothermal pyrolysis processes, the main gaseous products were CO₂, CO, NO, CH₄ and C₂H₆ under thermal shock conditions, and these gases released follow a fixed sequence: concentrations of CO, CO₂ and NO firstly increased to the peaks and then decreased with the rising of CH₄ and C₂H₆. At last the CO₂ concentration increased again. The major emission oxidizing gas which may induce ignition of Mg melt is CO₂ and the environmental hazardous components are NO and NH₃.

1. Introduction

As a kind of chemical binder, Polybenzyl Ether Phenolic (PEP) has been widely used in casting industry to make sand mould [1]. The resin sand mould made by this binder is called PEP-SET sand mould. The binder was a mixture of urethane/phenolic resin (0.6 wt.% PEP-SET 5230) and isocyanate (0.6 wt.% PEP-SET 5112) [2]. The sands blend with PEP binder are hardened by means of the condensation reaction between phenol-formaldehyde resin and polyisocyanate, which is catalyzed by tertiary amine [3]:

During casting processes, molten metal is poured into a mould with an interior of a desired shape. For castings with a hollow interior, sand cores are used. Generally, the components cast using clay-bonded or sodium-silicate sand mould cannot meet the demand of high dimensional accuracy and surface quality serviced in aeronautic and astronautic industries. The resin-bonded mould/cores, especially PEP-SET ones, are commonly employed in the casting processes of high quality Mg alloy components. After pouring molten metal into the mould, the sand mould/core surfaces are covered by liquid metal, numerous mixture emission gases generate as a result of the thermal decomposition of PEP binder. The interaction between Mg alloy melt and the thermal

decomposition products condensed in the mould will take place during the solidification of the casting, resulting in oxidation of casting surface, even ignition of the metal.

As without oxygen inside sand cores, the thermal decomposition of the polyurethane binder can be assumed to be done only by pyrolysis [4]. Many researchers have studied the emissions of various casting materials used in foundries, such as bituminous coal, cellulose, phenolic urethane binder (PUB), and collagen-based binder, for controlling environmental hazardous gases [5–8]. Dungan compared the pyrolysis products of three different resins using mass spectrometry and found that major thermal decomposition products from the Novolac, PU, and furan resins were derivatives of phenol, benzene, and furan, respectively [9]. Lytle identified that the main emission gases of pyrolysis of novolac resin and phenolic urethane resin were carbon monoxide, methane, ethane, and propene [10,11]. Tiedje suggested that the most important compounds emitted from all the three different binders: furfuryl alcohol (FA), phenolic urethane (PU) and resol-CO₂ (RC) were benzene and carbon monoxide [12]. Wang identified that most of the emissions from pyrolysis of furan no-bake sand moulds were released within the temperature range of 350–700 °C [13].

However, less analytical work has been done regarding the pyrolysis

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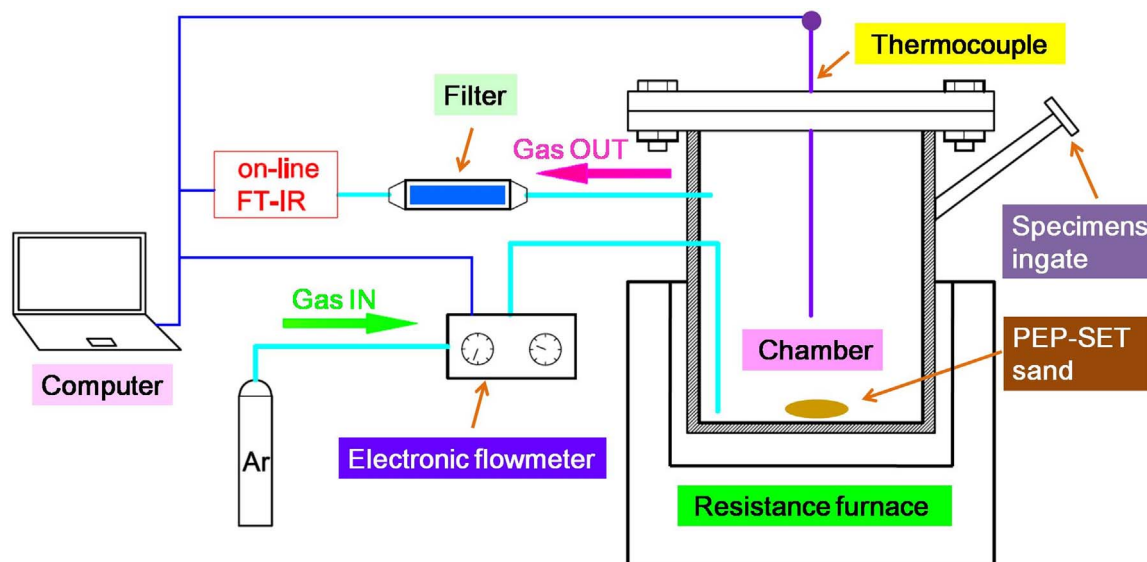


Fig. 1. A schematic diagram of the experimental set-up for thermal shock test.

of PEP-SET sand in the production of magnesium alloy castings, which releases gases harmful to workers, reacting with magnesium melt and causing ignition occasionally.

According to the heating conditions, the sand mould can be approximately divided into two regions. The surfaces of mould and cores directly contacting with melt are expected to rise to a high temperature (near pouring temperature) in several seconds, and the heating conditions of these regions are similar with thermal shock. However, the temperature of regions below the surface of mould and cores gradually increase as a result of heat conduction. The heating process of these regions can be identified as non-isothermal pyrolysis with a relatively low heating rate. Different methods were employed to investigate the two kinds of pyrolysis processes. Thermogravimetry (TG) is a commonly used method to analyze the pyrolysis kinetics and products [14–17]. TG coupled with Fourier Transform infrared spectroscopy (FTIR) was employed in this study to establish the corresponding relationship between weight loss of PEP-SET sand and the main components of its products in the process of non-isothermal pyrolysis. The variations of emission gases of PEP-SET sand under different thermal shock conditions were identified by on-line FTIR in order to investigate the gas released from the surfaces of mould cavity. The results obtained in this study are useful to comprehensively understand the thermal decomposition process of PEP-SET sand mould, and provide data for further investigating of hazardous gases control and analyzing the ignition mechanism of Mg alloy casting by PEP-SET sand mould.

2. Materials and method

2.1. Materials

The PEP-SET sand used in this study was prepared following the foundry factory techniques. A reaction mixture was made of phenol-formaldehyde resin and polyisocyanate with equal weight. Two kinds of PEP-SET sand specimens were prepared for TG-FTIR slow pyrolysis and on-line FTIR thermal shock respectively. In order to get enough emission gas during TG-FTIR experiments, the total weight of binder in the specimens was 10% of the sand, which was higher than the requirement in foundry practices. After intensive blending and hardening, the PEP-SET sand mould was ground to individual sand particles with weight about 3 ~ 5 mg. For the purpose of simulating the mould cavity environment as accurate as possible, the PEP-SET sand specimens for on-line FTIR thermal shock test were prepared with 1.5 wt% binder addition of the total weight, similar to the actual foundry practice. The

specimens were manufactured into cylinders with dimensions of $\Phi 10 \times 50$ mm.

2.2. TG-FTIR analysis

Thermogravimetric Analysis (TGA/SDTA851, Mettler Toledo) coupled with a FTIR spectrophotometer (Nicolet5700, OMNIC) was employed to investigate the mass loss and evolution of gaseous products during the pyrolysis of PEP-SET sand. Stainless steel transfer pipe and gas cell used to connect TG and FTIR were heated to 180 °C to avoid the condensation of some pyrolysis products. High purity N₂ gas was used as carrier gas. Passing N₂ for 30 min until the background baseline was corrected by FTIR, and then the specimen was inserted to the TG reaction chamber. For the purpose of keeping the analysis of emission gas by FTIR simultaneous with the mass loss detected by TG, a relatively large N₂ flow rate of 100 ml min⁻¹ was chosen. For each test, around 45 mg PEP-SET sand was used. The specimens were heated from 50 °C to 800 °C at different heating rates of 10, 20 and 50 °C min⁻¹, respectively, and then kept at 800 °C for 5 min. The analysis of FTIR started from 80 °C and spectrum scanned every 16s. The spectral region was set at 4000–650 cm⁻¹.

2.3. FTIR on-line analysis

An apparatus was built to connect with the on-line FTIR (BCT2-GASMET FTIR Dx4000), and the schematic diagram of experimental set-up is shown in Fig. 1. A steel reaction chamber sealed with rubber gasket and screws was inserted into a resistance furnace. A tube placed on the chamber was designed as Ar gas inlet, and the flow of Ar can be adjusted through an electronic flowmeter controlled by a computer. The evolved gas flowed out from the chamber and get into the on-line FTIR through a filter with desiccant. Both temperatures in reaction chamber and the analysis results of on-line FTIR were monitored by a computer.

Before on-line detection, an Ar gas flow with a rate of 2L min⁻¹ was employed to eliminate air in the system and lasted for about 30 min until the pure background for FTIR test was obtained, followed by a decreased Ar gas flow rate of 1L min⁻¹, and then the resistance furnace started heating. PEP-SET sand specimens were quickly put into reaction chamber through the ingate when the chamber had reached desired temperature and on-line FTIR was ready for analysis. The emissions of PEP-SET sand at thermal shock temperatures of 500 °C, 600 °C, and 700 °C were investigated, respectively.

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