

Extract from the annual report 2018
To the website: www.ist.fraunhofer.de/en.html

CHARACTERIZATION OF COATED POLYMER IMPLANT STRUCTURES

3D-printed porous polymer framework structures, so-called scaffolds, present an interesting new approach for the treatment of missing bone fragments. For the ideal ingrowth of new bone cells, chemical modification of the polymer surface is necessary. This can be performed through, for example, coating with an atmospheric-pressure plasma jet in which layer-forming precursors with the desired functional chemical groups are utilized. In order to optimize the deposition processes and to identify important influencing variables it is necessary to measure the chemical groups. Within the framework of a project funded by the European Union, the Fraunhofer IST has therefore been investigating methods for the characterization of functional coated polymers.

The approach

Nucleophilic groups such as amines and imines react selectively with 4-trifluoromethyl benzaldehyde (TFBA). This can be exploited in order to determine their density on surfaces. For this purpose, the samples to be measured are first exposed to TFBA vapors and subsequently ventilated (see Figure 2). The quantity of remaining TFBA molecules per area can be analyzed by means of various spectroscopic methods such as Fourier transform infrared spectroscopy with attenuated total reflection (ATR-FTIR), X-ray photoelectron spectroscopy (XPS) or electron probe microanalysis (EPMA) and is a measure of the density of the nucleophilic groups. In the case of three-dimensional, porous substrates, the penetration depth of the coating and the homogeneity in the depth are also of great interest. For the characterization of the penetration depth of coatings with 3-aminopropyl trimethoxysilane (APTMS) in scaffolds, these were derivatized immediately following coating with TFBA and were subsequently sliced. Along the cut edge, both the layer thickness and the density of the groups were determined by means of EPMA.

The results

The investigations into APTMS coatings at the Fraunhofer IST have shown that scaffold structures with a thickness of 10 mm can be completely coated with a plasma jet without a significant decrease in layer thickness or group density (see Figure 1). Through higher process gas flows, higher layer thicknesses are achieved.

For the investigation of electrophilic MSA-VTMS layers (maleic anhydride and vinyltrimethoxysilane as plasma copolymer), the coated samples were stained with methylene blue solution. Via electrostatic interactions, this dye binds to the carboxyl groups of the MSA. The coating did not penetrate the scaffold completely. As shown in the diagram on the next page (below), the observed penetration depth was only 4 to 6 double layers of the scaffold filaments. The cause could be in the considerably lower vapor pressure of the MSA. Again, an increased process gas flow can also increase the penetration depth here.

1 Penetration of an additive manufactured structure.

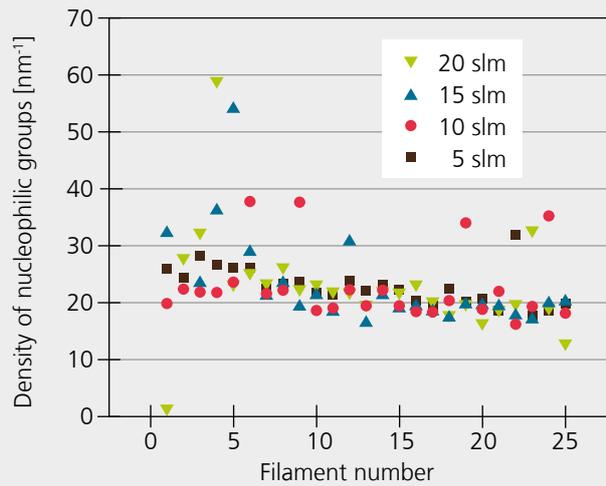
2 Experimental setup for TFBA derivatization.

Outlook

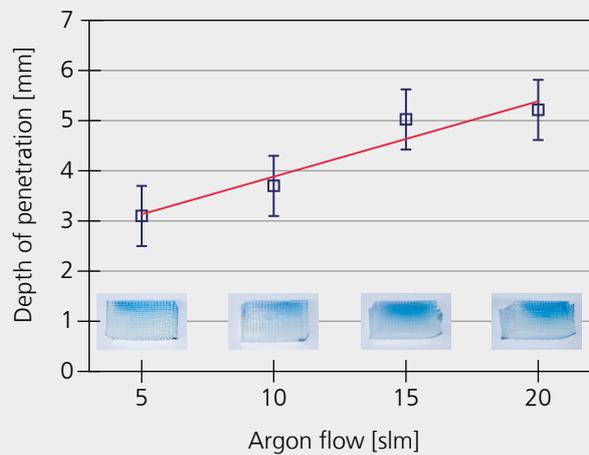
With the applied techniques, the coatings on the scaffold structures could be verified, together with their chemical activity. Both are important prerequisites for further optimization of the process parameters and for pursuing in-vitro and in-vivo investigations into cell growth.

The project

The project "Functionally graded Additive Manufacturing scaffolds by hybrid manufacturing", in short "FAST", was funded via the funding agreement No. 689925 from the European Union research and innovation program Horizon 2020. <http://project-fast.eu/en/home>



pp-APTMS: Layer thickness and group density in dependence on penetration depth.



Electrophilic MSA-VTMS layers dyed with methylene blue.

CONTACT

Dr. Thomas Neubert
Phone +49 531 2155-667
thomas.neubert@ist.fraunhofer.de