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The behavior of gypsum-bonded investment in the gold jewelry casting process

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ABSTRACT

Gypsum-bonded investment, which is commonly used in gold casting, can cause several defects such as gas porosity from the decomposition of gypsum. Thus, the insight study in the behavior of gypsum-bonded investment can lead to resolve the problem in casting process. It consists of SiO_2 (α-cristobalite and α-quartz) as a refractory and gypsum as a binder. During burn out process, the dehydration of gypsum, the inversion of cristobalite and quartz and the III–II $CaSO_4$ transition were determined using differential scanning calorimetry (DSC) coupled with thermogravimetry analyzer (TGA) and in-situ X-ray diffraction (in-situ XRD). Calcium carbonate ($CaCO_3$) layer on the gold product surface after casting was investigated by X-ray photoelectron spectroscopy (XPS). It is believed to be attributed to the reaction between CO_2 from residues of carbonized wax and CaO from the decomposition of $CaSO_4$. This observation is may be responsible for high roughness and low quality in the gold casting.

1. Introduction

Investment casting or lost wax casting provides high precision of product details and superior surface quality of the products. Hence, this casting process is suitable for complex shape products in jewelry industry. For the investment casting of gold and silver, the jewelry manufacturers typically prefer to use gypsum-bonded investment as its thermal properties are appropriate for commercial-grade alloys casting [1–6] and this type of investment has lower price compared to other types of investments. Regarding investment casting process, there are several sensitive parameters that must be considered to achieve supurior casting product quality. These include types of alloys and waxes, type and quality of investment powder [7–10], and the reaction between investment mold and molten metal during casting [11–13].

Traditional gypsum-bonded investment powder consists of a mixture of calcium sulfate hemihydrate ($CaSO_4{\cdot}0.5(H_2O)$) binder and SiO_2 (α-quartz and α-cristobalite) refractory [1,7,14,15]. To prepare mold for investment casting, gypsum-bonded powder must be mixed with water until it become slurry. The slurry will then be invested and set to form a green mold. During setting, calcium sulfate hemihydrate ($CaSO_4{\cdot}0.5(H_2O)$) is transformed to calcium sulfate dihydrate ($CaSO_4{\cdot}2(H_2O)$) giving moderate strength to the green mold. This mold is then heated to burnout internal wax and strengthen the ceramic mold. In the process of heating and burnout, $CaSO_4{\cdot}2(H_2O)$ crystal lost its water and becomes anhydrate ($CaSO_4$) at temperature above 140 °C, then at temperature around 250 °C α-cristobalite transforms to β-cristobalite and at temperature 572 °C α-quartz transforms to β-quartz [1]. Further heating to temperature around 900 °C to 1260 °C, calcium sulfate decomposes into calcium oxide and sulfur dioxide gas [16]. This thermal decomposition could result in the reduction of mold strength. Unfortunately, the period of decomposition temperature is in the same range as casting temperature of gold alloy and thus heating profile must be controlled carefully [17]. Upon heating the formation of calcium silicate and sulfur trioxide at temperature about 1000 °C could also occur according to the following reaction [15]:

$$2CaSO_{4(s)} + SiO_{2(g)} \rightarrow Ca_2SiO_{4(s)} + 2SO_{3(g)}$$

The resulting sulfur-containing gases (i.e. SO_3 or SO_2) could be trapped in the molten metal during solidification causing porosity defects which appear as shiny round pores in the final casting products [1]. In

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addition, residue wax from incomplete burnout could react with calcium sulfate producing gas porosity close to the surface of jewelry products [18,19].

This study aims to gain better insights into the thermal behavior of gypsum-bonded investment, the reaction in casting process and the difference between prior and post polishing surfaces of the jewelry casting products. To achieve better understanding, several advanced characterization techniques such as Differential Scanning Calorimetry (DSC) coupled with Thermogravimetry Analyzer (TGA), In-situ X-ray Diffraction (In-situ XRD), Field Emission Scanning Electron Microscope (FE-SEM), X-ray Photoelectron Spectroscopy (XPS) were used to investigate the behavior of investment from the beginning to the end of investment casting process. These better insights in the investment material will be beneficial to the jewelry manufacturers for the development of burnout cycle and casting process in the jewelry production. For example, the local factory can reduce the burnout time that will increase the productivity and may improve the rate of product rejection due to sub-standard quality which is found to be 2–5% in the local factory.

2. Experimental procedures

The chemical composition of commercial gypsum-bonded investment powder (G-CAST APEX, Gesswein Siam Co., Ltd., Thailand) and commercial 18 K yellow gold alloy were analyzed using X-ray Fluorescence (XRF). The phases and compositions of the investment powder were also analyzed using X-ray diffractometer (XRD) (PANalytical, EMPYREAN, The Netherlands). The investment powder was characterized at ambient temperature and the diffraction angles data (2θ) were collected between 10°–90°. The data-collecting step was set to 0.1° while the period of counting was 40 s per step. The Rietveld refinement of the resulting XRD pattern was performed using HighScore Plus software. Furthermore, the particle size distribution, morphology and chemical distribution of the powder were measured using Particle Analyzer (Mastersizer-2000, Malvern Instruments Limited) and Field Emission Scanning Electron Microscope (JSM-7001F,JEOL) equipped with Energy Dispersive X-Ray Spectrometer (EDS) (INCA PentaFETx3, Oxford) respectively.

Next, to prepare investment slurry, fresh investment powder was mixed with water following the recommendation from the powder manufacturer which has a water/powder ratio of 38 ml water per 100 g powder. After mixing for three minutes, the slurry was poured into alumina crucible pan to prepare a green part sample for thermal analysis. The thermal characteristics of the green part were studied using differential scanning calorimetry coupled with thermogravimetry analyzer (Netzsch STA449 F3 Jupiter thermal analyzer, Germany). The green part was then heated up following burnout cycle given by the powder manufacturer (as shown in Fig. 1). The heating profile can be summarized as follows: (1) heating from room temperature to 150 °C at a rate of 2.5 °C/min, (2) isothermal holding for 2.5 h, (3) heating up to 375 °C at an increased rate of 4 °C/min, (4) isothermal holding for an hour, (5) heating up to 725 °C at a rate of 3 °C/min, (6) isothermal holding for 4 h, (7) cooling down to casting temperature at 600 °C with 2 °C/min, (8) final isothermal holding for an hour. During heating, oxygen gas feeding was set to the flow rate of 50 ml/min. Subsequently, the microstructure of burned-out investment was investigated by SEM-EDS and compared with that of the raw investment powder.

The structural change of the investment during burnout cycle process is the key to control both physical and mechanical properties of the mold. Therefore, in-situ XRD experiment was conducted to capture any change in structure of the investment mold starting from the green mold through the end of burnout process. In this experiment, during heating, the measurements were done at room temperature, 70 °C, 150 °C, 250 °C, 375 °C, 580 °C, 725 °C and cooled down to 600 °C respectively. Note that these measured temperatures were selected based on typical burnout cycle of jewelry investment and the reaction temperatures observed from DSC/TG experiment.

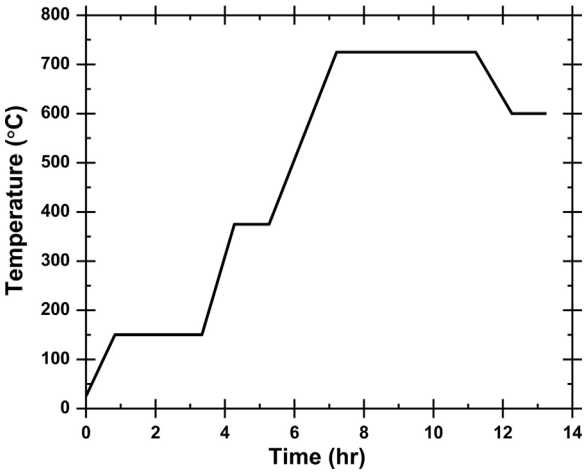

Fig. 1. Burnout cycle of the gypsum-bonded investment.

Finally, two gold alloy samples with 75% purity (18K-gold) were cast by a commercial jewelry casting process. The chemical composition of the gold alloy is 75.1%Au, 12.3%Ag and 12.6%Cu. In this study, the investment flask with a size of 3"x4" was used for cast the two gold samples in centrifugal casting machine. Induction heating was used to melt the gold alloy and the centrifugal casting was done under Argon gas atmosphere. The casting temperature and flask temperature were set to 980 °C and 600 °C, respectively. After casting, the first gold sample was kept un-polished and the second one was polished following the typical procedure of jewelry industry. The X-ray photoelectron spectroscopy was then performed on these two samples to characterize surface reaction between molten metal and the investment mold during casting as well as to study the effect of polishing process on the reaction layer.

3. Results and discussion

3.1. The morphology and chemical compositions of the investment powder

The X-ray diffraction pattern of the commercial investment powder is shown in Fig. 2. The result shows that the powder contains hexagonal quartz, tetragonal cristobalite and gypsum hemihydrate ($CaSO_4{\cdot}0.5(H_2O)$). The content of each phase was quantitatively determined by Rietveld refinement method. The result reveals that the powder consist of 34.26%wt. quartz (ICDD No. 01-070-7345), 33.85% wt. cristobalite (ICDD No. 01-077-8627), and 31.89%wt.

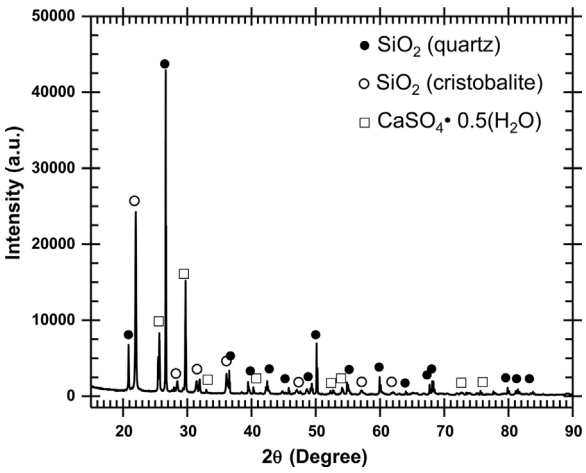

Fig. 2. XRD pattern of gypsum-bonded investment powder.