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# SELECTIVE LASER SINTERING OF POLYAMIDE 12/FLAME RETARDANT COMPOSITIONS

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## INTRODUCTION

Additive manufacturing is a relatively new technology raising an increasing interest. Among the different techniques available, Selective Laser Sintering (SLS) is receiving much attention in literature due to its great flexibility (1). SLS has demonstrated substantial potential for the production of parts for various applications, especially aerospace and defense industries. However, for these applications, parts should exhibit specific properties and high level of flame retardancy. To achieve this objective, relevant additives must be incorporated into SLS powder formulations. Still, the addition of these particles could induce changes in polymer properties, especially melt and crystallization temperatures and powder rheology. All these variations in powder characteristics can lead to parts with functional properties, such as fire resistance, not suitable for technical applications. However, few studies concerns the addition of nanoparticles to SLS powder formulations which are dedicated to evaluate their influence on the mechanical and electromagnetic properties of SLS parts (2,3).

Furthermore, it should be pointed out that, compared to injection moulding, SLS parts generally show a decrease in mechanical properties compared to traditional processes (extrusion/injection). Concerning flame retardancy, at the best of our knowledge, there is no study in the literature that concerns the influence of flame retardants on sintering of powders.

Thus, to evaluate the potential properties of commercial polyamide 12 (PA12) used in the SLS process, different flame retardants and fillers were dispersed in the polyamide powder by mechanical mixing. Specimens of composite were manufactured by the SLS process and characterized by differential scanning calorimetry, thermogravimetric analysis, cone calorimeter and scanning microscope observations. Finally, 3D printed specimens are compared with similar compositions processed using injection moulding.

## EXPERIMENTAL

### *Materials*

Commercial powdered polyamide 12 (PA2200, EOS), ammonium polyphosphate (Exolit® AP422 and AP423-Clariant), phosphinate (Exolit® OP950), melamine polyphosphate (Melapur® 200 Basf), cyanurate melamine (Melapur® MC50-Basf) and Aflammit® PCO900 (Thor) flame retardants were used as received.

### *Preparation*

Polymer and flame retardants were added and mixed with the help of a powder mixing station (EOS). Plaques of 80x80x4 mm were 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  $12.0 \pm 0.5$  mg and were heated from 50 °C to 250 °C at a rate of 10 °C.min<sup>-1</sup>, followed by a cooling ramp at

10 °C.min<sup>-1</sup> 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<sup>-2</sup> 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

In SLS, 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 some formulations containing flame retardants, which could prevent or limit the sintering of parts.

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

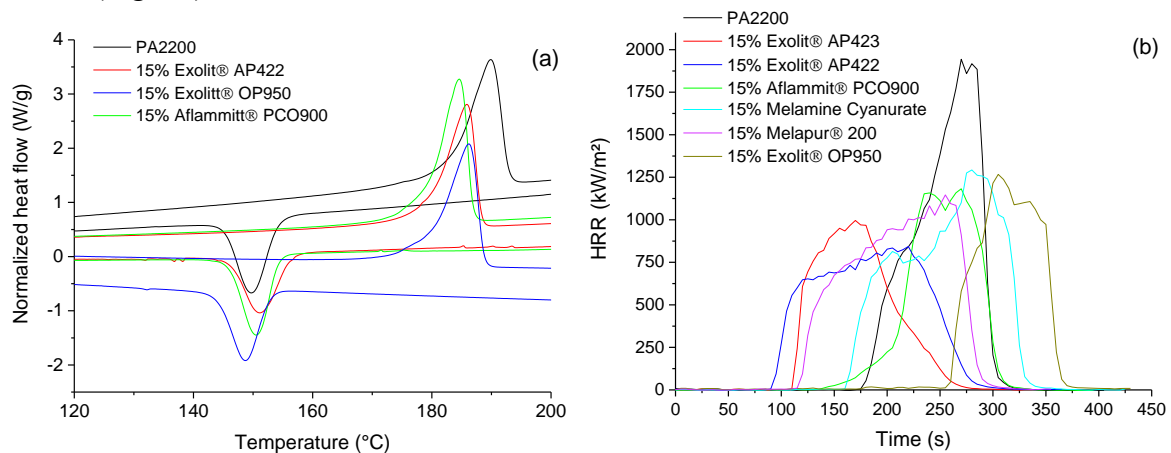


Fig .1 a) DSC results of some PA2200 composites; b) Cone calorimeter results.

Microscope analysis allowed to observe that the addition of flame retardants has an important influence on coalescence of polymer particles, leading to a decrease on the coalescence velocity. Nevertheless, powder rheology as well as thermal stability and ability for the mixed powders to coalesce have to be taken into account to achieve cohesive parts (4). Finally, only a part of the flame retardants systems investigated could be proposed for flame retarded SLS parts.

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## References

- 1) I. Gibson, D. Shi. *Rapid Prototyping Journal*, **4**, 129 (1997)
- 2) S. Yuan, Y. Zheng, C.K. Chua, Q. Yan, K. Zhou. *Composites Part A: Applied Science and Manufacturing*, **105**, 203 (2018)
- 3) D. Drummer, D. Rietzel, F. Kühnlein. *Physics Procedia*, **5**, 533 (2010)
- 4) D.L. Borell, T.J. Watt, D.K. Leigh, B. Fulcher. *Physics Procedia*, **56**, 147 (2014)