Beamline: BM28

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Detailed mapping of the localized crystalline structure in rapid-formed automotive thermoplastic composite laminates

INTRODUCTION

Thermoplastic composites (TPCs) such as glass fibre reinforced polyamide laminates offer a unique material solution for low-carbon mobility applications, enabling high-volume manufacture of inherently sustainable (recyclable) light-weight components. High-performance TPC parts are produced via a number of techniques including rapid stamp-forming and bladder inflation processes. This yields a complex, transient (non-isothermal) crystallisation environment leading to a distinctly anisotropic crystalline structure in the part. Such variations in structure inevitably lead to local variations in material properties; mapping this inhomogeneity is critical to predicting performance. Mapping these variations can reveal the local processing history of the material and is critical for the development of accurate process simulation tools. Here, we used microfocus WAXS to obtain a map of the crystalline morphology of an industrially-processed polyamide (PA66) braided laminate material with glass fibre (PA66-GF). The material is a comingled polyamide glass braided structure which was consolidated using a recently developed rapid bladder forming process [1]. For the measurements presented here a single laminate thickness was consolidated which enables localised mapping of the polymer morphology around fibres.

EXPERIMENTAL

X-ray mapping measurements were performed on BM28 using a wavelength of 0.89 Å and 10 μm microfocus beam. Samples were positioned vertically in the beam and moved on the X-Y translation table with mapping in steps of 50 μm over scan length of 2.4 mm (i.e. 48 discrete measurements). 2D WAXS patterns at each step, were obtained for 60 s using the Pilatus 1M detector, positioned at a distance of 250 mm from the sample. A microscope was positioned at the sample position to correlate the beam position with the sample. Figure 1 shows the sample set-up on the beamline and microscopy images of the scan start and end positions covering glass and polymer rich areas. It is worthwhile noting that, prior to manufacture, the glass rovings are co-mingled with PA fibres. During the bladder forming process, the PA flows to form a consolidated sheet product. Therefore, whilst some regions will have high glass content, it was expected PA would be observed throughout. The scan presented here began in a glass rich region and progressed into a polymer rich zone, before returning to a glass rich region.

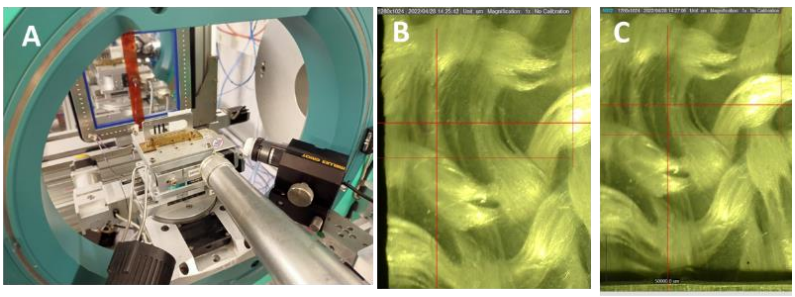

Figure 1. A: sample mounted on beamline. B: microscope image scan start position and C: scan end position.

RESULTS AND DISCUSSION

2D WAXS patterns were taken as the X-ray beam mapped across the sample. Figure 2A shows the two-dimensional WAXS of a complete scan across the glass matrix and a bundle of fibres (as depicted in Figure 1B and 1C). Here, it is clear that there are changes in the scattering patterns from the glass regions to polymer regions e.g. initially the first 4 frames, show more diffuse scattering indicting a region with high glass content. When the X-ray beam enters the polymer rich region the 2D WAXS patters start to show the PA crystalline structure. Figure 2B shows the change in integrated scattering intensity across the scan region of 0 – 1.0 mm, as the X-ray beam mapped the area. There are clear changes in intensity as the sample is mapped with the

polymer regions showing increased scattering intensity compared with the glass regions. To understand the variation in polymer and glass regions 1D WAXS patters were obtained by integrating over a 5° sector on the equator of the 2D WAXS patterns.

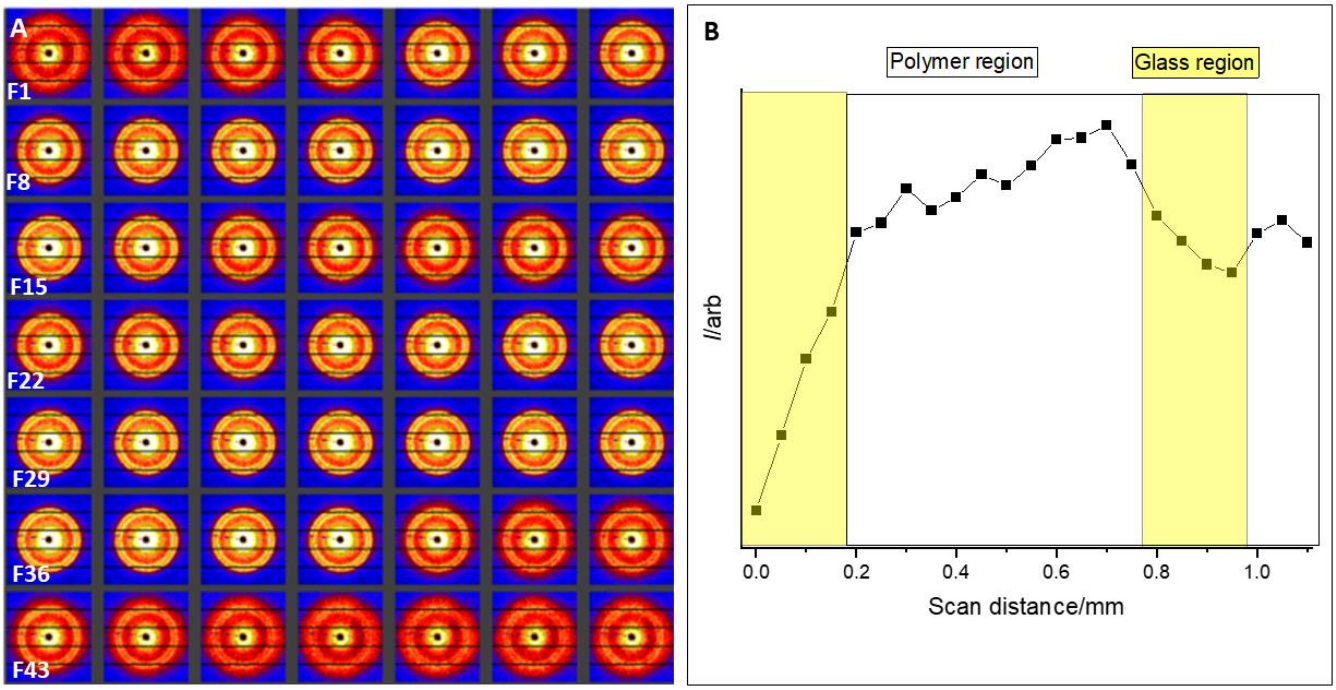

Figure 2. A: 2D WAXS of all frames of data through the scan. B: Change in integrated scattering intensity across a region of the mapping area between 0 and 1.0 mm.

Figure 3 shows the 1D WAXS data selected frames of data across the mapping area. The two prominent peaks in the 1D WAXS are the (100) and (010)/(110) of the PA66 stable α crystalline phase, which is formed on slow cooling. The 1D WAXS from the glass region (F1) shows that the peaks are evident but are broad and have low intensity indicating less well-developed crystallites. As the scan moves into the polymer rich region the peaks develop with increasing intensity and decreased width indicating well-formed and regular PA66 crystallites.

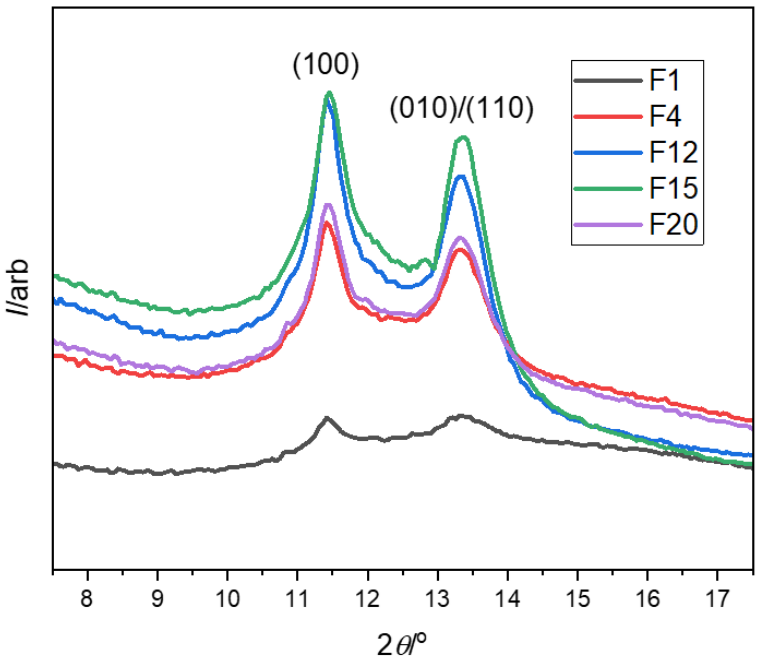

Figure 3. A: 1D patterns of selected frames through the mapped area.

CONCLUSIONS

The results show that good flow of the resin is achieved during the newly developed rapid moulding process. Despite the short processing time a polyamide α crystal structure arising from the controlled cooling is seen throughout the sample. This shows that despite high process TAKT time needed for automotive part production, a stable component can be produced. Further work is under way to establish if there are localised differences in morphology arising close to the fibre edges that may play a role in part quality.

References

[1] A. Singh, N. Reynolds, C.R. Carnegie, C. Micallef, E.M. Keating, J. Winnett, A.E. Barnett, S.K. Barbour, D.J. Hughes, A novel route for volume manufacturing of hollow braided composite beam structures, Advanced Manufacturing: Polymer & Composites Science 5(4) (2019) 224-229.