

Tailoring glass fiber surface for the polymerization and crystallization of anionic polyamide 6

Achraf Belkhiri, Olivier de Almeida, France Chabert, Nick Virgilio, Hélène Welemane, Valérie Nassiet

▶ To cite this version:

Achraf Belkhiri, Olivier de Almeida, France Chabert, Nick Virgilio, Hélène Welemane, et al.. Tailoring glass fiber surface for the polymerization and crystallization of anionic polyamide 6. International Conference on Manufacturing of Advanced Composites ICMAC 2021 (Virtual event), Oct 2021, London, United Kingdom. pp.0. hal-03565924

HAL Id: hal-03565924

https://hal.science/hal-03565924

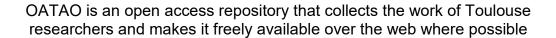
Submitted on 11 Feb 2022

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.



Open Archive Toulouse Archive Ouverte



This is an author's version published in: http://oatao.univ-toulouse.fr/ 28761

To cite this version:

Belkhiri, Achraf and De Almeida, Olivier and Chabert, France and Virgilio, Nick and Welemane, Hélène and Nassiet, Valérie Tailoring glass fiber surface for the polymerization and crystallization of anionic polyamide 6. (2021) In: International Conference on Manufacturing of Advanced Composites ICMAC 2021 (Virtual event), 20 October 2021 - 22 October 2021 (London, United Kingdom). (Unpublished)

Any correspondence concerning this service should be sent to the repository administrator: tech-oatao@listes-diff.inp-toulouse.fr

Tailoring glass fiber surface for the polymerization and crystallization of anionic polyamide 6

BELKHIRI Achraf¹, DE ALMEIDA Olivier², CHABERT France¹, VIRGILIO Nick³, WELEMANE Hélène¹ and NASSIET Valérie¹

1 : Laboratoire Génie de Production (LGP), Université de Toulouse, INP-ENIT, Tarbes, France
E-mail : achraf.belkhiri@enit.fr, france.chabert@enit.fr, helene.welemane@enit.fr , valerie.nassiet@enit.fr
2 : Institut Clément Ader, Université de Toulouse, CNRS UMR 5312, IMT Mines Albi, UPS, INSA, ISAE-SUPAERO, Campus Jarlard, 81013 Albi,
France

E-mail : <u>olivier.dealmeida@mines-albi.fr</u>
3: CREPEC, Department of Chemical Engineering, Polytechnique Montréal, Montréal, Canada <u>nick.virgilio@polymtl.ca</u>

Monomer polymerization after the impregnation of reinforcements is a relevant option for the manufacturing of thermoplastic composites by liquid processes (infusion or RTM). In the case of polyamide 6 synthesized by this process, the system composed of monomers, catalyst and activator reacts within the fibrous environment. Therefore, fiber surface chemistry is a crucial parameter because it does not only influence the polymerization and crystallization processes, but it also controls the fiber-matrix adhesion.

This project aims at adapting the surface chemistry of glass reinforcements so as to strengthen the interactions at the composite interfaces, but also in order to promote the polymerization and crystallization processes of anionic polyamide 6 (PA6). Indeed, the chemistry of surface reinforcement strongly affects polymerization kinetics and therefore final properties of glass fibers / PA6 composites. This work was carried out with glass microbeads comparable in size to the diameter of glass fibers.

First, the glass particulates must be adequately calcinated since the reaction catalyst is highly sensitive to moisture and surface-bound hydroxyl groups. As a result, the evolution of the hydroxyl groups surface was monitored as a function of calcination time by thermogravimetric analysis (TGA) and Fourier transform infrared spectroscopy (FTIR). The hydroxyl groups surface density was then carefully controlled in order to enhance the synthesis of anionic PA6. Then, a silane coupling agent was grafted on the glass particulates to improve interfacial adhesion and to promote even further the polymerization and crystallization of PA6. The grafting process was assessed by TGA and FTIR. Differential scanning calorimetry (DSC) analyses demonstrate that the competition between re-hydroxylation and condensation of the silane on the glass surface during the grafting process needs to be carefully balanced in order to maintain fast polymerization kinetics.

Once the silane grafting protocol was optimized, different silane-based coupling agents were compared. Three types of interactions at the particulate-matrix interface were considered. First, the glass surface was treated with a hydrophobic silane minimizing the interactions at the interface. Second, an amino-silane leading to the formation of weak bonds (hydrogen bonds) between the glass surface and the matrix was investigated. Finally, the glass beads were also treated with a silane end-functionalized with the reaction activator, allowing the formation of covalent bonds between the particulates surface and the matrix.

After treatment, the glass particulates surfaces were characterized using contact angle measurements, TGA, FTIR, X-ray photoelectron spectroscopy (XPS) and confocal microscopy. The results confirm successfully silane grafting onto the glass surface. Then, glass particulates/anionic PA6 composites synthesis was investigated by DSC to examine the influence of the chemical nature of the treatment on the polymerization time and the degree of crystallinity. The DSC results, combined with the surface characterization results, allowed to determine the effect of the surface treatment on the polymerization and crystallization, and thus to optimize the synthesis process.

Next, the fiber-matrix interfacial adhesion will be evaluated by means of tensile tests on composite specimens, allowing to quantify the influences of the grafting process and type of silane on the resulting macroscopic mechanical properties. Conclusions will help in getting an optimal interaction at the reinforcement / matrix interface, and in improving the design of composite fiber reinforced polymers.