Acceleration of Monomer Transfer Moulding using Microwaves

Future Composites Manufacturing Research Hub Feasibility Study

Final report

UoN Personnel

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Collaborators:

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2) University of Edinburgh
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Period of project

01/03/18 – 30/09/18

Summary of project

Purpose:

To investigate the feasibility of using microwave heating to accelerate in-situ polymerisation sufficiently that monomer solutions could be used directly to make composite articles.

Scope:

1) Assess the feasibility of accelerating in-situ polymerisation of a glass fibre filled monomer solution.
2) In-situ Polymerisation Phase 1 – On Small scale (10's of grammes), compare the manufacturing parameters observed in such a process to define the potential benefits of using microwave heating.
3) In-situ Polymerisation Phase 2 – Scale the production of a microwave in-situ article to the kilogramme scale, to manufacture a proof of concept demonstrator panel using a commercially available microwave oven.

Industrial relevance:

1) Feasibility study on thermoplastic composites, to define polymer chemistry:
   a) Allow for interrogation of polymer matrix quality
   b) Allow for identification of influence of the glass fibre on the polymer matrix

2) Focus on next generation tooling, to define if microwave heating would allow:
   a) The development of "out of autoclave" processes,

b) The development of bespoke, low cost tooling, i.e. reduced need for in-mould heating strategies and reduced differential thermal profiles across mould.
c) The use of monomer should allow low pressure moulding, potentially prevent filler segregation and enhanced fibre/matrix interfaces.
d) Additive manufacture of "simple" moulds for the manufacture of bespoke components,
e) Rapid polymerisation/fast temperature cycling producing increased through-put.

Commercial opportunities:

1) Areas considered during Feasibility Study
   a) Medical Devices
   b) Automotive manufacture and Prototyping
   c) Fashion led construction articles e.g. furniture

2) Potential areas accessible beyond the Feasibility Study
   a) High performance automotive components (carbon fibre)
   b) Aerospace components

Hub priority areas:

1) High rate deposition and rapid processing technologies.
2) Inspection and in-process evaluation.

Hub identified Grand Challenges of Relevance:

1) Enhanced process robustness via understanding underpinning science
2) Develop high rate processing.
3) Diversification into emerging sectors by developing new technology

Key stakeholders:

1) University of Nottingham
2) University of Edinburgh
3) Sheffield AMRC
4) MX Group Ltd.

Project plan

Objectives

O1 - Establish benefits of using microwave heating for (a) glass fibre pre-preparation and (b) the resultant quality of matrix polymers produced when in contact with glass fibre.

O2 - Define benefits that are related to the preparation of composites from a monomer pre-solution rather than a polymer resin precursor system.

O3 - Produce small scale flat panel via microwave polymerisation (Instrumented domestic microwave).

O4 - Produce large scale flat panel via microwave polymerisation (Vötsch oven).

Deliverables/success criteria

D1 - Mould designed and built to account for requirements/restrictions of both Monomer Transfer Moulding (MTM) and Microwave Heating/Processing, taking into consideration any manufacturing limitations due to materials choice

D2 – Manufacturing parameters determined for successful small-scale component.

D3 – Manufacturing parameters determined for successful composite panel in Vötsch oven.

D4 – Demonstrate panel (D3) has equivalent/better properties to conventional MTM panel.

Progress against objectives and deliverables: See details in Appraisal of project achievements

O1 - Objective delivered

O2 - Objectives delivered

O3 - Objective partially delivered
Produce small scale flat panel via microwave polymerisation (Instrumented domestic microwave). Highlighted difficulties in mould construction with monomer.

O4 - Objective not attempted due to time limitation

Appraisal of project achievements

1) The project demonstrated that polymerisation of a composite part using electromagnetic (EM) heating is feasible and use of the EM heating gave manufacturing benefits of the small scale.
   a) Accelerated polymerisation of polycaprolactone (PCL) specimens was achieved by using EM heating both in unimodal and multimodal microwave cavities.
   b) Heating of the system was extremely rapid, indicating very good EM interaction demonstrating proof of principle.

2) E-glass fibres were determined to be effectively microwave transparent, both with and without sizing present. Dielectric calculations indicate a 6 cm penetration depth, potentially enabling direct heating through a 12 cm thick part.

3) The presence of the fibres in a conventionally processed reaction system was determined to have a retarding effect on the reaction, which was attributed to water on the fibre surfaces. This produced a matrix polymer of inferior quality.

4) EM heating was shown to deliver efficient in-situ drying of the glass fibre, in advance of monomer injection, this efficient removal of water from the glass surface resulted in a significant improvement in the matrix polymer.

5) Inclusion of glass fibres was shown to influence the temperature profile of the in-situ process that required further investigation and process design

6) Development of an integral mould that used monomer as the feed stock proved problematic. A microwave transparent mould was successfully developed, utilising a PTFE cavity with Nylon backing support and fittings but was not used on the larger scale.

Appraisal of project conclusions

1) The combination of glass drying and accelerated polymerisation of the glass/PCL components was successfully achieved using EM heating. This combination produced a matrix polymer that was close to the theoretically targeted molecular weight, thus these materials would be predicted to exhibit the desired material properties.

2) A lower conversion layer was observed on the outside of the parts when glass fibre was included in the system, when using EM heating. This was attributed to (a) heat loss at the edge of the part as the mould will now be the coldest area in the EM system and (b) conduction of the heat from centre to edge by the imbedded fibres. This was observed for the first time because of the small size of the part, thus the large surface to volume ratio exacerbated the effect.

3) If the mould was not correctly positioned in the multimodal cavity of the AMRC small instrumented microwave reactor, inhomogeneous heating in the sample was noted. This was attributed to the lack of a "mode stirring" capability leading to the development of hot spots in certain areas.

4) EM heating of the monomer solution was so rapid that the PID controller settings could not tuned the system with sufficient efficiency. Thus, the reaction temperature could not be maintained in as tight a window as would be preferred.

5) Initial trial panels for the planned production trials at the AMRC used vacuum infusion techniques to fill the mould. However this lead to two issues:
   a) The wetting the fibres caused poor filling of the bag thus a poor composite being produced. This is potentially an effect of an inappropriate size being present on the commercially supplied glass fibre.
   b) The loss of adhesion of the tacky tape due to the monomer behaving like an organic solvent to destroy the "tack" and resulting in the poor filling of the bag.

Key outcomes

1) EM heating is effective in accelerating the heating and polymerisation of ε-caprolactone monomer.

2) EM heating is effective in removing water from glass fibres and so produced a better matrix polymer.

3) EM heating can produce glass/PCL composite parts, but further work is needed to understand the effect of the following on the temperature profile through a mould:
   a) The presence of fibres
   b) The design of a multi-modal cavity to achieve EM field homogeneity.
   c) The choice of material of construction used for the mould to reduce heat loss to the mould.

4) Identified the need for pressure filling rather than vacuum if monomer based infusion is to be successfully achieved. This may influence mould design.

Overall Project Summary

1) The project completely achieved objective O1 and O2 and partially achieved O3
2) Has delivered deliverable D1 and D2.

Project Success

The project was considered to have achieved a Limited success overall.

Criterion definitions

Limited success – production of mould and polymer only specimen to show proof of principle

Complete success – production of composite panel using microwave processing that exhibits equivalent or superior properties to composite made via conventional MTM