Report on the STSM of D. GROSSIN on University of Birmingham 1st to 30Th September 2014

Purpose of the STSM

This STSM [1] is related to WG2 "Manufacturing and characterization of 3D-porous scaffolds" (in charge of tasks 4: "Process Machining development" and 6: "Characterization of parts").
Hydroxyapatite (HAP) is currently used for a large number of medical applications. One of the drawbacks of HAP processing is that it decomposes at high temperatures. In order to process and manufacture HAP coatings for medical implants thermal decomposition needs to be controlled. Substituted hydroxyapatites have been studied since 1969, in CIRIMAT at INPT and one important outcome from this research is the development of thermally stable hydroxyapatites that are promising materials for 3D laser printing (e.g., chlorapatite).The main aim of this STSM is to study the elaboration of 3D-porous thermally stable substituted hydroxyapatite and non-substituted hydroxyapatite scaffolds by 3D laser sintering and their systematic microstructural characterisation. Optimized powders will be elaborated before and during this STSM, and the 3D laser printing facility located at the School of Metallurgy and Material in the University of Birmingham will be used. Visiting of other COST members or web conferences can be scheduled, in order to create standard characterisation procedures and tools for COST members.
Benefits for applicant: Access to industrial 3D printing device and valorisation of produced substituted hydroxyapatites.

Description of the work carried out during the STSM

<u>Plan of the work to be carried out :</u>
Task A: Preliminary experiments of ceramic 3D laser printing to setup equipment parameters.
The interaction between powder materials and laser using different powder and processing parameters will be studied and the following will be evaluated: (i) spread out behaviour of raw materials, (ii) obtained materials, (iii) dimensional 3D accuracy. Optimisation of 3D laser printing parameters will be performed. (3 weeks)

Task B: Evaluation of samples produced:
(i) Physico-chemical and structural characterisation: three-dimensional distribution of phases obtained using the micro-CT at the University of Birmingham (1 week)
(ii) Macro/Microstructural characterisation: determined at various scales from nano to macro. This work will be completed at INPT. Some XRD can be performed at the University of Birmingham but time restrictions will impose continuation of the project at INPT.

[1] Short Term Scientific Mission of COST action MP1301 : NEWGEN (1st Call)

Description of the main results obtained

3D-printing of SLS/M was not realized due to technical difficulties (Argon) but will be performed as soon as possible when the device capability will be restored. Physical characterizations are intended to evaluate the ability of the powder to be implemented for 3D-printing by SLS/M specially powder flowability. In addition, we rely on the size and the observation of the morphology of the particles. The flowability, morphological, and laser interaction characteristics were performed during this study.

- Flowability measurements were carried out using (i) a special funnel as recommended by the European Pharmacopeia, for the determination of flow times and (ii) Gioia method "flowdex" device for the determination of "powder viscosity". These results showed low flowability of the substituted-HA compare to HA.
- Morphological and microstructural studies were performed using (i) SEM and image analysis (ii) Aerosol laser granulometer in dry condition. It appears that the finer particles agglomerate around the larger which could prevent the powder to flow easily through the funnel. This is partly attributed to the use of the air jet mill, which generated a large amount of fine particles in very short time.
- Laser interaction, the transmittance of the NIR radiation (sample absorbed of radiation) of each powder at wave length of SLS/M were estimated by extrapolation. For all powders, weak absorptions were estimated. In this case, fine control of SLS/M parameters is required in order to create 3D-materials. The increase of absorption will be studied during a different mobility activity funded by JECT-trust, and will be focused on the co-substituted hydroxyapatite, cationic substitution of calcium. These substitutions have been studied mainly for optical properties.

Future collaboration with the host institution

This study is a part of an ongoing research collaboration that will set the basis to seek further national funding in UK and France to continue the project. We have already obtained specific support in order to facilitate research between INPT and University of Birmingham:

(i) After this STSM activity, Dr Grossin will complete a new mobility (funded by a French sponsor) from 6th OCT to 31st OCT 2014. This work is on a similar topic and optimized hydroxyapatite (HA), Fluoroapatite (FA) and Chlorapatite (ClA) powders will be produced.

(ii) From the 10th January to 28th of Februry 2015 (JECS-trust support), Dr Grossin will work in UoB on co-substituted powder materials (e.g. cationic substitution of calcium in ClA, FA, HA).

(iii) In June 2015, the 2-year European-Australian project "NEXT-3D" [2] will start (Marie Skłodowska-Curie Action). This project is a staff exchange program between Australian/European companies or laboratories on the field of 3D-printing. In this project the main European beneficiaries are the UoB and INPT.

Confirmation by the host institution of the successful execution of the STSM

See Birmingham's letter.

[2] "Next generation of 3D multifunctional Materials and coatings for biomedical applications" call H2020-MSCA-RISE-2014