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PA 12 NANOCOMPOSITES AND FLAME RETARDANT COMPOSITIONS PROCESSED THROUGH SELECTIVE LASER SINTERING

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INTRODUCTION

Additive manufacturing is raising an increasing interest. Among the different techniques available at industrial level, Selective Laser Sintering (SLS) allows very complex shapes to be processed but requires specific properties for the powders used to build the final objects (1). These characteristics makes this technique suitable for the production of prototypes or parts for various applications, especially aerospace and defense industries. However, these industries have some of the toughest performance standards and needs the production of parts with improved properties as flame retardancy.

Whereas the processing is well controlled for usual pristine polymers devoted to this technique such as PA11 and PA12 (2), there is still a deficit in basic process knowledge, especially in system containing additives. In particular, it may become critical for polymer blends or polymers containing micro- or nano-particles. SLS of polymers is faced with various challenges concerning suitable material systems, process strategies and part properties. A better understanding of the interactions between the additives and the processes of selective laser melting linked to the resulting part properties is necessary.

The aim of this work is to investigate PA12 particulate composites containing submicronic talcs and kaolinite as well as flame retardants and finally binary compositions of flame retardants and sumicronic mineral particles. Microstructure and fonctional properties of 3D printed specimens are studied and compared with similar compositions processed using injection moulding.

EXPERIMENTAL

Materials

Commercial powdered polyamide 12 (PA2200, EOS), ammonium polyphosphate flame retardant (Exolit® AP423-Clariant), kaolinite (Paralux–Imerys) and talc (HAR T84) were used as received.

Preparation

Polymer and fillers were added and mixed with the help of a powder mixeing station (EOS). Plaques of 80x80x4 mm where prepared using a SnowWhite equipment (Sharebot) with a powder temperature of 180 °C.

Characterizations

Differential Scanning Calorimetry (DSC) measurements were performed using a Perkin Elmer Pyris Diamond equipment. Sample masses were of 12.0 ± 0.5 mg and were heated from 50 °C to 250 °C at a rate of 10 °C.min^{-1}, followed by a cooling ramp at 10 °C.min^{-1} from 250 °C to 50 °C. Cone calorimeter experiments were carried out using a Fire Testing Technology apparatus with an irradiance of 35 kW.m^{-2} according to ISO 5660 standard. Particle coalescence of the powders was investigated using a Laborlux 11POLS microscope, set in visual transmission mode connected to a Leica DFC420 camera and a Linkam Microvision temperature controlled stage, which enabled the observation of the evolution of particles coalescence with an increase in temperature.

RESULTS AND DISCUSSION

The SLS process needs certain requirements that the material has to fulfil. With selective laser sintering, melt and powder both exist over the entire period of building. To achieve this two-phase mixture area, the building chamber is pre-heated to just below the melting point of the material (3). It is essential for the crystallization temperature of the blend to be clearly below than melting point. Fig.1a shows the influence of some components used in this study on the SLS processing window. A narrowing of temperature sintering range can be observed for all formulations containing flame retardants and mineral fillers which could prevent or limit the sintering of parts.

Addition of flame ratardants allowed the production of SLS parts with an improved flame retardancy to be achieved, leading to a reduction in peak of heat release a a function of filler type and loading, but at the expense of a shorter time to ignition (Fig. 1b).

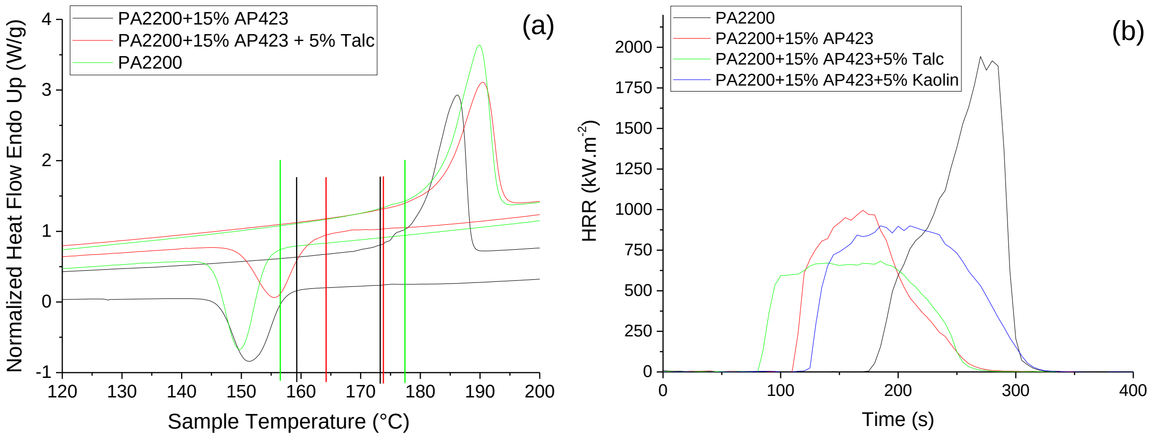

Fig .1 a) DSC results of some PA2200 composites; b) Cone calorimeter results.

Microscope analysis allowed observing that the addition of flame retardants and fillers had an important influence on coalescence of polymer particles, leanding to a decrease in coalescence velocity. Even so, it appears that for all fillers and flame retardants SLS process could be performed. Nevertheless, powder rheology as well as thermal stability and ability for the mixed powers to coalesce have to be taken into account to achieve cohesive parts (4). Finally, only few combinations of submicronic particles and flame retardants could be proposed for flame retarded SLS parts.

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