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The first European benchmark exercise on squeeze flow testing of high-performance carbon fibre sheet moulding compounds

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Abstract. High-performance C-SMCs typically consist of long carbon fibre reinforcement with high fibre content and can be compression moulded at elevated temperatures and pressures to form parts with complex geometries. Squeeze flow testing has been increasingly adopted for experimentally characterising the flow behaviour of C-SMCs, but the reliability and repeatability

of the testing results are low due to the lack of standardised methods. This paper presents a benchmarking exercise on C-SMC squeeze flow testing, jointly delivered by 14 European research institutes, with the objective of quantifying the variabilities in the testing results and identifying their sources.

Background

High-performance carbon fibre sheet moulding compounds (C-SMCs) are a type of discontinuous fibre reinforced composite material characterised by long carbon fibre reinforcement (typically 1 inch / 25 mm roving length) and high fibre content (greater than 40 wt.-%). C-SMCs can be compression moulded under high pressure (typically greater than 100 bar) to form parts with complex geometries, offering great design flexibility in addition to superior mechanical properties. Understanding the flow behaviour of C-SMCs is key for accurately predicting manufacturing outcomes. This knowledge not only enables design for manufacturing but also allows for the prediction of process-induced variations in mechanical properties. Squeeze flow is a type of flow induced by compressing a material between two parallel mould surfaces. Squeeze flow testing is an experimental method for characterising the rheological and mechanical behaviour of a material utilising a squeeze flow regime. This method has been adopted by several research groups for characterising the flow behaviour of SMCs (Fig. 1) [1-5] for several reasons: firstly, compared to other flow characterisation techniques, squeeze flow testing is particularly advantageous for highly viscous materials experiencing high internal stresses during flow, as it can be easily implemented using a universal testing machine or a press; secondly, it allows testing of materials experiencing a high level of wall-slippage (although this causes complications in processing the experimental data) [1]; last but not least, compression moulding processes are largely dominated by squeeze flows. However, due to the lack of a standardised testing procedure, the reliability and repeatability of this testing method remain low, while the random nature of SMC materials introduces additional challenges in investigating the variabilities caused by the testing procedure. Furthermore, recent studies have demonstrated that the flow behaviour of high-performance C-SMCs differs considerably from that of traditional SMCs with glass fibre reinforcement fibres and low fibre content, which has been well-studied in the past [5]. This difference highlights the necessity for further investigation into high-performance C-SMCs. This paper presents a benchmarking exercise on the squeeze flow testing of high-performance C-SMC (referred to as C-SMC in the rest of the paper), jointly delivered by a collection of European research institutes with in-house developed squeeze flow testing facilities. The main objective of the benchmark exercise is to improve the reliability and repeatability of the testing through an understanding of the sources of variabilities in the measured data, and ultimately moving towards the development of a testing standard. It should be noted that the benchmark exercise is currently going through a data collection process, and no formal data analysis has been performed. Therefore, this paper will focus on presenting the methodology of the benchmark exercise.

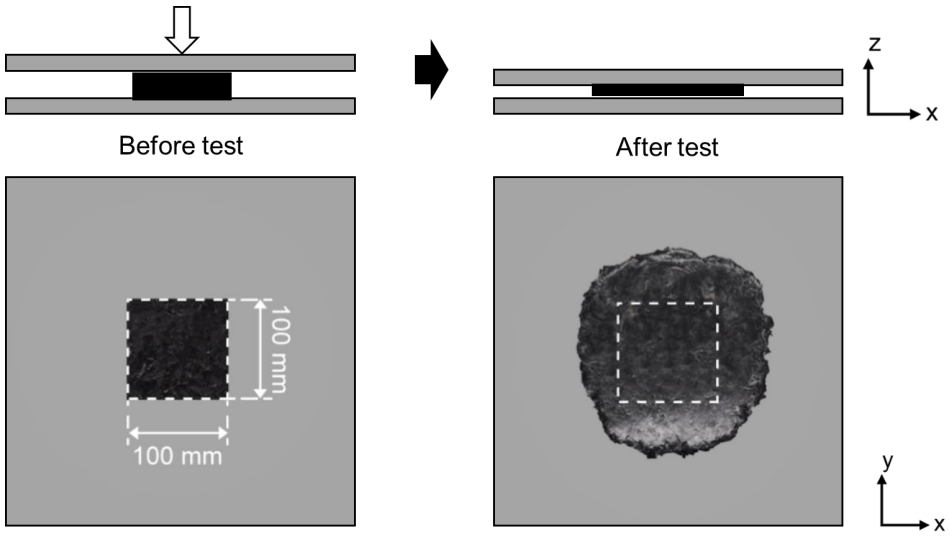

Figure 1 – Schematic illustration of the C-SMC squeeze flow test

Benchmark participants

The benchmark exercise involves 14 participating institutes across Europe, with a total of 10 squeeze flow testing facilities. Participating institutes include Ecolé Centrale de Nantes (ECN) and HESAM université (HESAM) in France, Leibniz-Institut für Verbundwerkstoffe (IVW) in Germany, Johannes Kepler University Linz (JKU) in Austria, Karlsruhe Institute of Technology (KIT) and Simutence GmbH (SIM) in Germany, KU Leuven (KUL) in Belgium, University of Twente (UTW) and ThermoPlastic composites Research Center (TPRC) in the Netherlands, RISE Research Institutes of Sweden AB (RISE) in Sweden, Technical University of Munich (TUM) in Germany, University of Bristol (UOB) in the United Kingdom, and Warwick Manufacturing Group (WMG) and University of Sheffield (UOS) in the United Kingdom.

Table 1 – Key parameters of the squeeze flow testing facility at participating institutes

Institute	Platen size [mm]*	Heating method	Force limit [kN]	Speed limit [mm/s]
ECN & HESAM	220	Heated platens	100	8.3
IVW	345	Heated platens	6500	5.0
JKU	Ø200	Heated platens	50	10.0
KIT & SIM	300	Heated platens	500	8.3
KUL	Ø280	Thermal chamber	250	10.0
RISE	Ø100	Heated platens	100	8.3
TUM	250	Thermal chamber	250	3.0
UOB	Ø200	Heated platens	100	8.3
UTW & TPRC	Ø246	Heated platens	40	5.0
WMG & UOS	350	Heated platens	250	10.0

*The value indicates the edge length of a square platen by default; where a Ø symbol is used, the value indicates the diameter of a circular platen

During the preparation stage of the benchmark exercise, each participating institute completed a participant questionnaire with details of their testing setup, and the information was subsequently used to determine the prescribed testing conditions of the benchmark. The information collected from the questionnaire was broadly related to the design of the testing fixture, the capacity of the testing machine, and the instrumentation used for measuring physical quantities. A summary of the key parameters of each participant's testing facility is provided in Table 1, as these parameters

were the most crucial when determining the testing procedure; the remaining information collected from the participants will not be provided in this paper due to limited space.

Materials

The materials tested in this benchmark exercise are commercially available vinyl ester (VE) C-SMCs supplied by Polynt Composites Germany, with five different specifications selected from Polynt's SMCarbon® 24 product series. The common parameters in all five materials are the resin recipe (resin code 24) and a fibre length of 25.4 mm (1 inch). The goal of this material selection is the evaluation of the influence of carbon fibre content (40 wt.-%, 50 wt.-%, 60 wt.-%) and fibre tow size (3K, 12K, 50K) on the required pressing forces and flow behaviour during processing. Details on the material specifications are summarised in Table 2.

Table 2 – Summary of the materials tested in the first European C-SMC squeeze flow benchmark exercise

Product code	Fibre tow size	Fibre length [mm]	Fibre weight fraction [%]	Target areal weight [g/m^2]
24 CF50-50K	50K	25.4	50	1800
24 CF50-12K	12K	25.4	50	1800
24 CF50-3K	3K	25.4	50	1800
24 CF40-12K	12K	25.4	40	1800
24 CF60-12K	12K	25.4	60	1800

All C-SMC materials have a nominal cure time of 35 seconds per millimetre part thickness at a recommended mould temperature of 125-140°C [6-10]. The resin system is designed to be processable for a period of 12 weeks at room temperature (up to 23°C), or 6 months at -18°C [6-10]. The benchmark exercise has adopted a room-temperature transport and storage approach. All materials were first shipped to IVW in the 1st week after the manufacturing date. IVW subsequently repackaged the materials for each participant, and all participants received their materials by the end of the 7th week after the manufacturing date. All tests were performed between the 9th week and the 12th week after the manufacturing date, with the majority of the participants completing their characterization by the end of the 11th week. During transportation and storage, the raw materials were kept in sealed packages with the original protecting films on both surfaces of the material to prevent Styrene evaporation, intrusions of foreign object debris, and adverse environmental exposure. In addition, the temperature and humidity of the materials were recorded throughout the duration of the benchmark exercise, from the manufacturing date to the completion of all the tests, to identify any potential aging effects of the materials.

Testing procedure

Preliminary testing. Preliminary testing was conducted at IVW shortly after receiving the materials from Polynt (the material supplier), to finalise the prescribed testing conditions (presented later in the paper) for all participants. The preliminary testing was performed using the material 24 CF50-3K, where square specimens of 100 mm edge length consisting of six individual plies were tested. Specimens were compressed from an initial cavity height of 10 mm, when the top platen was just above the specimen surface, to a final cavity height of 3 mm (7 mm displacement in total), at constant closing speeds of 0.5 mm/s and 3 mm/s. It was found that the specimens had overall dimensions of approximately 250 mm x 250 mm after testing regardless of the closing speed, and the maximum forces were approximately 16 kN for 0.5 mm/s closing speed, and 140 kN for 3 mm/s closing speed. The preliminary testing was to ensure that the prescribed testing conditions were suitable for each individual participant's testing capabilities (see Table 1); therefore, detailed testing results will not be presented in this paper. Specimen preparation. Two specimen configurations were tested by each participant, both square in shape, and consisting of six

individual plies with an edge length of either 100 mm or 50 mm. During specimen preparation, all plies within the same specimen were aligned in the same direction when stacked, as shown in Fig. 2. During specimen preparation, individual plies were marked with the material name, ply number (corresponding to the ply cutting position), and an arrow showing process direction, to prevent mistakes during ply cutting and handling, and to ensure that the material direction could be traced after testing. To minimise environmental exposure and aging during specimen preparation, participants were required to cut the specimens no more than 4 hours before testing and reseal the remaining material until further use. The protective films were removed no more than 15 minutes before testing. The mass and average thickness of each specimen was measured after stacking the plies.

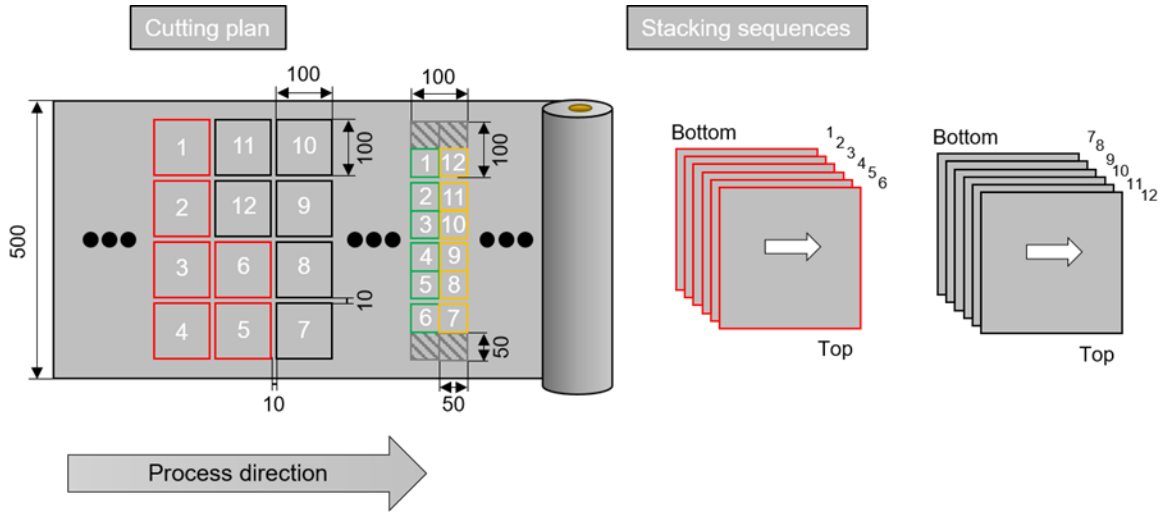

Figure 2 – Specimen cutting and stacking plan followed by each participant of the benchmark

Testing setup. All tests were performed using an open-cavity and constant mass configuration, as shown in Fig. 1, meaning the material should flow freely without obstructions (e.g., sidewalls) and no overflow from the tooling platens should occur. All tests were performed at a top and bottom tooling temperature of 140 °C. Participants were free to use their available heating methods (summarised in Table 1) but were asked to record the steady-state temperature distribution on the platen surfaces prior to the start of each test, if possible.

To ensure the boundary conditions were consistent across all participants and all tests, both testing platens were lined with polyimide (e.g., Kapton or Upilex) films coated with the participant's own choice of release agent. A standard polyimide film thickness of 100 µm was suggested to all participants, but other thicknesses were also allowed. The actual polyimide film thickness information, as well as the release agent used, was collected from participants. All participants were required to cut the polyimide films to a large enough size to cover the entire testing surface and secure the films to the upper and lower platens using heat-resistance tape (e.g. Kapton tape). Participants were advised to draw/print a locating guide at the bottom of the film, indicating the designated sample loading position (see Fig. 3), to ensure that the samples were consistently loaded at the centre of the testing surface. Participants were required to replace the polyimide films at the beginning of each new testing day. In addition, participants were required to inspect the conditions of the film after each test, and if any damage to the film was observed, the film should be replaced immediately, and the last test should be discarded and re-performed.

The release agent selected by individual participants was to be suitable for VE resins, with high temperature stability. Participants were advised to apply the release agent following the manufacturer's recommendation, paying special attention to the amount and frequency that it

needed to be applied (e.g., some release agents need to be re-applied after a certain number of mouldings).

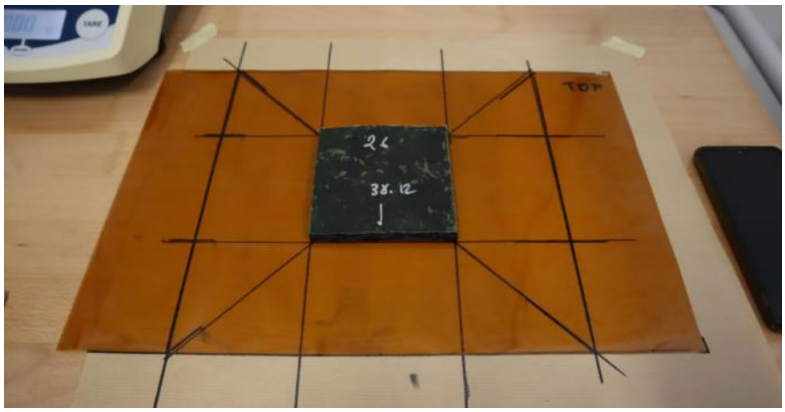

Figure 3 – Example sample locating guide printed onto the polyimide lining film

Testing parameters. All tests were performed at a target mould temperature of 140°C, which was the upper limit of the manufacturer's recommended temperature range as given in the Material section. All tests were performed within a 10 s specimen loading time, defined as the time from the specimen was first in contact with the testing surface, to the start of the test. The reason for prescribing the specimen loading time was mainly to minimise the variabilities in the initial specimen temperature and its influence on the resin viscosity and degree of cure across all tests and all participants. The specimen loading time of 10 s was determined after consulting all participants.

All tests were started at an 11 mm initial cavity height, excluding the thickness of the polyimide film as shown in Fig. 3a, and stopped at final cavity heights of 7 mm, 5 mm and 3 mm (equivalent displacements of 4 mm, 6 mm and 8 mm). Participants were required to hold the specimen at the final cavity height for a minimum of 210 s after the end of each test, to ensure the specimen was sufficiently cured for handling before opening the cavity again for specimen removal. A constant closing speed of 0.5 mm/s was applied to all tests, because according to the preliminary testing results, this speed resulted in 88.6% lower maximum force compared to the higher closing speed of 3 mm/s (16 kN vs. 140 kN), which was achievable for all participants according to Table 1. Furthermore, this speed was considered reasonable for compression moulding of C-SMCs and had been adopted by several participants in the past.

As mentioned in the Specimen preparation section, two specimen sizes were investigated in this benchmark exercise, including square specimens of 50 mm and 100 mm edge length. Generally, the critical RVE size of discontinuous fibre composites was found to be approximately 4 times the fibre length (e.g., approximately 102 mm); the 50 mm specimens were included to further investigate the specimen size effects in squeeze flow testing and also to accommodate participants with smaller platen sizes. Table 3 summarises the prescribed specimen sizes for each set of cavity heights for all participants. Based on the preliminary testing results, a 100 mm specimen was expected to have overall dimensions of 250 mm x 250 mm when compressed from 11 mm to 3 mm (8 mm equivalent displacement), and consequently, a 50 mm specimen was expected to have an overall dimensions of 125 mm x 125 mm. Therefore, participants with platen sizes greater than 250 mm were required to test 100 mm specimens, whereas participants with platen sizes equal to or smaller than 250 mm were required to test 50 mm specimens. In addition, participants with platen sizes greater than 250 mm were advised to also test 50 mm specimens under the largest displacement of 8 mm (cavity height 11 mm to 3 mm), to enable benchmark of the testing results across all participants.

Six repeats were performed for each set of testing parameters listed in Table 3. To eliminate systematic drifting caused by material curing and/or variations in the operators' and testing machines' behaviour as a function of time, a randomised testing approach was implemented in this benchmark exercise: each participant was assigned a unique, randomly generated testing sequence, where all five materials were tested in random order under a same set of testing parameters (referred to as a testing block), and the order of materials was varied between all testing blocks and all participants.

Table 3 – Summary of prescribed specimen sizes for each set of cavity heights. A closing speed of 0.5mm/s was used in all tests

Initial cavity height [mm]	Final cavity height [mm]	Specimen size [mm]	ECN & HESAM	IVW	JKU	KIT & SIM	KUL	RISE	TUM	UoB	UTW & TPRC	WMG & UOS
11	3	100	X	X		X	X		X		X	X
11	5	100	X	X		X	X		X		X	X
11	7	100	X	X		X	X		X		X	X
11	3	50	X	X	X	X	X	X	X	X	X	X
11	5	50			X			X		X		
11	7	50			X			X		X		

Data analysis

As mentioned in the Introduction section, the benchmark exercise is currently going through the data collection process, in preparation for the statistical analysis of all participants' testing results, from which the reliability and repeatability of the current testing methods will be assessed, and the sources of variabilities in the testing results will be investigated. Data management. An in-house system, "PKDE," developed by IVW, was used for data management in this benchmarking exercise. The system provides a tree-like structure (see Fig. 4) to enable complete tracking of the material's history, such that individual testing specimens can be traced back to the approximate location on the original C-SMC roll it was cut from. As shown in Fig. 4, each node of the data management tree represents an activity in the course of the benchmark exercise and is assigned with a unique UUID. To identify the real equivalent to the nodal activity, the UUID and the activity name are converted into a QR code. The metadata of each activity contains all the data collected for the specific specimen, including specimen information, testing parameters and testing results etc.

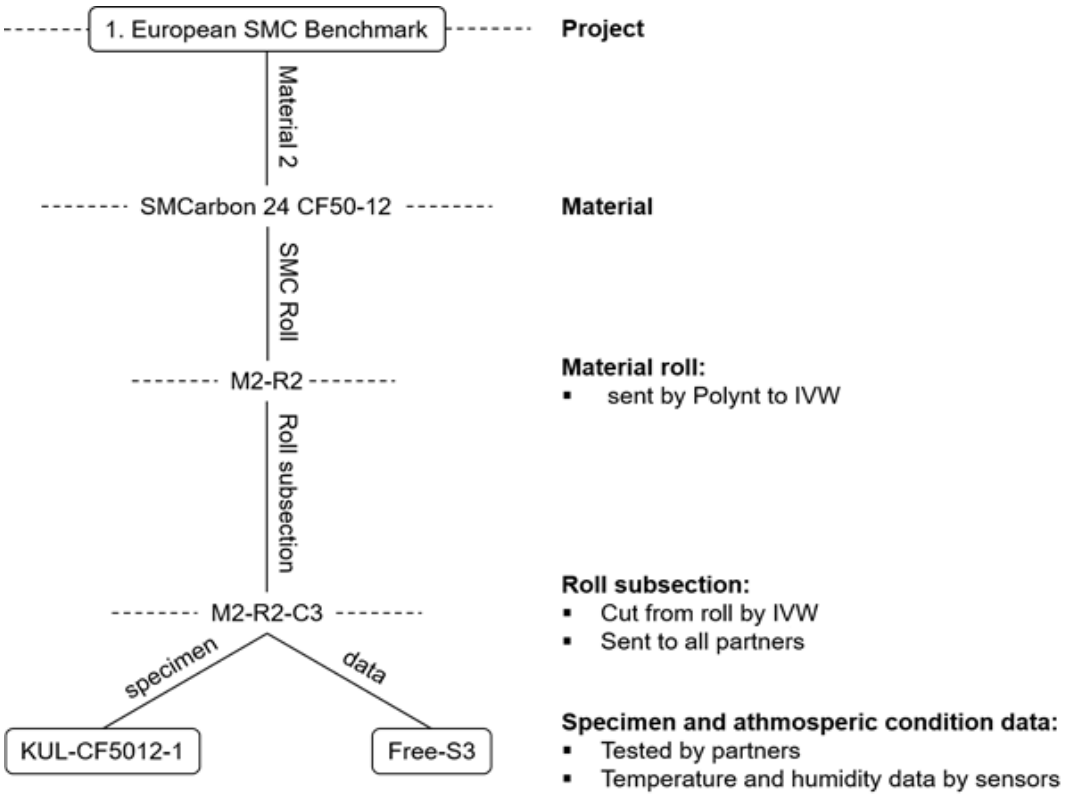

Figure 4 – Structure of PKDE data management tree on the example of the materials used in the benchmark exercise

Data collection. The metadata for each participant were effectively collected by two parties according to Fig. 4, where IVW collected the data related to "Material", "Material roll" and "Roll subsection" during redistributing the materials, and the participant collected the data related to "Specimen and atmospheric condition data" during storage, specimen preparation and testing.

In practice, the data collected from participants were divided into four sets, including:

- Specimen information and testing parameters. Prior to the start of the testing campaign, each participant was provided a spreadsheet consisting of the list of all specimens in the partner-specific testing sequence, where each specimen came with information on material, specimen label, UUID and all prescribed testing conditions and testing parameters; following each test, participants would complete the spreadsheet with information on testing machine, operator, release agent, testing date and time, and specimen mass and initial thickness. The completed spreadsheets were collected after the completion of all testing
- Testing data. Participants were required to collect synchronised data for forces, displacements and/or cavity heights, measured using their usual instrumentation with a sampling rate of 100 Hz or higher during each test. Participants with additional instruments, such as pressure transducers and thermocouples, were encouraged to utilise these instruments to record additional data. The testing data are being collected as individual csv files without any post-processing
- Specimen images. Planar view images of each surface of an individual specimen (see Fig. 5) are being collected to enable analysis of flow-front developments. Participants were advised to use a scanner for taking images, setting up as an A3-sized scan with the specimen centrally located in the scan area. In each image, the specimen surface was marked with the process direction, along with the QR code for the specimen's UUID, and a ruler was positioned near the specimen parallel to the process direction (see Fig. 5). Participants were

recommended to use full-colour scanning settings with a minimum of 600 dpi. The images are being collected as JPG or BMP files

- Temperature-humidity data. Participants were required to export the temperature and humidity data as csv files from the sensors enclosed in the shipping package after completing the test campaign

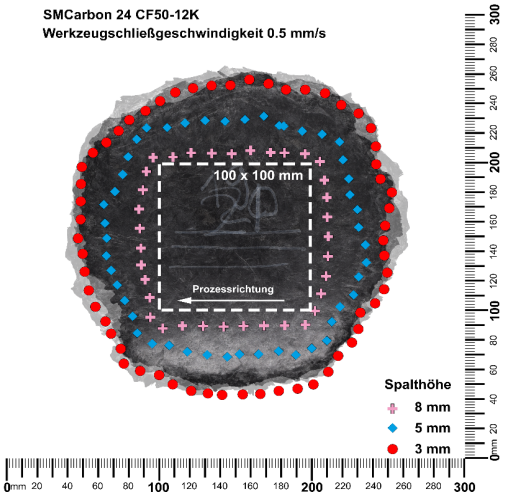

Figure 5 – Example of flow-front development for SMCarbon 24 CF50-12K specimens. Please note that as it is impossible to reuse the specimen after testing, the outlines from 8 mm, 5 mm, and 3 mm displacements were taken from different specimens.

Anticipated outcome

The anticipated research findings include:

- A quantification of variability in squeeze flow test results across 14 European research institutes and identification of potential sources of this variability
- Insights into the flow behaviour of C-SMCs, particularly on the influence of fibre roving size and weight fraction
- Preliminary guidelines for a standardised squeeze flow testing procedure
- Generation of reliable experimental data to enable a follow-up benchmark exercise on C-SMC compression moulding simulation

Conclusions

The methodology of a benchmarking exercise on C-SMC squeeze flow testing has been described in detail. The benchmark exercise involves 14 European research institutes with a total of 10 different in-house developed squeeze flow testing facilities. Five C-SMC materials are being investigated to understand the effects of fibre content and rowing size on the flow behaviour of C-SMCs. A well-prescribed testing procedure has been implemented where preliminary testing has been performed to make sure the proposed testing parameters are suitable for each participant. Detailed instructions have been provided on specimen preparation, testing setup, and testing procedure, where each participant has followed a unique, randomly generated testing sequence. Statistical analysis of the testing results will be performed after all the data have been collected from the participants and uploaded to the in-house developed data management system.

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