

How to Prepare SMC and BMC-like Compounds to Perform Relevant Rheological Experiments?

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Abstract The study of the rheology of injected or compression moulded compounds like SMC or BMC is made particularly difficult by the high content and the intricate arrangement of their fibrous reinforcement. For these two types of compounds, inappropriate rheological testing protocols and rheometers are often used, which leads to a very large scatter of the experimental data. This study describes specific sampling and specimen's preparation methods, as well as dedicated rheometry devices to test their rheology. Following the proposed protocols, it is possible to obtain rheological measurements showing low scatter of the recorded stress values: about $\pm 10\%$ for SMC and about $\pm 15\%$ for BMC-like compounds.

Keywords Polymer composite materials · Rheology · Measurements scatter · SMC · BMC · Fillers

1 Introduction

A large scatter is usually observed when measuring the rheological behaviour of Sheet Moulding Compounds (SMC) or Bulk Moulding Compounds (BMC), for instance. Scatter over $\pm 20\%$ or even $\pm 30\%$ for the measured stresses is not scarce. Minimizing such a scatter by optimizing the specimen preparation and the testing

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conditions is crucial to properly gauge, for instance, if the differences observed between the rheological behaviour of several batches of the same materials are due to a bad preparation of the tested specimens or to weak variations of the constituents of the compounds induced by the upstream processing or to bad storage conditions. Such information is of primary importance from an industrial point of view and has to be preferably obtained before parts are produced.

At first sight, the natural scatter of measurements performed using SMC or BMC-like compounds can be explained by the microstructural heterogeneity (thickness variations, basis weight variations, porosity gradients, etc.) of such materials: see the examples given in Figs. 1c and 3 and for an industrial SMC.

Furthermore, SMC and BMC are composites that can evolve before compression moulding due to the styrene evaporation. The limitation of this phenomenon diminishes the scatter of the rheological tests. For similar reasons, the conditions of the maturation (thickening) stage have also to be carefully controlled in the case of SMC. Thus, controlled environment conditions are necessary.

Nonetheless, a large scatter is not only due to microstructural defects or formulation evolution. It is well known that it is indeed possible to obtain relevant average rheological values and scatter when the dimensions of the specimens are well chosen with respect to the fibre length [1, 2]. This particular length can be considered as a characteristic dimension of the microstructure which determines the dimensions of the experimental Representative Elementary Volume (REV) of the specimens. Experimentally, it is usually admitted that, under homogeneous loading conditions (such as those studied in this work), the specimens have to contain at least five to ten times the length of the characteristic heterogeneity in order to respect a proper scale separation condition. Consequently, the experimental setups that can be used for testing the studied compounds must have large dimensions [3, 4]. Such a dimensional constraint also induces the use of rheometers having large dimensions and testing machines with high load capacity. Thus, the required apparatuses have an unusual design: squeeze flow [5–7], simple compression [1, 2], plane strain compression [2, 8, 9, 18], simple shear [1, 10], and even spiral flow [11] setups. Lubricated compression tests (simple or plane compression tests) have to be preferred to squeeze flow despite the simplicity of this latter [12]. Indeed, the mechanical fields (stress, strain, strain rate) for tests such as squeeze flow or spiral flow display substantial heterogeneity so that the above scale separation condition may be questionable. Besides, and consequently, these tests often require the use of *a priori* assumptions on the rheological constitutive law of the composites [6, 13, 14]. Instead, lubricated tests give direct estimations of the rheological parameters with a much better homogeneity of mechanical fields within specimens [15].

Taking into account all these aspects and experimental constraints, the objective of this contribution is to show how to minimise the experimental scatter during lubricated simple compression tests performed using SMC or BMC. For that, all potential non intrinsic sources of scatter due to for instance uncertain storage conditions, inappropriate protocol of specimens' preparation, not well prepared specimens, not adapted testing protocols, bad lubrication of the compression platens have been withdrawn to focus on other possible intrinsic sources of scatter. Here the specimens with the largest possible dimensions were chosen to fulfil the previously exposed conditions concerning the REV.

2 Materials and Rheometer

2.1 SMC

Four references of low profile SMC were studied. All were supplied by Mixt Composites Recyclables (MCR, Tournon-sur-Rhône, France). The reference SMC was reinforced with glass fibre bundles made up of 600 sized fibres having a diameter of $15\text{ }\mu\text{m}$ (Owens Corning, Chambéry, France). These bundles had a cross section which could be considered as elliptical with a major axis equal to 0.5 mm and a minor axis of 0.1 mm . Their length was 25 mm . The fibre mass fraction was 25% . This corresponds to a fibre bundle volume fraction of $\approx 18.8\%$. The polyester paste of the reference SMC was filled with calcium carbonate (CaCO_3) fillers called A,

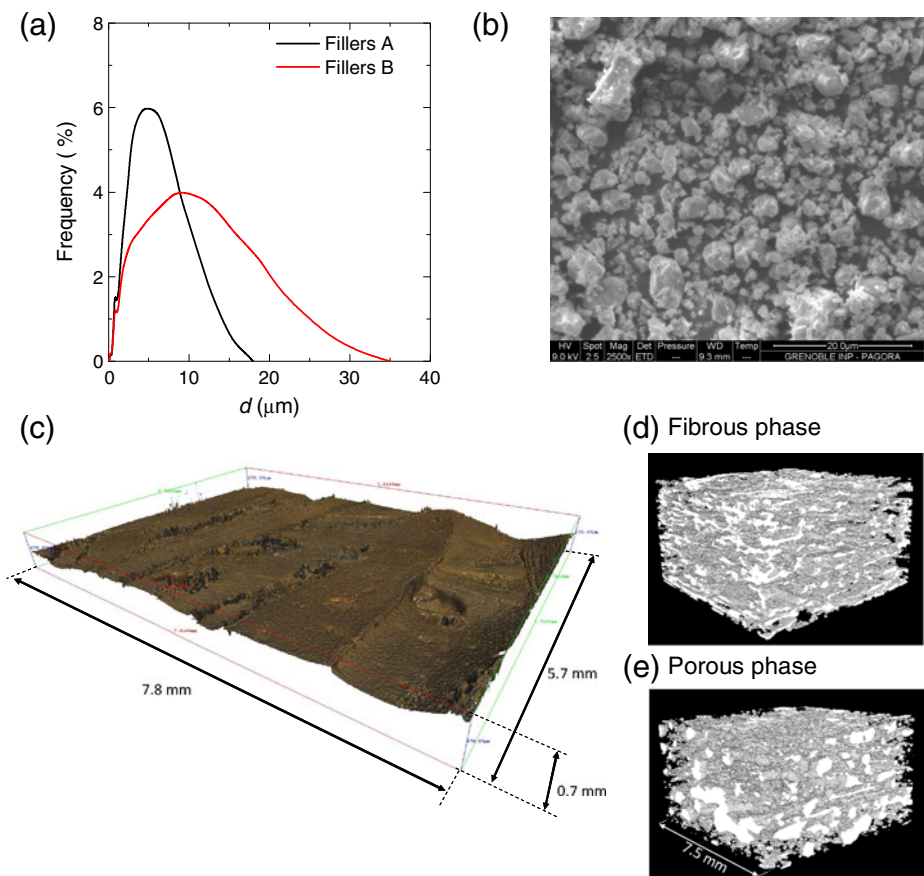


Fig. 1 (a) Grain size distribution of the CaCO_3 mineral fillers used in the reference SMC or SMC 2 and SMC 3. (b) MEB images of the CaCO_3 fillers A. (c) Topographic view of the surface of SMC 4 before compression moulding. (d) 3D View of the fibrous reinforcement of an industrial SMC. (e) 3D View of the porosity ($\approx 15\%$) of the same SMC. Images acquired by X-ray holotomography at the European Synchrotron Radiation Facility (ESRF) on the beamline ID19

whereas SMC 2 was filled with calcium carbonate fillers called B. In SMC 3, the weight content of mineral fillers of A type was increased of 2% compared with the weight content of the reference SMC. In SMC 4, the fibre volume fraction was lower than in the reference SMC and equal to 10.8% instead of 18.8%.

Figure 1a–e illustrate the heterogeneity of the properties of the constituents of SMC together with the heterogeneity of the bulk and surface SMC microstructure properties. The grain size distributions of the fillers A and B, measured using a laser granulometer (Cilas, Particle Size Analyzer 1190L, Orléans, France), are given in Fig. 1a, for instance. The average diameter of particles A is about $5.3\ \mu\text{m}$, whereas the average diameter of particles B is about $8.6\ \mu\text{m}$. The distribution of the size of the A fillers (Fig. 1b) is comprised in a narrow range between 0.03 and $17\ \mu\text{m}$ with a peak distribution at $5\ \mu\text{m}$, whereas the size distribution of the B fillers is wider, that is to say comprised between 0.03 and $32\ \mu\text{m}$ with a peak distribution at $10\ \mu\text{m}$. Figure 1c shows a topographic image (Alicona, Infinite Focus, Graz, Austria) of a SMC sheet measured over a surface of $6 \times 7\ \text{mm}^2$. This picture displays an extremely heterogeneous and wavy surface. The maximum wave amplitude is about $400\ \mu\text{m}$. Its origin is due to the calendering operation which was required to impregnate the fibrous network with the polymeric paste during SMC processing. Such topography is commonly observed for compounds as SMC. In Fig. 1d,e, the inner structure of SMC in their initial state, *i.e.* after impregnation and maturation, and before moulding, is revealed by images acquired using the X-ray synchrotron holotomography. SMC exhibits a fibrous reinforcement phase where fibre bundles are mainly in-plane orientated. The bundle content is so high that each bundle has

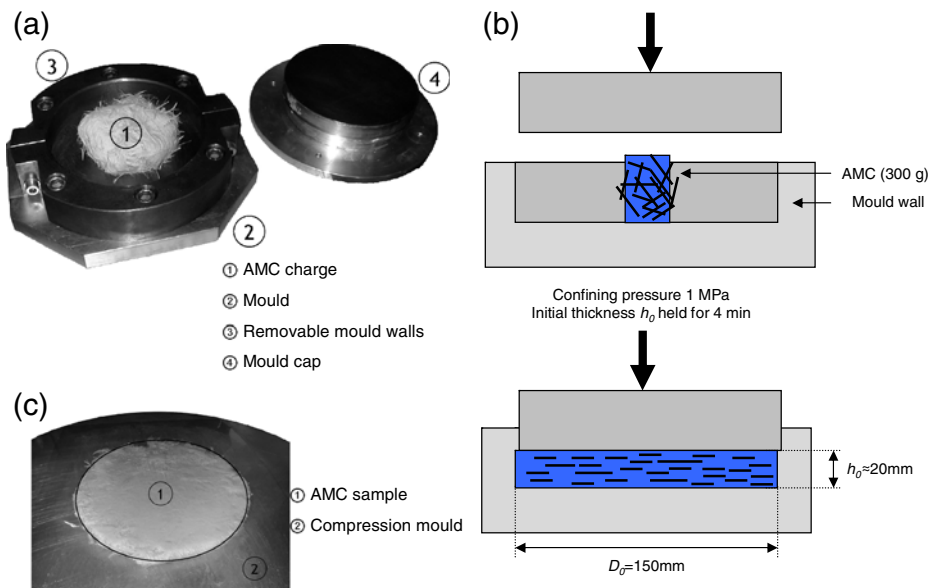


Fig. 2 (a) Photograph of an industrial Advanced Moulding Compound before moulding in the preparation mould. (b) Schematic view of the AMC specimen preparation process and the resulting in-plane fibre orientation. (c) Photograph of an AMC specimen prepared following the specifically designed preparation process

several contacts with its neighbours: materials like SMC can be considered as very concentrated fibrous suspensions. The Fig. 1e shows another aspect of the complexity of the microstructure of SMC: before compression moulding the pore content is very high: the volume fraction of pores is about 15% in the current case. Obviously, such porosity can induce compressibility during rheological tests. Note that the origin and the behaviour of porosity during SMC flow and curing is not well described in the literature [16]. Such microstructural features obviously lead to use adapted rheometers having large dimensions so that the tested specimens have sufficient dimensions to contain several times the size of these various kinds of heterogeneity.

2.2 BMC

The tested BMC was also supplied by MCR. It is known under the commercial name of AMC for Advanced Moulding Compound and is usually processed using a patented process developed by the Inoplast company (France) [20]. The composition of the tested AMC was nearly identical to that of the previous reference SMC, without any thickening agents and fewer mineral fillers (one percent less). Only one formulation of this material was used. Before forming, the fibrous architecture of such materials is extremely disordered as it can be observed in Fig. 2a. This is a major problem to design reliable rheological testing procedures.

2.3 Compression Rheometer

Lubricated simple compression tests were performed using a setup consisting of two coaxial and parallel circular polished plates [1, 2]. This equipment was mounted on a MTS mechanical press (maximum capacity of 20 kN and maximum cross-head velocity of 1,000 mm/min). The surface of the compression platens was coated with a mixture of silicone grease (Molydal Al/Si 3653) and silicone oil (Julabo Thermal H5S) to ensure an homogeneous compression flow of the specimens during the tests. The compression force F together with the current thickness h of the deformed specimen were measured during the tests. These latter were performed at constant compression strain rate $\dot{\varepsilon} = \frac{\dot{h}}{h} = 0.001 \text{ s}^{-1}$ or 0.1 s^{-1} in isothermal conditions at ambient temperature (20°C).

For SMC, fifteen lubricated simple compression tests were performed per testing conditions. For AMC, twenty lubricated simple compression tests were performed. The specimen's preparation procedure are explained below. Within this procedure, the definition of the initial thickness h_0 of the specimens is specified. In the following, for all tests, the average nominal compression stress $\sigma_0 = 4F/(\pi D_0^2)$ (first Piola-Kirchhoff stress) was calculated (D_0 being is the initial specimen diameter) and was plotted as a function of the average logarithmic compression strain $\varepsilon = \ln(h/h_0)$ (Hencky strain).

3 Intrinsic Variability: The SMC Case

Figure 3 illustrate the variability of the microstructural properties of SMC 4 inside the produced sheet. Several cylindrical specimens were cut in various locations of an enrolled sheet of 15 m in length and 0.40 m in width. The diameter D_0 of

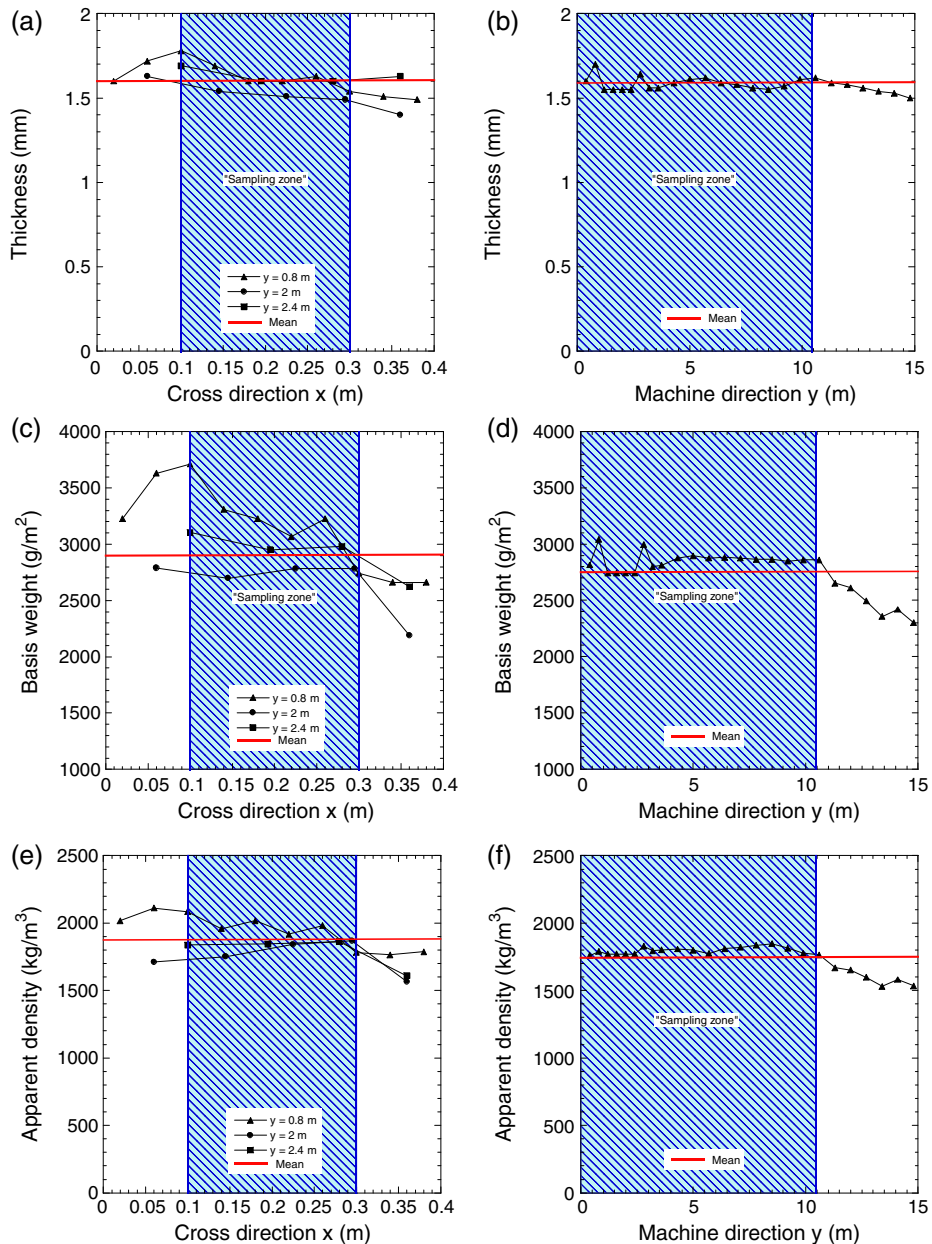


Fig. 3 Measurements of thickness (a), of basis weight (c) and density (e) for specimens cut along the cross directions at various locations along the machine direction. Measurements of thickness (b), of basis weight (d) and density (f) for specimens cut along the machine directions at various locations along the cross direction

these specimens was equal to 150 mm. The thickness e_0 , the mass m_0 of these specimens were measured to calculate the basis weight $W_0 = 4m_0/(\pi D_0^2)$ as well as their apparent density $\rho_0 = W_0/e_0$. Note that it was not easy to measure the thickness

Table 1 Mean thickness, mean basis weight and apparent density together with their associated maximum deviation (given in percentage) cut along the cross direction x and machine direction y of a SMC 4 roll

	Thickness (mm)	Basis weight (g/m ²)	Apparent density (kg/m ³)
Cross direction x			
All specimens	1.6 ($\pm 12\%$)	2900 ($\pm 28\%$)	1870 ($\pm 16\%$)
Sampling zone	1.6 ($\pm 7\%$)	2980 ($\pm 11\%$)	1890 ($\pm 7\%$)
Machine direction y			
All specimens	1.6 ($\pm 8\%$)	2760 ($\pm 17\%$)	1750 ($\pm 13\%$)
Sampling zone	1.6 ($\pm 7\%$)	2850 ($\pm 7\%$)	1800 ($\pm 3\%$)

The “sampling zone” is described in Fig. 3

of SMC sheets as they exhibited a pronounced but usual roughness, see Fig. 1b. The estimated thickness was then measured using a Vernier calliper and performing five measurements per specimen. The measured thickness corresponds certainly to the thickness measured on the peak of the waves shown in Fig. 1b. Figure 3a–f show the variations of the thickness, the basis weight and the apparent density of specimens cut along the cross direction x and the machine direction y of the considered sheet. A large scatter of the measurements can be observed in this figure, particularly along the edges of the rolls, *i.e.* for $x \leq 0.1$ m and $x \geq 0.3$ m, and in the end of the roll, for $y > 10.5$ m. Table 1 shows the maximum deviation around the mean thickness, the mean basis weight and the apparent density for specimens taken along the machine and cross directions in the complete roll or in the hatched “sampling zone” (see Fig. 3). The variations observed for these values are due to the manufacturing process of SMC. Many problems can arise during this process: heterogeneous distribution of the glass fibre bundles or of the porosity, flow of the paste in the edges of the rolls, local rearrangement of the glass fibre bundles during calendering. The sharp decrease of these parameters at the end of the roll can be directly related to the storage conditions of SMC. It is for instance possible that at the centre of the rolls or at their extremities, the SMC is prone to flow due its own weight or due to the weight of the superimposed layers before the maturation of the paste is advanced enough. Taking into account these observations, the specimens used in the rheological studies were not cut close to the edges of the rolls, but in the zone of the rolls defined for $x \in [0.1 \text{ m}, 0.3 \text{ m}]$ and $y \in [0 \text{ m}, 10.5 \text{ m}]$ and illustrated by the hatching in Fig. 3, so as to better analyse the scatter of the rheometry experiments. In this zone, the SMC has quite homogeneous microstructural properties. The maximum deviations of the thickness, basis weight and apparent density measured along the machine direction were all estimated in this zone. This explains why such values are lower in the machine direction than in the cross direction (Table 1).

4 Specimens' Preparation and Experimental Trends

4.1 BMC/AMC

It seems difficult to perform lubricated simple compression tests using AMC or BMC due to their greatly disordered fibrous architecture, on the contrary to SMC where the fibrous network is in-plane orientated.

In a first series of tests, the experimental protocol proposed in Orgéas et al. [19] for BMC taken at the output of an injection screw was followed to prepare specimens of AMC. This protocol consisted of taking the exact mass of compound to prepare a specimen in the shape of a disk with a diameter of 200 mm and a thickness of 20 mm. First a ball of BMC was manually prepared. Next this specimen was slightly compressed in a cylindrical mould to flatten its surface and obtain a disk-like shape. We tried to follow this specimen preparation route with the AMC. However, the tests performed using the as-prepared specimens unfortunately had a maximum deviation of $\pm 60\%$. Such scatter is obviously too wide to pretend to perform relevant control or characterisation rheological tests for such compounds. The scatter is mainly due to the disordered architecture of the fibrous reinforcement of the compound obtained following this protocol and to the length of 25 mm of the fibre bundles, which is bigger than the thickness of about 20 mm. This was not the case for the BMC characterised at the outlet of the injection screw since the flow through the screw drastically reduced the fibre length. Furthermore, it could be observed that the as-prepared specimens had a poor cohesion and a tendency to “loft” (swell). This was not the case of the previous studied BMC that was taken at the outlet of an injection screw too.

Therefore, similarly to a previous study dealing with the rheology of fibre reinforced mortars [17] with long fibre bundles or fibres, the strategy to overcome this problem consisted of giving a proper planar orientation to the fibre bundles of the AMC specimens. This microstructure mimics that of SMC for which the scatter is limited, as previously observed. A new methodology to elaborate the specimens was thus adopted. It consisted first of preparing the specimens in a closed mould to orientate the bundles of the fibrous reinforcement in the plane of the specimens, as described in Fig. 2b. The detail of the elaboration protocol is listed below.

- 300 g of AMC were taken from the storage bin. This mass depends on the size of the mould and on the expected specimen thickness. In the present case, the closed mould allowed specimens with a diameter of 150 mm to be obtained. Besides, 300 g of AMC compounds gave specimens having a thickness of 20 mm.
- This mass was mixed to form a very slender cylinder (height ≈ 60 to 80 mm) so that this compound could flow over the largest possible distance when the preparation mould was closed. During such flow, the bundles orientate along the main plane of the specimen. This first stage is of primary importance and must be undertaken with care.
- Then the mould was closed using a manual press and a closure pressure of 1 MPa was maintained during 4 min. As a result, the lofting of the specimen was drastically reduced during demoulding.
- The close mould was equipped with removable walls. These walls were preliminary removed just before performing the rheological test to avoid the fibrous reinforcement of the specimen to loft. Figure 2c shows a photograph of an as-prepared AMC specimen. The initial thickness h_0 was defined just after the wall removal phase.

4.2 SMC

The protocol used to prepare SMC specimens is based on the procedures that are industrially used. It encompasses the following steps:

- Cylindrical specimens with a diameter equal to 200 mm were precisely cut from the SMC rolls in the zone of interest defined in the previous section. It should be noticed that such precautions are not adopted during the preparation of industrial SMC charges. In the present case, they were necessary to minimise the experimental scatter of rheological measurements.
- Three cylinders were then stacked.
- Then a pre-compression of 0.5 mm of the specimens was performed before testing to flatten the SMC surface in contact with the mould platens and to reduce the core porosity and, as a result, to outgas air or other gaseous products. This latter porosity is located within the SMC sheets due the impregnation process (see Fig. 1e) or at the interfaces between the SMC sheets due to the important roughness of SMC surfaces (see Fig. 1c) [21]. The initial thickness h_0 was defined after the pre-compression phase.

4.3 Measurement Scatter

Twenty lubricated simple compression tests were performed in isothermal conditions at ambient temperature using AMC specimens. The specimens were prepared following the previous protocol. The error bars shown in Fig. 4 indicate a maximum

Fig. 4 Compression stress-strain curves obtained for AMC at an axial strain rate $\dot{\varepsilon} = 0.1 \text{ s}^{-1}$

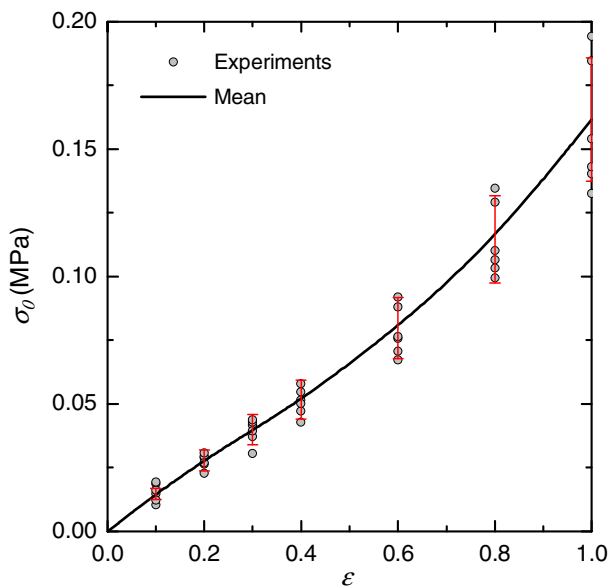
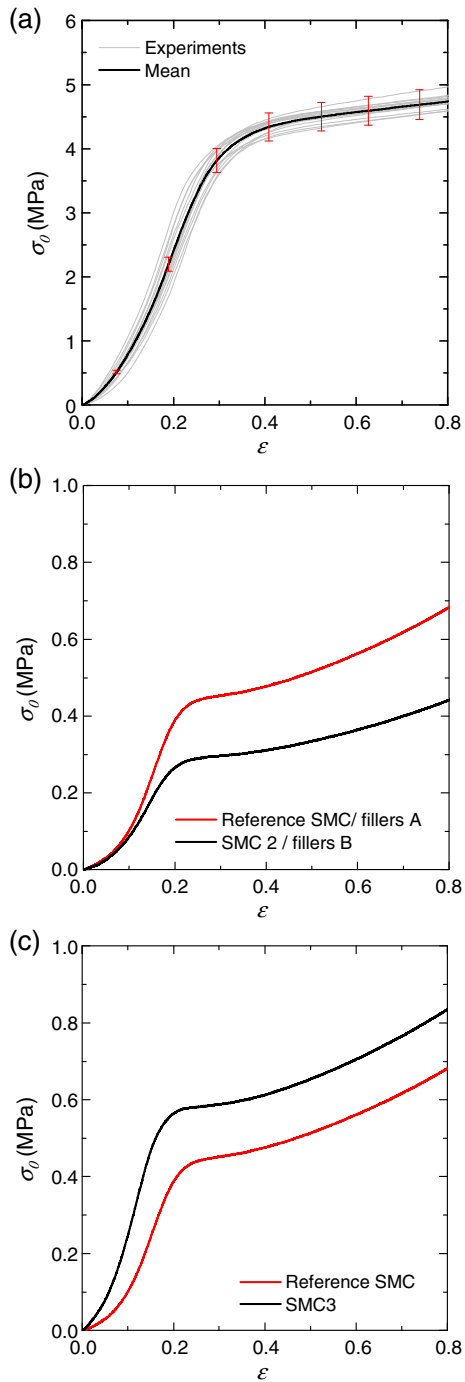


Fig. 5 (a) Compression stress-strain curves performed at an axial strain rate $\dot{\varepsilon} = 0.1 \text{ s}^{-1}$ for the reference SMC. (b) Compression stress-strain curves performed at an axial strain rate $\dot{\varepsilon} = 0.001 \text{ s}^{-1}$ for the reference SMC and the SMC 2: influence of the filler size distribution. (c) Compression stress-strain curves performed at an axial strain rate $\dot{\varepsilon} = 0.001 \text{ s}^{-1}$ for the reference SMC and the SMC 3: influence of the filler content



deviation of the stress-strain measurements equal to $\pm 15\%$. Taking into account the adopted processing route for AMC specimens, such a scatter seems to be reasonable compared with that obtained for SMC (see below). This observation allows both the use of the specific specimen processing route and the testing procedure to be validated for AMC. It should be noticed that the initial fibre length is certainly not deteriorated by the specimen processing route on the contrary to what happens during the industrial processes. Indeed, in that case, the compound is injected into the mould by an injection screw or by a system including actuators and rubber tubes (see for instance the patented system described in [20]). Thus, the rheological results obtained with such prepared specimens have to be considered as upstream data that are actually characteristic of the rheological behaviour of these compounds and that can be used for rheology control purposes. But these data could also be evaluated using compounds taken at the outlet of the injection system to test their rheological behaviour when they flow inside the mould or to perform simulation of moulding operations [18, 19].

Figure 5a displays the evolution of the nominal compression stress σ_0 as a function of the average compression strain ε . The error bars depict that the maximum deviation of the experimental measurements is moderate, equal to $\pm 10\%$. This limited scatter denotes the efficiency of the precautions used to carry out the lubricated simple compression tests. It denotes that the scatter of similar measurements performed using industrial mass-production SMC compounds can be astonishingly weak, even though the specimens are cut in circumscribed areas of the SMC rolls to obtain such results.

4.4 Some Original Rheological Trends Observed for SMC

By following the experimental precautions of the proposed testing methodologies, it is then possible to reveal some rheological trends that could not be observed otherwise due to the overlapping of the rheological data. For instance, Fig. 5a–c illustrates that it is possible to highlight the influence of parameters such as the filler granulometry or the filler content on the SMC rheology. In Fig. 5b, it is interesting to note that the compression stresses decrease as the size distribution width of the fillers increases. A quite similar effect of the filler granulometry distribution width has been already highlighted in the case of BMC [19]. The explanation of such phenomenon would require further investigation but may be related to the lowering of flow localisation phenomena occurring in the SMC paste when the polydispersity of filler particles increases [22, 23]. Figure 5c shows that both the stress levels and the typical shape of the compression curves are modified when increasing the filler content. The flow stress levels are indeed higher as the filler content is increased (even slightly) in such compounds. These two observed effects show that the paste rheology, more precisely the variety of its fillers, has a noticeable effects on the SMC viscous behaviour.

5 Conclusion

Testing methods have been developed to obtain reliable data during rheological simple compression tests for industrial SMC and BMC-like compounds. For industrial

SMC, a $\pm 10\%$ maximum deviation of the compression results can be expected when testing a limited amount of this compound. When using such protocol, interesting influences of materials' parameters like the fillers can be revealed for instance. For BMC-like compounds, the specimen preparation is very specific as the fibre length and orientation with respect to the thickness of tested specimens can be very important. An elaboration method was proposed to orientate the fibre bundles in the plane of the specimens, and to reduce significantly the scatter of rheological measurements. Indeed, using this protocol, a maximum deviation of $\pm 15\%$ can be obtained, which is a great progress compared to the $\pm 60\%$ of the rheological results obtained using the previously proposed specimen preparation method. Such testing methods can be efficiently used to perform rheological control tasks in an industrial context and for laboratory research work.

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