

A comprehensive investigation on the temperature and strain rate dependent mechanical response of three polymeric syntactic foams for thermoforming and energy absorption applications

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ABSTRACT

Polymeric syntactic foams (PSF) are a kind of composite material, which consists of a polymer matrix reinforced by hollow thin wall glass micro-spheres. Because of their low density, low moisture absorption, relatively high specific strength and stiffness, PSF are often employed in aerospace and submarine applications in which they are subjected to a wide range of temperature conditions. Due to the temperature sensitivity of the polymer matrix, the physical and mechanical behaviour of PSFs is highly sensitive to temperature variations. Besides, their dynamic response is significantly affected by the strain rate. This research investigates the temperature and strain rate dependence of the mechanical behaviour of polyurethane, epoxy and nylon syntactic foams under compressive and tensile loads. The selected materials have in common that their mechanical characteristics, thermal properties, and dimensional stability make them suitable for both impact engineering and thermoforming applications. The experimental results, acquired over a temperature range from -25°C to 100°C at low and high strain rates, reveal a clear interplay between temperature and strain rate effects on the mechanical behaviour of the materials under investigation. This synergy is observed to vary based on both the matrix material and the loading mode. Notably, the rate dependency of polyurethane and nylon syntactic foams is significantly influenced by the testing temperature during compression, while in tension the influence of the temperature on the rate dependency is moderate or negligible. In contrast, the rate dependency of the epoxy syntactic foam remains largely unaffected by the testing temperature. Deformation and failure mechanisms were analysed by examining the failure surface of the tested samples using SEM micrographs.

1. Introduction

Polymer syntactic foams (PSFs) are composites of a polymer matrix and micro-balloons. The inclusion of hollow micro-balloons enhances their mechanical properties, particularly the compressive strength, without significant increases in density. Additionally, they exhibit lower moisture absorption compared to open-cell or conventional closed-cell foams [1,2]. These advantages, including high specific mechanical properties, energy dissipation, excellent thermal properties, and low moisture absorption, make them suitable for various applications in submarine, automotive, and aerospace industries [2].

The mechanical response of polymer syntactic foams, composed of polymer, micro-balloons, and air, is determined by their interaction

during deformation [3]. Extensive studies have examined their compressive and tensile properties under quasi-static loading and room temperature conditions [4–8]. Due to the rate-dependent nature of the polymer matrix and the dissipative deformation and failure of the micro-spheres, their mechanical behaviour is significantly influenced by the deformation rate [9,10]. Application of syntactic foams include lightweight protective structures [11], radomes, blast mitigation and sport equipment [2].

Measuring the dynamic properties of polymer-based materials poses challenges related to their mechanical and acoustic characteristics. The Split Hopkinson Bar apparatus is commonly used for assessing the mechanical response of materials at high strain rates [12,13]. The accurate evaluation of stress and strain during dynamic loading requires

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Table 1
Properties of the investigated materials.

MATERIAL	HYTAC® XTL	HYTAC® C1R	POLYURETHANE SYNTACTIC FOAM
TYPE	thermoplastic syntactic foam	thermoset copolymer syntactic foam	thermoplastic syntactic foam
MATRIX	polyamide	epoxy	polyurethane
FILLER	hollow glass microspheres	hollow glass microspheres	hollow glass microspheres
DIAMETER OF FILLER	20–50 μm	5–30 μm	10–130 μm
WALL THICKNESS OF FILLER	approx. 0.6 μm	approx. 0.4 μm	approx. 1.15 μm
VOLUME FRACTION	0.5	0.5	0.5
DENSITY	672–737 kg/m^3	800–864 kg/m^3	700 kg/m^3
THERMAL CONDUCTIVITY	0.12 W/m K	0.17 W/m K	N/A
CTE	$63 \times 10^{-6} \text{ m/m/}^\circ\text{C}$	$57.6 \times 10^{-6} \text{ m/m/}^\circ\text{C}$	N/A

achieving force equilibrium and uniform deformation, which is particularly complex for materials with low stress wave propagation velocity [14,15]. Furthermore, the signal-to-noise ratio of the stress and strain histories measured during dynamic experiments on soft materials can be problematic, often necessitating the use of low-impedance, high-force sensitivity instrumented bars [16]. Several studies have explored the dynamic behaviour of polymer syntactic foams at room temperature [9, 10], and researchers have employed various techniques to understand strain rate effects with respect to materials composition and reinforcement [17–21].

Syntactic foams are used in various environmental conditions, as their properties are crucial for oil, gas, marine, and aerospace applications [22,23]. The temperature sensitivity of the polymer matrix [24] affects significantly the physical and mechanical behaviour of PSFs. Therefore, studying the temperature dependence of the properties of PDFs is essential.

Numerous studies have analysed how their physical properties vary with temperature [25–28]. Arezoo et al. [29] measured separately the effects of temperature and strain rate on the compressive response of Rohacell foams. Song et al. [30] addressed the effects of temperature and adiabatic heating on the dynamic compressive behaviour of an epoxy syntactic foam. Zhang et al. [31] investigated the effects of strain rate, temperature, and confinement on the compressive response of an epoxy syntactic foam.

However, the open literature is scarce of thorough investigations on the concurrent effects of temperature and strain rate in different loading modes. Most of the current literature investigates the mechanical response of syntactic foams predominantly in compression, and the dynamic behaviour in tension is often neglected, particularly for what concerns impact applications at sub-ambient temperatures.

In this work we present an extensive investigation on the combined effects of temperature and strain rate on the dynamic behaviour of a polyurethane, an epoxy based, and a polyamide based syntactic foams for thermoforming and energy absorption applications. The variation of strength and energy absorption with strain rate and temperature is examined in detail. The dynamic properties of the above-mentioned foams are investigated in compression and tension to assess the dependency of their response to different deformation mechanisms and motivate the development of novel constitutive models. Scanning electron microscope (SEM) micrographs are examined to understand the deformation and failure mechanisms of the studied materials.

2. Materials

The foam materials investigated in this work comprise three different polymer matrices with uniform dispersion of hollow glass microspheres. All materials were commercially available and sourced as plates of 25 mm thickness. The first foam examined is a polyurethane syntactic foam with hollow glass microspheres of diameter ranging from 10 to 130 μm and average wall thickness of 1.15 μm . The material is characterised by excellent impact strength, pronounced rate dependence, dimensional stability and chemical resistance. These characteristics make it suitable for energy absorption and thermoforming applications. The volume

fraction of the micro-balloons, defined as the ratio of the volume occupied by the spheres divided by the total volume, is approximately 0.5. Detailed information on the above foam are available in Ref. [11]. The second foam is an epoxy-based syntactic foam produced by CMT Materials,¹ commercially available under the name of HYTAC® C1R. This material is particularly suitable for aerospace and thermoforming applications due to its high strength and durability, especially when high friction is needed, and high-speed rubbing scenarios are likely. The hollow glass microspheres have diameter included between 5 μm and 30 μm , average wall thickness of 0.4 μm and volume fraction approximately equal to 0.5. Additionally, the mechanical response of the polyamide matrix syntactic foam HYTAC® XTL, produced by CMT Materials¹ is also investigated. This material is particularly suitable for thermoforming and impact energy absorption applications due to its high toughness, low thermal expansion, low thermal conductivity and high strength. The diameter of the microspheres in this foam varies between 20 μm and 50 μm , with an average wall thickness of 0.6 μm . The volume fraction of the micro-balloons is approximately 0.5. A summary of the available microstructural and physical properties for the three materials is provided in Table 1.

To visualise the microstructure of the three foams these were machined into small cuboidal blocks and polished using sandpaper of P1000 grade to ensure the exposure of the microspheres. Air refrigerant-based propellant cleaner was used to minimise the presence of dust. The microstructure of the three materials, visualised using a ZEISS Axio Lab. A1 optical microscope, showed uniform dispersion of the microspheres (Fig. 1).

Differential scanning calorimetry (DSC) and dynamic mechanical analysis (DMA) were conducted to determine the glass transition temperatures (T_g) of the selected syntactic foams. Single cantilever beam samples of dimensions 40 mm $\times$ 5 mm $\times$ 2.5 mm were utilised for DMA analysis. The parameters for the DMA analysis were the following: deformation strain equal to 0.1 %, frequency of 1 Hz and temperature increase rate of 3 $^\circ\text{C/min}$. The DSC analysis was conducted using circular cylindrical specimens of diameter equal to 5 mm and thickness of 1.2 mm. A temperature increase rate of 10 $^\circ\text{C/min}$ was used for DSC characterization. The storage modulus, loss modulus and tan delta as function of temperature for the polyurethane foam, Hytac C1R and Hytac XTL foams are depicted in Fig. 2a–c. Fig. 3 presents the heat flow change along with temperature increase during DSC.

Figs. 2a and 3a indicate that the glass transition temperature of the polyurethane matrix ranges between -59.82°C and -33.64°C with a mid-point of -49.50°C . These values are in line with the values reported in literature for polyurethane [32].

Figs. 2b and 3b indicate the glass transition temperature for the HYTAC® C1R range between 147.16°C and 168.04°C with a mid-point of 157.45°C . This is in line with the glass transition reported in Ref. [33]. Additionally, a beta relaxation appears between -75°C and -50°C . As HYTAC® C1R is a copolymer epoxy based syntactic foam, this transition may be attributed to a secondary component in the

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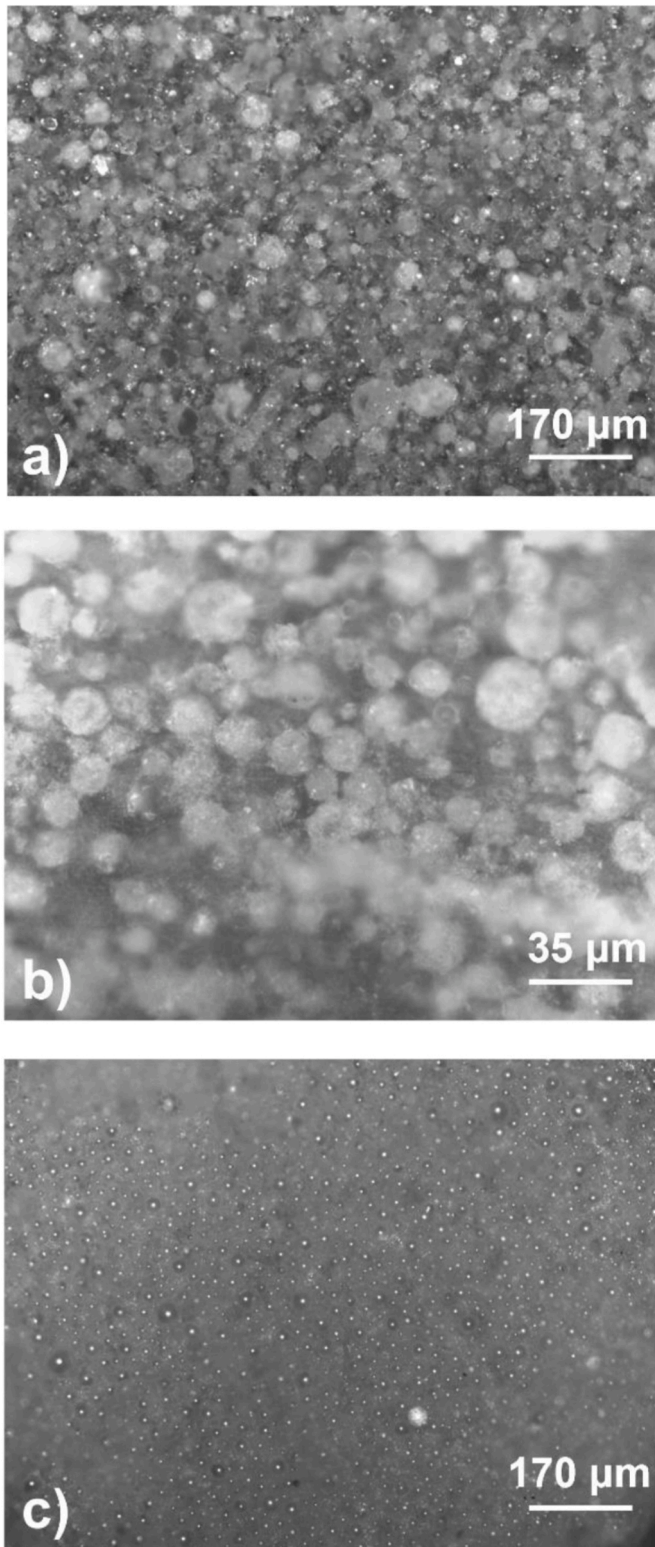


Fig. 1. Optical microscope images illustrating the microstructure of: (a) Hytac® XTL polyamide syntactic foam (b) Hytac® C1R epoxy copolymer syntactic foam and (c) polyurethane syntactic foam.

copolymer blend constituting the matrix. This is in line with the considerations reported in Ref. [34]. Similar considerations on the beta transition in epoxy resins are reported in Refs. [34–36].

The DSC analysis carried out on the HYTAC® XTL foam highlight a melting temperature of 213.43 °C (Fig. 3c). The DMA analysis features

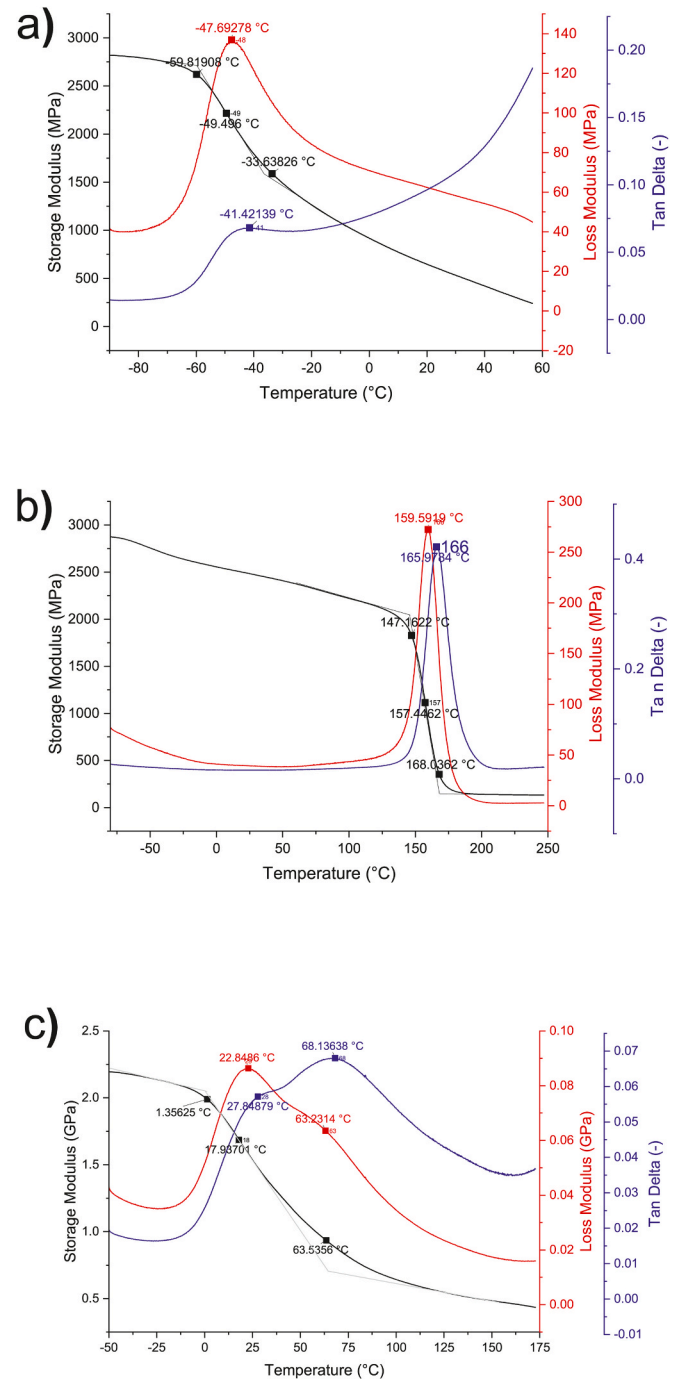


Fig. 2. Storage modulus, loss modulus and Tan Delta as function of temperature for: (a) Polyurethane syntactic foam; (b) Hytac® C1R foam; (c) Hytac® XTL foam.

an apparent double glass transition between 1.36 °C and 63.54 °C (Fig. 2c), which may be ascribed to morphological features present in semi-crystalline polymers, such as nylon [37]. The variation between the upper glass transition and the lower glass transition increases with the increase of crystallinity [37]. The measured glass transition and melting temperatures are in line with the ones reported in Refs. [38–40].

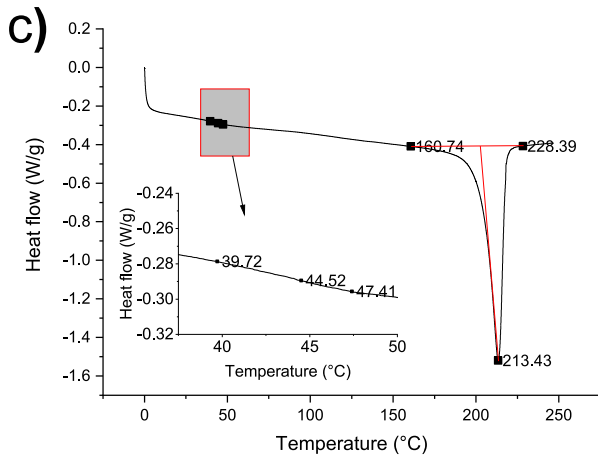
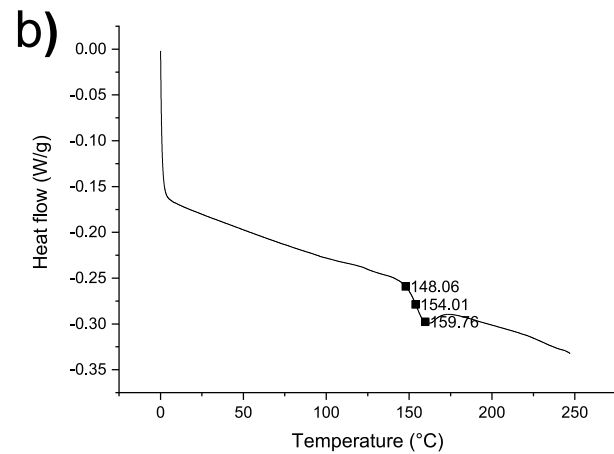
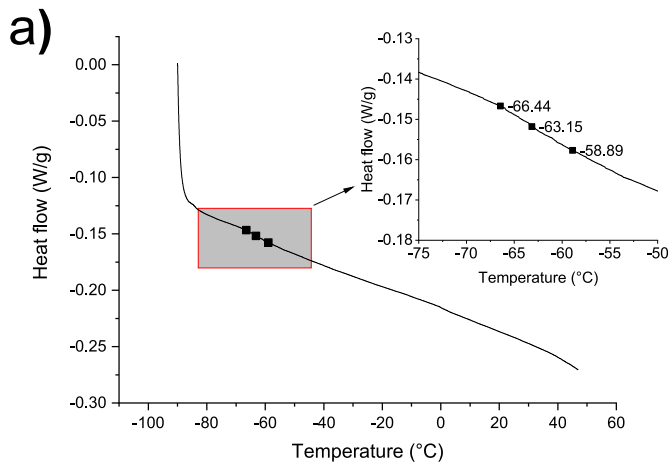


Fig. 3. Heat Flow Change during DSC analysis for: (a) Polyurethane syntactic foam; (b) Hytac® C1R foam; (c) Hytac® XTL foam.

3. Specimen design

3.1. Compression specimens

Circular cylindrical specimens of diameter 5 mm and height 2.5 mm were employed for high-rate and quasi-static compression experiments

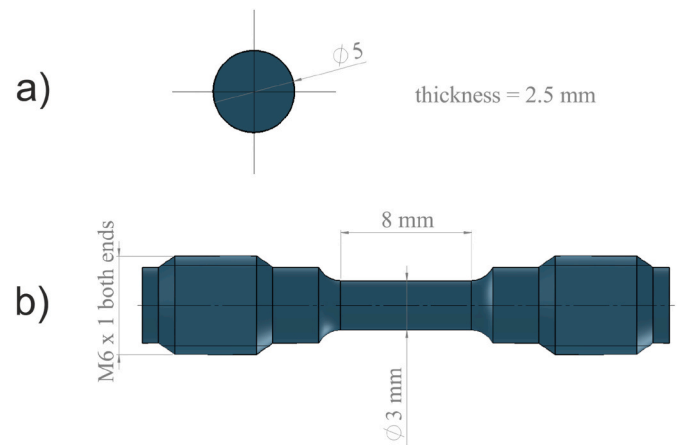


Fig. 4. Geometry and dimensions of the compression and tensile samples adopted in this study.

on the HYTAC® C1R and HYTAC® XTL foams (Fig. 4a). Previous studies [14] suggested that the optimal aspect ratio (defined as the ratio between height and diameter of the sample) to ensure uniaxial loading conditions while minimising inertial effects in high strain rate experiments should be between 0.5 and 1. The dimensions and aspect ratio of the samples used in this study were designed to facilitate the obtainment of dynamic equilibrium conditions and, at the same time, allow for future studies on the effect of moisture absorption on the compressive response of the studied materials. Analogous specimen geometry was employed in Ref. [41].

Samples of same aspect ratio but diameter equal to 10 mm were adopted for the compressive experiments on the polyurethane syntactic foam addressed in this study. The choice of this diameter was prompted by the enhanced signal to noise ratio obtainable on this softer material when adopting larger samples. Additional experiments on samples of dimensions as in Fig. 4a were conducted to allow for future studies on the effects of water intake on the compressive response of the material.

3.2. Tension specimens

Quasi-static and high-rate tensile experiments were conducted using the axisymmetric dog-bone specimen of diameter 3 mm and gauge length 8 mm depicted in Fig. 4b. It is noted that the use of metallic end caps was avoided to reduce the inertia and favour the achievement of force equilibrium conditions. As the materials under investigation allow for the manufacturing of the threads directly on the sample, M6 threads were manufactured at both ends for the connection to the incident and transmitted bars.

4. Experimental setup

4.1. Quasi-static experiments

Quasi-static experiments were carried out using a Zwick Z250 screw driven material testing machine. Experiments were conducted at a nominal strain rate of 10^{-3} s^{-1} in displacement control. Forces were measured using resistive 20 kN and 500 N loading cells for compressive and tensile loads respectively. During all compression experiments the platens were lubricated using a moderate amount of petroleum jelly to minimise friction and consequent barrelling of the samples. Suitable fixtures ensured the straightness of the samples during deformation. The recorded force histories were synchronised with the strains measured using a non-contact laser extensometer. To investigate the mechanisms

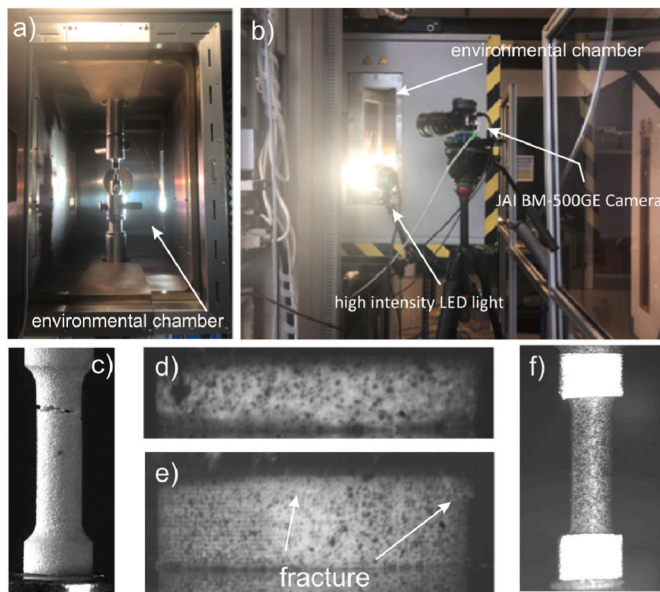


Fig. 5. Experimental setup employed during the quasi-static experiments (a, b) and pictures of tested samples at incipient failure: (c) tensile polyurethane foam sample at room temperature, (d) fully densified compression polyurethane foam sample at 0 °C, (e) failure of Hytac® C1R compression sample at 0 °C, (f) tensile Hytac® XTL foam sample at 0 °C with reflective strips for laser extensometer (bright white).

of deformation and failure the experiments were also recorded using high resolution cameras.² Strain histories during loading were analysed using the commercially available LaVision DaVis Digital Image Correlation software. It's important to note that certain materials require strain rates lower than 10^{-3} s^{-1} to achieve true isothermal straining. However, a previous study [11] confirmed that the mechanical response of a syntactic foam exhibited no appreciable difference between nominal strain rates of 10^{-4} s^{-1} and 10^{-3} s^{-1} . Therefore, the quasi-static experiments were conducted at nominal strain rate of 10^{-3} s^{-1} .

The temperature dependence on the mechanical responses of the three polymer syntactic foams was investigated at temperatures ranging between -25 °C and 100 °C . The chosen temperature range for the experiments represents typical operating conditions in aerospace and automotive applications. This includes the use of syntactic foams as core materials in sandwich structures, crash-absorbing components, protective structures for electronic components, bumpers, and materials for thermoforming applications.

The ambient temperature was prescribed using an environmental chamber provided with feedback-controlled system.³ Sub-ambient temperatures were obtained connecting a liquid nitrogen dewar to the inlet of the environmental chamber. The flow rate of the liquid nitrogen was regulated automatically. The temperature prior and during the experiments was measured via a built-in thermocouple positioned inside the environmental chamber. An additional type-K thermocouple was positioned close to the specimen to monitor its surface temperature. The sample was exposed to heating or cooling for additional 20 min after temperature equilibrium was reached. Heat transfer numerical simulations conducted in Abaqus CAE estimated that the homogeneity of the temperature within the sample was obtained in approximately 4.5 min. The experimental setup employed during quasi-static experiments is presented in Fig. 5.

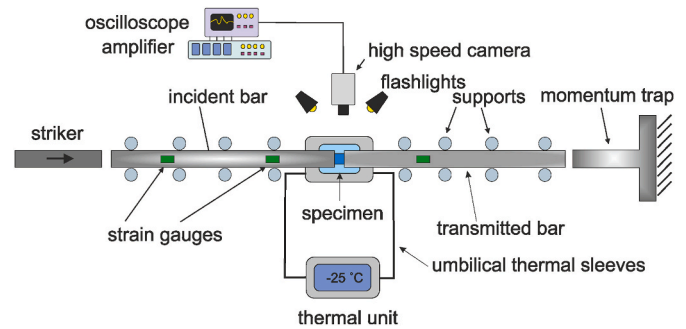


Fig. 6. Schematic of the Split Hopkinson pressure bar apparatus.

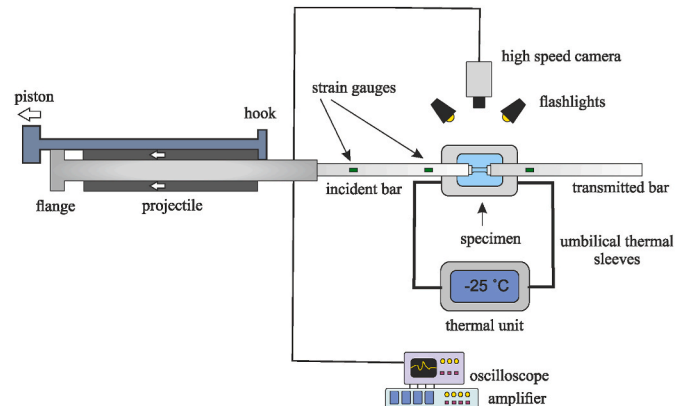


Fig. 7. Schematic of the Split Hopkinson tensile bar apparatus employed in this study.

4.2. High-rate experiments

The measurement of the high-rate response of polymeric syntactic foams poses peculiar challenges associated both to the mechanical response and to the acoustic properties of the materials. Firstly, the compression of the samples beyond the densification strain requires, particularly for ductile polymer foams, the generation of stress pulses of relatively long duration. To fulfil this requirement the compression high-rate experiments were conducted on a Split Hopkinson Pressure Bar (SHPB) [12] setup capable of generating stress pulses of duration exceeding 1.1 ms. The incident and transmitted bars were made of titanium 6Al4V with solid circular cross section of diameter equal to 16 mm. The length of input bar output bar and striker was equal to 2.7 m. The bars were supported by low friction bearings spaced at a distance of approximately 300 mm. Secondly, polymer based materials are characterised by relatively low stress wave propagation, resulting in a more challenging achievement of dynamic equilibrium conditions. The attainment of compressive force equilibrium conditions was facilitated, interposing 1 mm thick cardboard pulse shapers (436 g/m^2) between the striker and the input bar. A schematic illustration of the setup used during the experiments is illustrated in Fig. 6. A similar setup was employed in Ref. [16].

The measurement of the dynamic tensile response of polymeric materials presents additional challenges. As the strength of polymer foams is relatively low the signal to noise ratio of the measured signals can result inadequate. This experimental difficulty was overcome by using low mechanical impedance bars. Specifically, the input bar was made of Ti6Al4v and had a diameter equal to 10 mm and a length 2.5 m. A hollow aluminium bar of diameter equal to 7.95 mm, wall thickness of 1.47 mm and length 1.5 m was used as the output bar to increase the sensitivity to the transmitted force signals. A schematic illustration of the employed setup is depicted in Fig. 7. A detailed description of the

² JAI BM-500GE Camera.

³ Severn Thermal Solutions UK, Draycott Business Park, Cam, Dursley GL11 5DQ provided with Eurotherm 3508 controller.

Split Hopkinson Tension Apparatus (SHTB) can be found in Ref. [42]. The achievement of force equilibrium conditions during dynamic tensile experiments is even more challenging as the samples are characterised by larger gauge length. Dynamic equilibrium conditions were achieved by using 1 mm neoprene pulse shapers in combination with a striker of significant length (2.5 m).

The use of strikers of considerable length in both the tension and compression apparatuses leads to the unavoidable superimposition of incident and reflected waves into the input bar at the gauge location. Therefore it is not possible to distinguish the forward and backward travelling waves from the measurements of a single strain gauge. The determination of stress and particle velocities at the interface between the sample and the bars was obtained by bonding an additional strain gauge in the incident bar and by separating the incident and reflected waves using the algorithm described in Ref. [43]. The same algorithm was also used in Refs. [44,45].

The above particle velocities can be inferred from the recorded strain gauge signals as follows:

$$v_i(t) = \frac{\sigma_r(t) - \sigma_i(t)}{\rho_i c_i} \quad (1)$$

$$v_t(t) = \frac{-\sigma_t(t)}{\rho_t c_t} \quad (2)$$

Where $\sigma_i(t)$ and $\sigma_r(t)$ are the histories of incident and reflected stresses at the interface between input bar and specimen, $\sigma_t(t)$ is the history of the transmitted stress at the interface between output bar and samples, ρ_i and ρ_t are the densities of input and output bars while c_i and c_t are their longitudinal stress wave propagation velocities

Strain rate $\dot{\epsilon}(t)$, strain $\epsilon(t)$ and stress $\sigma(t)$ in the specimen can therefore be computed as:

$$\dot{\epsilon}(t) = \frac{v_i - v_t}{l} = \frac{1}{l} \left(\frac{\sigma_r(t) - \sigma_i(t)}{\rho_i c_i} + \frac{\sigma_t(t)}{\rho_t c_t} \right) \quad (3)$$

$$\epsilon(t) = \int_0^t \dot{\epsilon}(t) dt \quad (4)$$

$$\sigma(t) = \frac{1}{2A_s} \{ A_i [\sigma_i(t) + \sigma_r(t)] + A_t [\sigma_t(t)] \} \quad (5)$$

Where l is the length of the sample, A_i , A_t and A_s are the cross-sectional areas of incident bar, transmitted bar and specimen.

Strains in the samples were also measured by means of high-speed photography employing a high-speed camera (Specialised Imaging Kirana), typically operated at 2×10^5 fps during high-rate compression and 5×10^4 fps during high-rate tension experiments. Subsequent image analyses were conducted using the commercially available LaVision DaVis Digital Image Correlation software.

The temperature of the samples was prescribed by means of an environmental chamber equipped with feedback control system⁴ (Fig. 7). The system, specifically designed for the SHPB and SHTB, consists of an air temperature conditioning unit (ACU), a temperature controller, a sample enclosure and two thermally insulated umbilical sleeves connecting the ACU with the enclosure. The insulated sample enclosure is provided of two perpendicular transparent windows to allow the imaging of the sample during deformation and failure. Sub-ambient temperatures were obtained connecting a liquid nitrogen dewar to the inlet of the air conditioning unit.

The heated or refrigerated air is transferred to the enclosure through the sleeves, by means of an appropriate built-in fan enclosed in the system. The temperature of the samples during high-rate experiment

Table 2

Summary of the experiments conducted for this study.

type of test	material	average strain rate [s ⁻¹]	temperature [°C]	repetitions
Quasi-static compression	Hytac® XTL	10 ⁻³	-25, 0, 25, 50, 75, 100	3
	Hytac® C1R	10 ⁻³	-25, 0, 25, 50, 75, 100	3
	Polyurethane syntactic foam	10 ⁻³	-25, 0, 25, 50, 75, 100	3
Quasi-static tension	Hytac® XTL	10 ⁻³	-25, 0, 25, 50, 75	3
	Hytac® C1R	10 ⁻³	-25, 0, 25, 50, 75	3
	Polyurethane syntactic foam	10 ⁻³	-25, 0, 25, 50, 75	3
High-rate compression	Hytac® XTL	≈3000	-25, 0, 25, 50, 75, 100	3
	Hytac® C1R	≈3000	-25, 0, 25, 50, 75, 100	3
	Polyurethane syntactic foam	≈800	-25, 0, 25, 50, 75, 100	3
High-rate tension	Hytac® XTL	≈50	-25, 0, 25, 50, 75, 100	3
	Hytac® C1R	≈50	-25, 0, 25, 50, 75, 100	3
	Polyurethane syntactic foam	≈50	-25, 0, 25, 50	3

was varied between -25 °C and 100 °C and was measured via a built-in thermocouple located inside the sample enclosure. An additional type-K thermocouple was positioned on the incident bar in close proximity to the sample, to monitor the agreement between the temperature measured by built-in thermocouple in the chamber and the temperature near the specimen.

The uniformity of the temperature within the specimens prior to the experiments was assessed via preliminary heat transfer numerical simulations conducted in Abaqus CAE. These estimated that the temperature within the sample becomes homogeneous approximately 4.5 min after the temperature in the chamber reaches the desired target temperature.

It is noted that the elastic modulus of the Hopkinson bar rods changes with the temperature. The use of a thermal enclosure around the sample may therefore lead to local changes in the elastic modulus of the portion of incident and transmitted bars within and in proximity of the enclosure. These changes can result in alterations to the stress wave propagation velocity, acoustic impedance, and the distortion of elastic waves as they propagate through the temperature gradient. Additionally, this may lead to inaccurate readings from any strain gauge affected by the temperature [46].

However, considering that the elastic moduli of Ti6Al4V and aluminium remain nearly stable within the temperature range [47–50], and given the limited length (approximately 100 mm) of the incident and transmitted bars within the enclosure, we deemed the effects of heating or cooling at the bar ends caused by the thermal unit in the Split Hopkinson Bar apparatus to be negligible in this study.

This conclusion is supported by the achievement of dynamic equilibrium conditions, as assessed using the algorithm described in Ref. [43], which utilizes strain gauge readings from various locations on the incident and transmitted bars.

5. Results and discussion

This section presents the stress strain responses measured during compressive and tensile experiments on the three syntactic foams investigated in this study. Table 2 summarises the experiments, categorised in terms of applied strain rate and prescribed temperatures. Each experiment was repeated at least three times to assess the

⁴ Severn Thermal Solutions UK, Draycott Business Park, Cam, Dursley GL11 5DQ provided with Eurotherm 3508 controller.

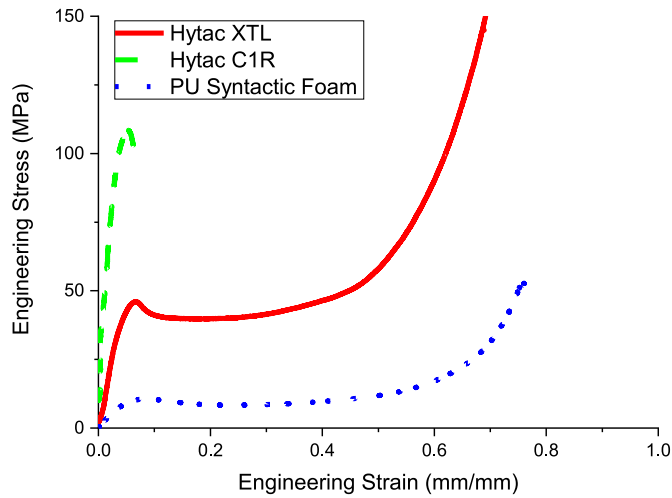


Fig. 8. Quasi-static compressive responses of the investigated foams at room temperature.

repeatability of the measured results. The analysis of the obtained responses allowed to define the temperature and strain rate sensitivity of the three materials for the two loading modes.

5.1. Quasi-static compression

Representative compressive responses of the three investigated syntactic foams under quasi-static loading at room temperature are presented in Fig. 8. At room temperature, both the polyamide based Hytac® XTL foam and the polyurethane (PU) syntactic foam exhibit the typical behaviour of an elastic-plastic cellular material, clearly divided into elastic, plateau and densification regions. Both foams are characterised by a ductile behaviour with an extensive plateau region before densification.

The slope of the elastic region of the stress-strain curve is determined by the reversible deformations of matrix and micro-balloons. At the end of the elastic region the stress reaches a peak, commonly referred as the compressive strength, followed by a plateau region, associated to the collapse of the hollow glass microspheres [51]. After the complete collapse of the glass microbubbles, opposite cell walls of the foam come into contact with each other, and the mechanical response is defined by the behaviour of the solid matrix [51].

The elastic moduli for the PU syntactic foam and the Hytac® XTL foam are 183.48 MPa and 1.22 GPa respectively. The quasi-static compressive strength of the polyurethane syntactic foams is equal to 10.63 MPa, measured at an engineering strain of 0.087. The Hytac® XTL foam is characterised by a compressive strength of 45.50 MPa corresponding to an engineering strain equal to 0.067.

Conversely, the room temperature quasi-static compressive response of the Hytac® C1R foam is characterised by a substantially brittle behaviour. The elastic modulus is equal to 2.32 GPa. The peak stress is reached at engineering strains in the region of 0.051 and it is followed by the failure of the sample, at strains of approximately 0.070. A similar behaviour was observed in Ref. [52] for epoxy syntactic foams of comparable density and volume fraction.

Fig. 9a–c, depict the quasi-static compressive responses of the three syntactic foams at temperatures ranging between -25°C and 100°C . It is noted that the charts report only one representative curve for each loading and temperature condition. Additional information on the scatter of the experimental results is reported in Section 4.5.

The plots highlight an evident temperature dependence of the compressive response for all materials. The compressive strength of the Hytac® XTL foam (Fig. 9a) reduces from 45.50 MPa to 22.17 MPa from room temperature to 100°C and increases up to 55.98 MPa and 68.96

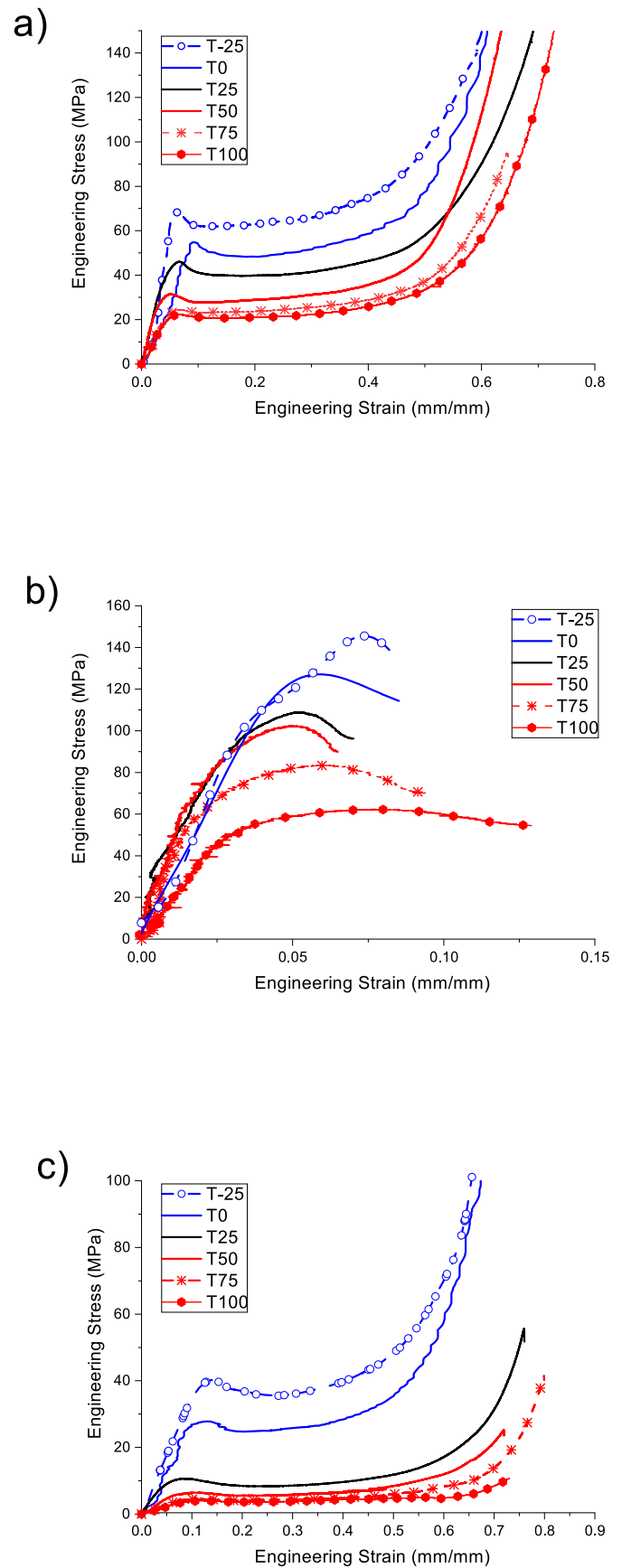


Fig. 9. Quasi-static compressive responses measured at various temperatures: (a) Hytac® XTL foam (b) Hytac® C1R foam and, (c) Polyurethane syntactic foam.

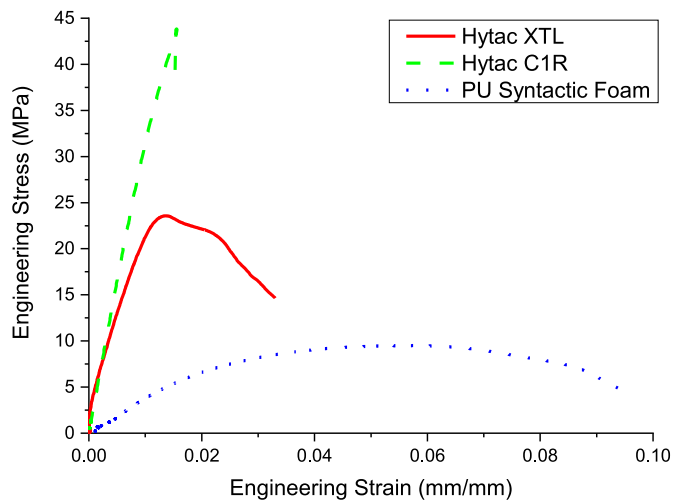


Fig. 10. Quasi-static tensile responses of the investigated foams at room temperature.

MPa at 0 °C and −25 °C respectively. This decrease of strength with temperature has been observed for the other material systems as well as in previous works reported in literature [30,53]. This happens because higher temperatures ease chain mobility, reducing the yield stress of the material. It is also noticeable that, while the strength of the material is affected by the testing temperature, the slope of the initial elastic phase and the extension of the plateau region do not appear significantly affected by this environmental parameter.

Fig. 9b presents the temperature dependence of the quasi-static compressive response of the Hytac® C1R foam. It is evident that the testing temperature affects both the strength and ductility of this copolymer epoxy based syntactic foam. The compressive strength at zero and −25 °C increases of the 21.38 % and of the 27.59 % with respect to the compressive strength measured at room temperature. Correspondingly the peak stress decreases of the 43.76 % when the temperature within the environmental chamber reaches 100 °C. The strain to failure varies between 0.070, at room temperature, and 0.013 measured at 100 °C.

The temperature dependence of the quasi-static compressive behaviour for the PU syntactic foam examined in this work is illustrated in Fig. 9c. The compressive strength of the material increases considerably when the temperature is decreased to sub-ambient temperatures, with the compressive strength increasing of the 276.71 % from room temperature to −25 °C. In comparison, the reduction of compressive strength appears to be moderate when increasing the temperature from room temperature to 100 °C. This is due to the fact that the glass transition temperature of the material is relatively close to the minimum temperature examined in this study, hence the asymmetry in the temperature dependence towards cryogenic temperatures. It is noted that, in this case, the slope of the elastic phase appears to be considerably affected by the temperature, with a substantial softening starting from 50 °C.

It is observed that, following the initial elastic phase, both Hytac® XTL and polyurethane syntactic foams display a peak in their compressive characteristics, followed by a softening phase leading to the plateau region. This decrease in stress corresponds to the onset of microsphere crushing and is caused by the relative mismatch between the strength of particles and polymer matrix [54]. The results illustrated

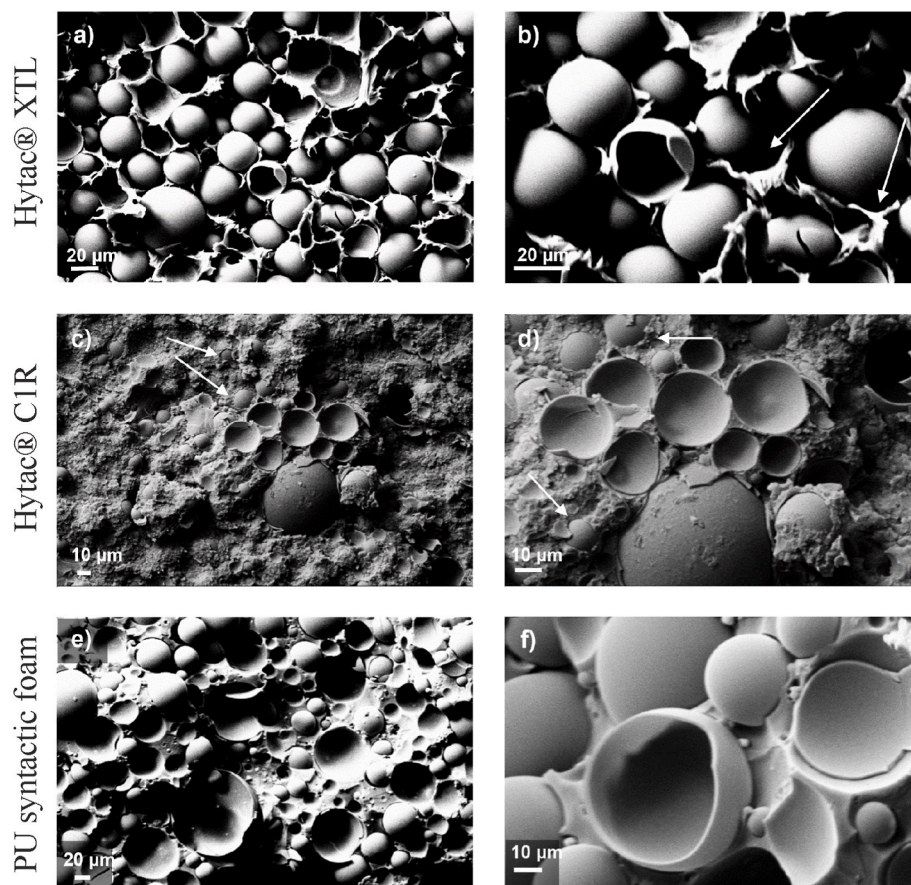


Fig. 11. Micrographs illustrating the failure surface of tensile samples tested at room temperature and a strain rate of 10^{-3} s^{-1} . Failure surface of (a), (b) Hytac® XTL polyamide syntactic foam (c), (d) Hytac® C1R epoxy copolymer syntactic foam and (e), (f) polyurethane syntactic foam.

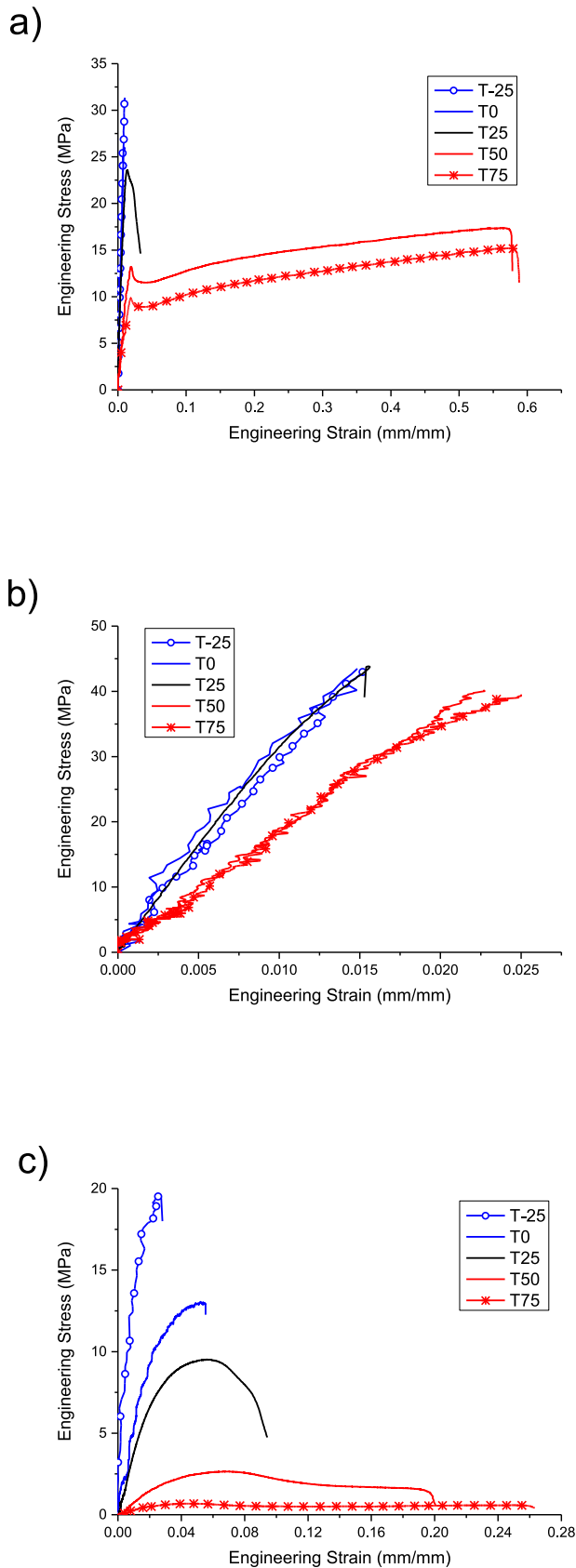


Fig. 12. Quasi-static tensile responses measured at various temperatures: (a) Hytac® XTL foam (b) Hytac® C1R foam and, (c) Polyurethane syntactic foam.

in Fig. 9a,c shows a decrease in the magnitude of this stress drop for increasing temperatures, plausibly due to the a more progressive crushing process at higher temperatures.

5.2. Quasi-static tension

Fig. 10 presents the quasi-static tensile responses of the polyamide, epoxy and polyurethane based syntactic foams at room temperature. The tensile response appears to be significantly different from the progressive deformation and subsequent densification observed in compression, with the three foams exhibiting a relatively brittle behaviour. The comparison of tensile and compressive strengths of the three foams denotes an asymmetry in the mechanical behaviour, this may be ascribed to different mechanisms of deformation and failure of matrix and micro-balloons during compression and tension loading [11]. The above asymmetry is particularly pronounced for the Hytac® XTL and Hytac® C1R foams. The average tensile strength of the former is equal to 22.91 MPa, compared to the 45.50 MPa measured in compression. The tensile average tensile strength of the Hytac® C1R is equal to 43.83 MPa, 59.36 % lower than the corresponding compressive strength. The tensile strain to failure for the XTL and C1R foams are 0.026 and 0.016 respectively, with corresponding elastic moduli of 1662.50 MPa and 2776.2 MPa.

The above tension-compression strength asymmetry is more limited for the PU syntactic foam investigated, being the measured average tensile strength of 9.37 MPa comparable to the compressive strength (10.63 MPa). The room temperature tensile response of the PU syntactic foams is noticeably more ductile, with elastic modulus and strain to failure equal to 234.94 MPa and 0.089.

Fig. 11 illustrates the failure surface of tensile samples tested quasi-statically at room temperature. The fracture morphology of the specimens was characterised using a Carl Zeiss Evo LS 15 VP Scanning Electron Microscope. The micrographs show that the failure of the microspheres occurs, predominantly, in perpendicular direction with respect to the direction of loading. This is consistent with previous observation from the relevant literature [11]. More in detail, Fig. 11a–d shows that only a small fraction of the micro-balloon appear fractured. The matrix of the Hytac® XTL foams shows sign of deformation and tear (arrowed in Fig. 11b), while the failure of the matrix of the Hytac® C1R foam appears substantially brittle with a large portion of microspheres embedded in the matrix at failure (arrowed in Fig. 11c and d). These observations suggest that, in tension, the failure of the polyamide and epoxy based syntactic foams is dominated by the failure of the matrix. The micrographs in Fig. 11e and f illustrate the failure surface of the studied PU syntactic foam. These show a larger fraction of failed glass micro-balloons as well as evidence of matrix-microsphere interfacial debonding. These observation suggest that the failure of this foam is due to a combination of the failures of matrix, interface and micro-spheres.

Fig. 12a–c, illustrate the temperature dependence of the quasi-static tensile response of the three syntactic foams examined in this study. For the sake of clarity, the plots include only one representative curve for each loading and temperature condition. Additional information on the scatter of the experimental results is reported in Section 4.5.

The charts delineate an evident temperature dependence of both tensile strength and strain to failure for both the polyamide and polyurethane syntactic foams (Fig. 12a,c). Conversely, the Hytac® C1R epoxy-based syntactic foam shows a negligible temperature dependence of the tensile strength and a moderate variation of the strain to failure for the range of temperatures considered in this study (Fig. 12b).

More in detail, the tensile strength of the Hytac® XTL foam increases from 22.91 MPa to 32.33 MPa when the temperature is varied from room temperature to -25°C , while it reduces to 15.87 MPa at 75°C (Fig. 12a). The effect of the temperature is even more evident in terms of strain to failure. The material behaves in a substantially brittle manner at sub-ambient temperatures with engineering strains to failure in the region of 1 %. At room temperature the strain to failure increases to

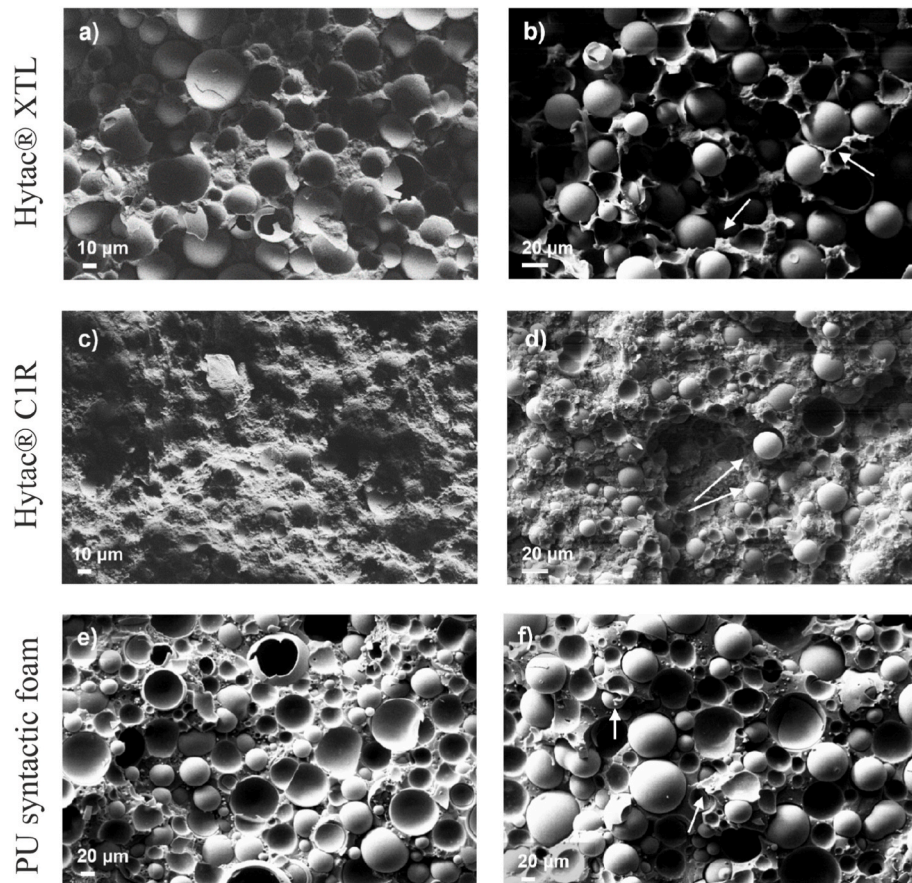


Fig. 13. Micrographs illustrating the failure surface of tensile samples tested at sub-ambient and higher than ambient temperatures at a strain rate of 10^{-3} s^{-1} . Failure surface of Hytac® XTL polyamide syntactic foam deformed at (a) -25°C and (b) 75°C ; failure surface of Hytac® C1R epoxy copolymer syntactic foam deformed at (c) -25°C and (d) 75°C ; failure surface of PU syntactic foam deformed at (e) -25°C and (f) 75°C .

approximately 3 %, while at temperatures of 50°C and beyond the material alters noticeably its response, with failure strains exceeding 55 %. This is explained by the fact that the glass transition temperature of the material lies within the range of temperatures considered in this study. Therefore, the material transitions to its rubbery states at higher temperature, with increased mobility of the polymer chain. It is worth noting that, after the initial elastic phase, the stress-strain curves obtained at 50°C and 75°C show a peak followed by a decrease in stress. This is plausibly due to the rearrangement of chain molecules at temperatures near the glass transition [55]. As the peak stress is reached, deformation begins to localize due to local softening. As deformation continues, more chain molecules are drawn and straightened in parallel.

A similar behaviour, with a gradual variation of both strength and strain to failure with temperature is shown in Fig. 12c. The strength of the examined PU syntactic foam reduces from 9.37 MPa to 2.45 MPa from room temperature to 75°C while it increases to 19.47 MPa at -25°C . The engineering strain to failure varies between approximately 3 % and 26 % when the temperature is varied between -25°C and 75°C . It is noted that the tensile response measured at 100°C is not reported for this material as it could not be resolved with adequate accuracy by the utilised load cell.

Fig. 12b depicts the temperature dependence of the Hytac® C1R foam. The tensile strain to failure varies from approximately 1.5 % to around 2.5 % increasing the temperature. Correspondingly the Elastic modulus reduced from 3.03 GPa to 1.66 GPa. The tensile strength of the material remains substantially unchanged varying the temperature, with a value of approximately 40 MPa.

Fig. 13 illustrates the influence of the testing temperature on the failure mode of quasi-statically deformed tensile samples. The

micrographs on Figs. 13a and b confirm that the failure of the polyamide-based syntactic foam, Hytac® XTL, occurs predominantly within the matrix, with only a small fraction of micro-balloons exhibiting fractures. However, there is a distinction: the matrix of the samples deformed at -25°C (Fig. 13a) appears jagged, indicating a brittle failure, whereas the matrix of the samples tested at 75°C shows clear signs of ductile deformation and tear (indicated by arrows in Fig. 13b). These observations align with the results presented in Fig. 12a and are consistent with the information regarding the glass transition temperature of the material as reported in Fig. 2c.

The micrographs in Fig. 13c and d depict a brittle failure of the samples manufactured from the Hytac® C1R foam, irrespective of the testing temperature. Nevertheless, certain differences are observed. The samples tested at a temperature of -25°C (Fig. 13c) are characterised by a failure surface in which the microspheres appear completely embedded in the matrix, whereas several micro-balloons become visible on the fracture surface of the samples deformed at a temperature of 75°C , as indicated by arrows in Fig. 13d. This suggests that, although the failure of this epoxy based syntactic foam is predominantly influenced by the failure of the matrix, a portion of the deformation may be attributed to debonding between the matrix and the microspheres.

Figs. 13e and f indicate that the deformation and failure mechanisms of the investigated PU syntactic foam might not be significantly influenced by the testing temperature. The specimens deformed at a temperature of 75°C appear to exhibit a moderately lower count of fractured microspheres and modest indications of ductile deformation within the matrix (highlighted by arrows in Fig. 13f). However, the quasi-static tensile failure of this material consistently results from a combination of matrix, micro-balloons, and interfacial failures,

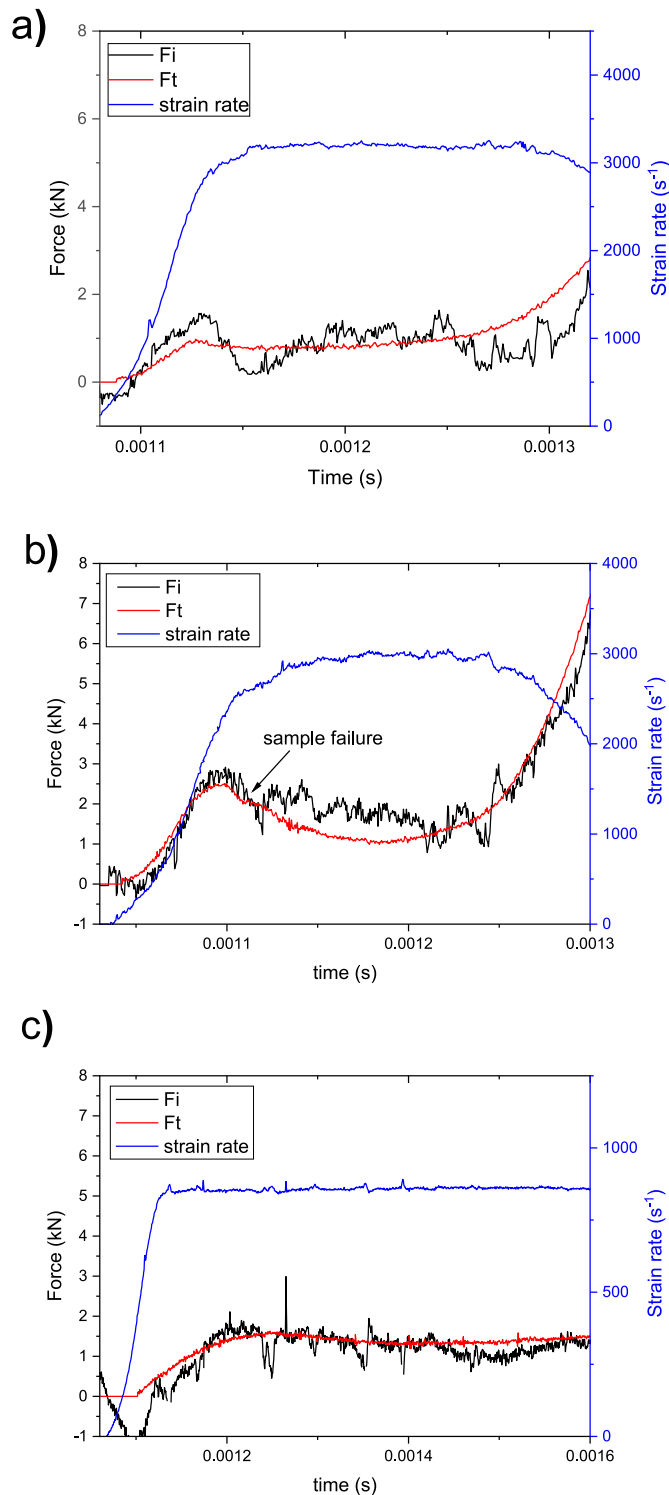


Fig. 14. Representative forces in the incident and transmitted bars and strain rate histories during high-rate compression experiments carried out at a temperature of 100 °C: (a) Hytac® XTL syntactic foam (b) Hytac® C1R syntactic foam and (c) Polyurethane syntactic foam.

regardless of the testing temperature.

5.3. High-rate compression

This section presents the results measured during high-rate compression tests conducted on the three syntactic foams investigated

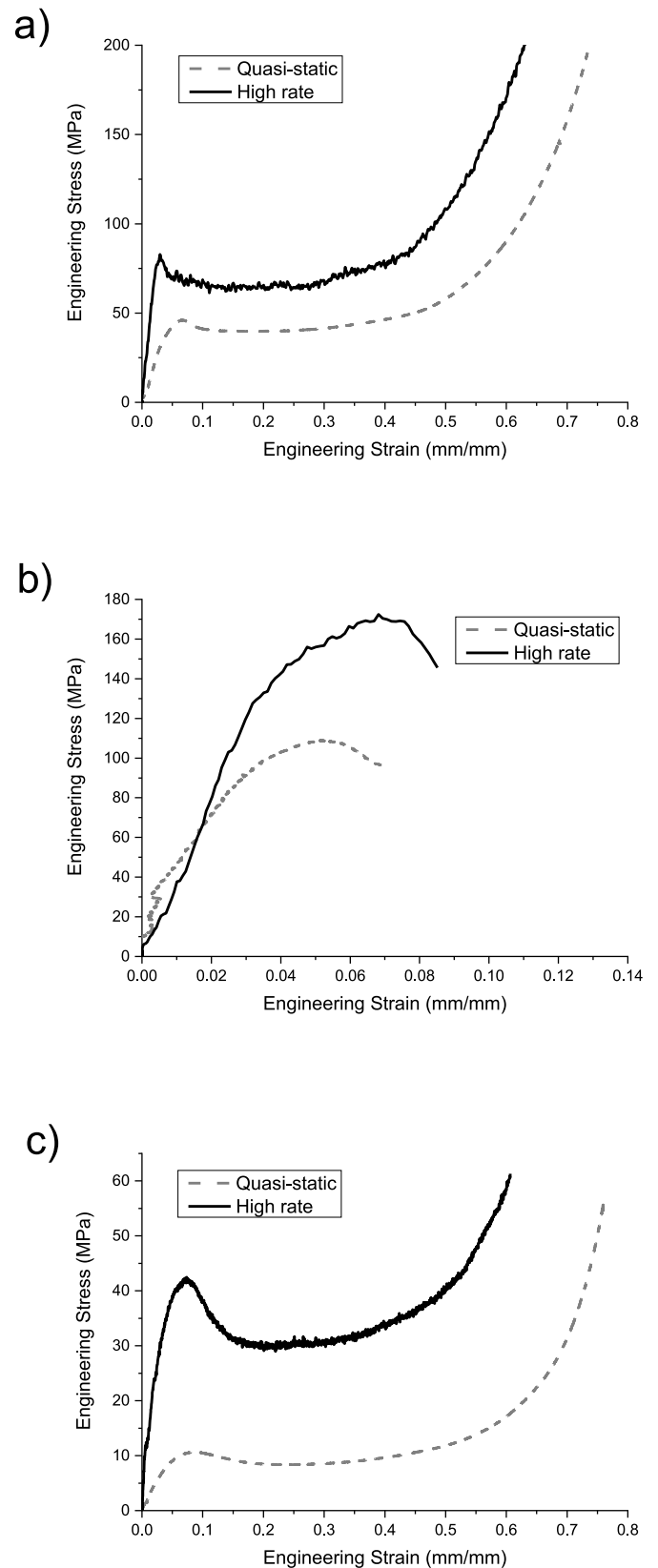


Fig. 15. Mechanical response measured at room temperature during quasi-static and high strain rate compression experiments: (a) Hytac® XTL syntactic foam (b) Hytac® C1R syntactic foam and, (c) Polyurethane syntactic foam.

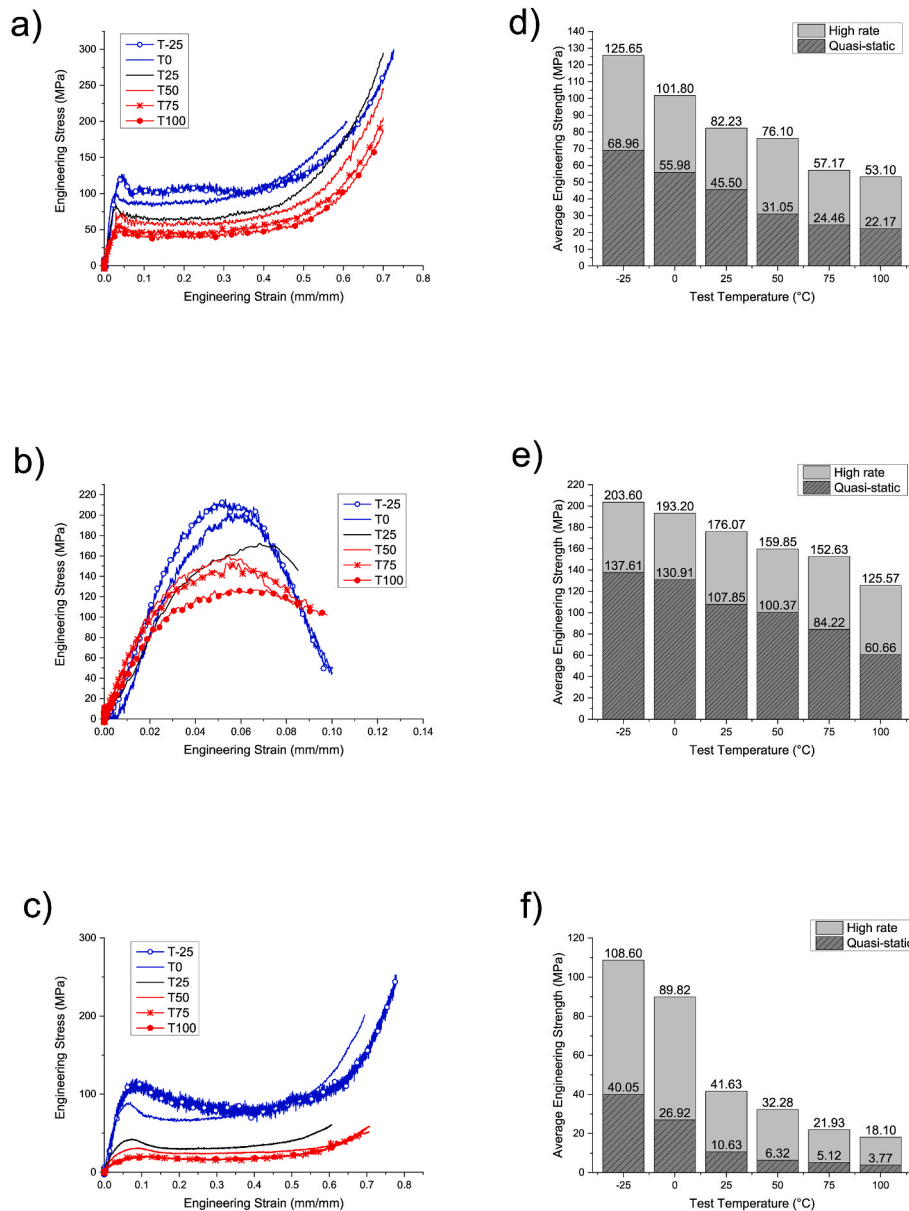


Fig. 16. Temperature dependence of the high-rate compressive response of: (a) Hytac® XTL syntactic foam (b) Hytac® C1R syntactic foam and, (c) Polyurethane syntactic foam. Rate dependence of the average compressive strength measured at different test temperatures: (d) Hytac® XTL syntactic foam (e) Hytac® C1R syntactic foam and, (f) Polyurethane syntactic foam.

in this study. The experiments were conducted at different temperatures to assess the combined effects of strain rate and temperature on the dynamic response of the materials.

The validity of the measured mechanical responses was verified by examining the achievement of force equilibrium conditions. Fig. 14 displays the time histories of the forces acting on the sample at the interface with the incident and transmitted bars for a set of representative experiments conducted at a temperature equal to 100 °C. Fig. 14 also presents the time histories of the measured strain rate.

It is noted that the achievement of dynamic equilibrium conditions is usually more challenging for higher temperatures. These validity conditions were attained more readily during experiments carried out at lower temperatures. All stress-strain characteristics reported in this section were measured during experiments in which dynamic equilibrium were achieved at the early stage of deformation.

Fig. 14a illustrates the incident and transmitted force histories measured on the Hytac® XTL syntactic foam during a dynamic

compression experiment carried out at 100 °C. It is visible that the incident force oscillates around the force measured on the transmitted bar in the initial phase of loading and then becomes approximately coincident with the force measured on the output bar. The time history of the strain rate, approximately stable during deformation confirms the validity of the experiment.

Fig. 14b presents the force histories measured on the input and output bars during an experiment on the Hytac® C1R foam conducted at the same temperature (100 °C). It is evident that dynamic equilibrium conditions were attained at the very early stages of deformation, corresponding to the elastic region of the stress-strain response of the material.

Fig. 14c reports the incident and transmitted force measured during a representative SHPB experiment on the Polyurethane syntactic foam. The strain rate is essentially constant and the forces at the incident and transmitted bars equalise when the output force is equal to around 900 N, corresponding to an engineering strain equal to approximately 2.5 %.

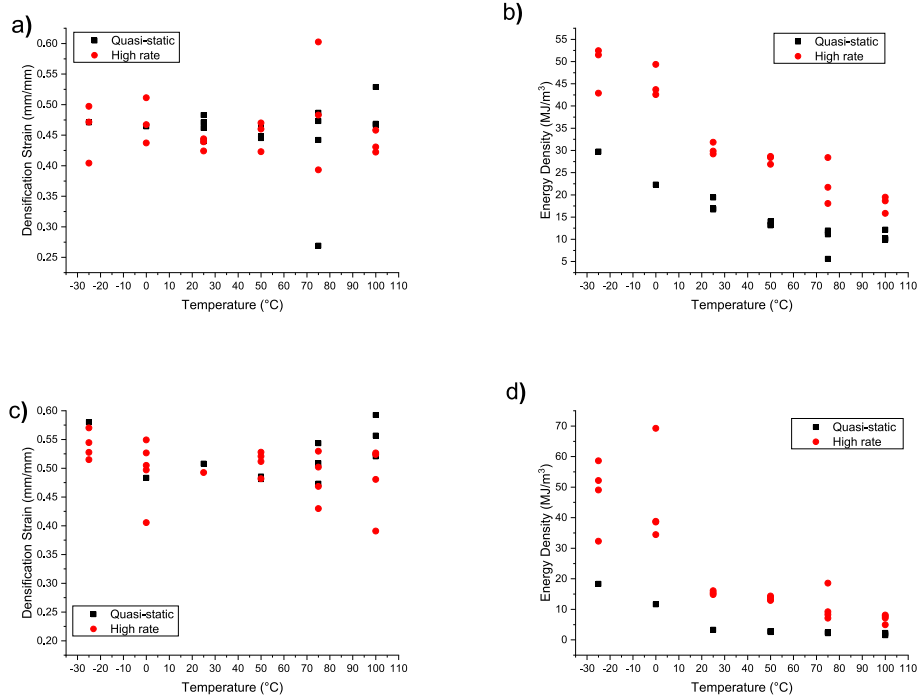


Fig. 17. Temperature and strain rate dependence of densification properties. Densification strain and energy density of (a,b) Hytac® XTL and (c,d) polyurethane syntactic foam.

Typical mechanical responses measured at room temperature at quasi-static and high rate of strain are reported in Fig. 15. The Hytac® XTL foam exhibit an evident rate dependency of the compressive strength and initial stiffness. The compressive collapse stress increases from 46 MPa to 82 MPa as the strain rate increases from 10^{-3} s^{-1} to approximately $3 \times 10^3 \text{ s}^{-1}$ (Fig. 15a). The Hytac® C1R foam also displays a clear rate dependence with the compressive strength increasing from 108 MPa in the quasi-static regime to 170 MPa at high strain rate (Fig. 15b) while the stiffness of the material appears to be nearly unaffected by the strain rate. The rate dependence of the polyurethane syntactic foam studied is even more evident with the compressive collapse strength increasing to approximately four times the strength measured during quasi-static compression (Fig. 15c).

A comprehensive summary of the strain rate and temperature dependence measured in compression on the three materials is provided in Fig. 16. Fig. 16a–c report representative responses measured during dynamic experiments at different temperatures, while Fig. 16d–f summarise the average compressive strengths of all experiments grouped by testing condition, i.e. temperature and strain rate. In this context, the compressive strength for the brittle foam Hytac® C1R is defined as the maximum absolute compressive stress in the engineering stress-strain curve.

Similarly, to the quasi-static regime, the plots outline a noticeable temperature dependency of the measured high-rate responses. The dynamic compressive collapse strength of the Hytac® XTL (Fig. 16a,d) foam increases from approximately 80 MPa at room temperature, to 102 MPa and 126 MPa at 0 °C and –25 °C respectively while it reduces to 53 MPa at 100 °C. Additionally, the diagram in Fig. 16d shows that the rate dependency of the material is affected by the testing temperature, as the increment in strength measured in the dynamic regime appears to be slightly larger at low temperatures (0 °C and –25 °C). This may be because the glass transition of the material occurs between 1 °C and 63 °C.

Fig. 16b,e summarise the temperature and rate dependence of the Hytac® C1R Foam. The dynamic strength of this material is affected by the imposed temperature, although in a lesser extent compared to the other materials examined in this study. The dynamic compressive

strength decreases from approximately 170 MPa to around 125 MPa from room temperature to 100 °C, while it increases to approximately 200 MPa at –25 °C. The rate dependency of the material appears substantially unaffected by the testing temperature, as the increase in strength appears approximately constant across all temperatures (Fig. 16e).

The temperature and rate dependence of the polyurethane syntactic foam examined in this study is detailed in Fig. 16c,f. The temperature dependency of the dynamic response is particularly evident, with a considerable increment of the compressive collapse strength at cryogenic temperatures. This increases from approximately 40 MPa at room temperature to around 90 MPa and 110 MPa at 0 °C and –25 °C, whilst it reduces to less than 20 MPa at 100 °C. It is also evident from Fig. 16f that the rate dependence of the material is considerably affected by the temperature, as the increment in strength observed at 0 °C and –25 °C is substantially larger than at other temperatures. This may be explained by the fact that the lowest temperature imposed during the experiments (–25 °C) is relatively close to the glass transition temperature range of the material.

The energy absorbed during deformation of the Hytac XTL and of the PU syntactic foam at different temperatures and strain rates is analysed and compared in Fig. 17. Here the densification strain is defined by the maximum of the energy absorption efficiency [56,57]:

$$\eta = \frac{1}{\sigma(\epsilon)} \int_0^{\epsilon} \sigma(\epsilon) d\epsilon \quad (6)$$

The energy density, i.e. the area under the stress-strain curve up to the densification strain, is computed to evaluate the energy absorption performance of the two foams. Figs. 17b and d illustrate how this is affected by temperature and strain rate.

The experimental data show that the densification strain appears not to be affected by strain rate and temperature as the variability of the measured dataset does not show any identifiable trend and it is, therefore, attributed to experimental scatter (Fig. 17a,c).

Fig. 17b shows that the energy density of the Hytac® XTL foam is, as expected, dependent on both the temperature and the strain rate. The data measured at quasi-static and high rate of strain identify two trends,

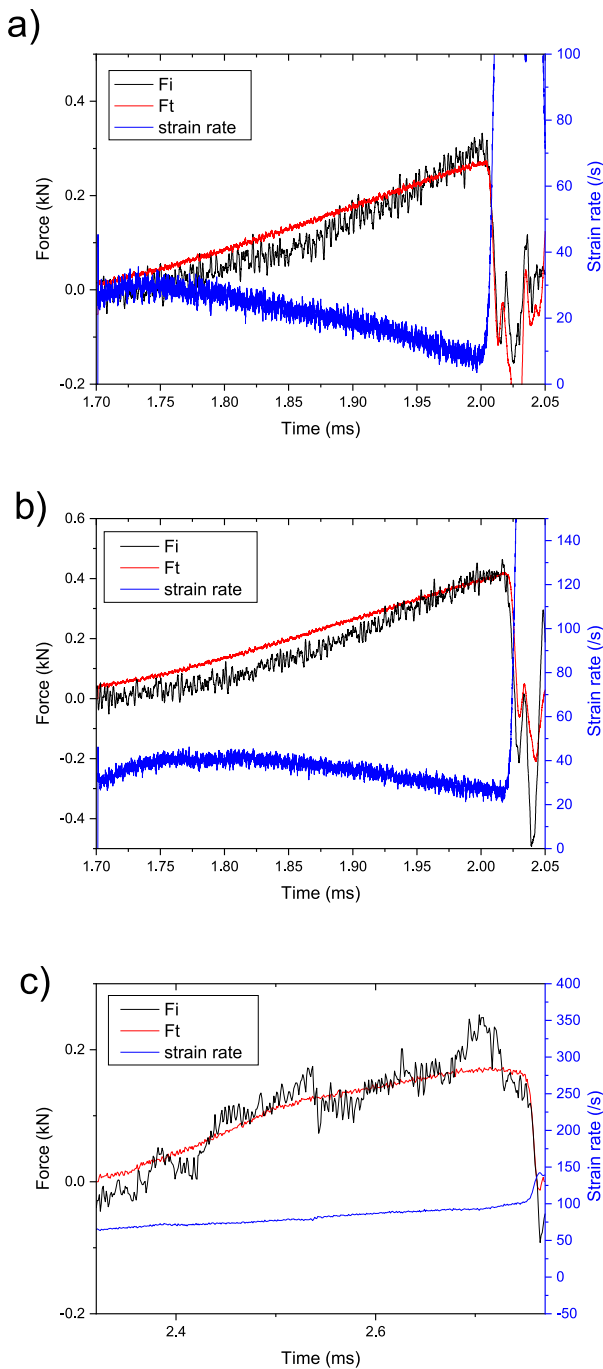


Fig. 18. Representative forces in the incident and transmitted bars and strain rate histories during high-rate tension experiments carried out at room temperature: (a) Hytac® XTL syntactic foam (b) Hytac® C1R syntactic foam and (c) Polyurethane syntactic foam.

approximately parallel to each other, but with a slight divergence observed at sub ambient temperatures. It can be therefore concluded that the rate dependency of the energy density is approximately constant for the range of temperatures considered in this study, with a gradual increase towards the sub ambient temperatures.

Fig. 17d illustrates the energy densities measured during quasi-static and dynamic experiments at different temperatures on the polyurethane syntactic foam. The experimental data highlight a strong dependency on both temperature and strain rate. It is evident that, in this case, the rate dependency of the energy density is affected by the temperature, as the

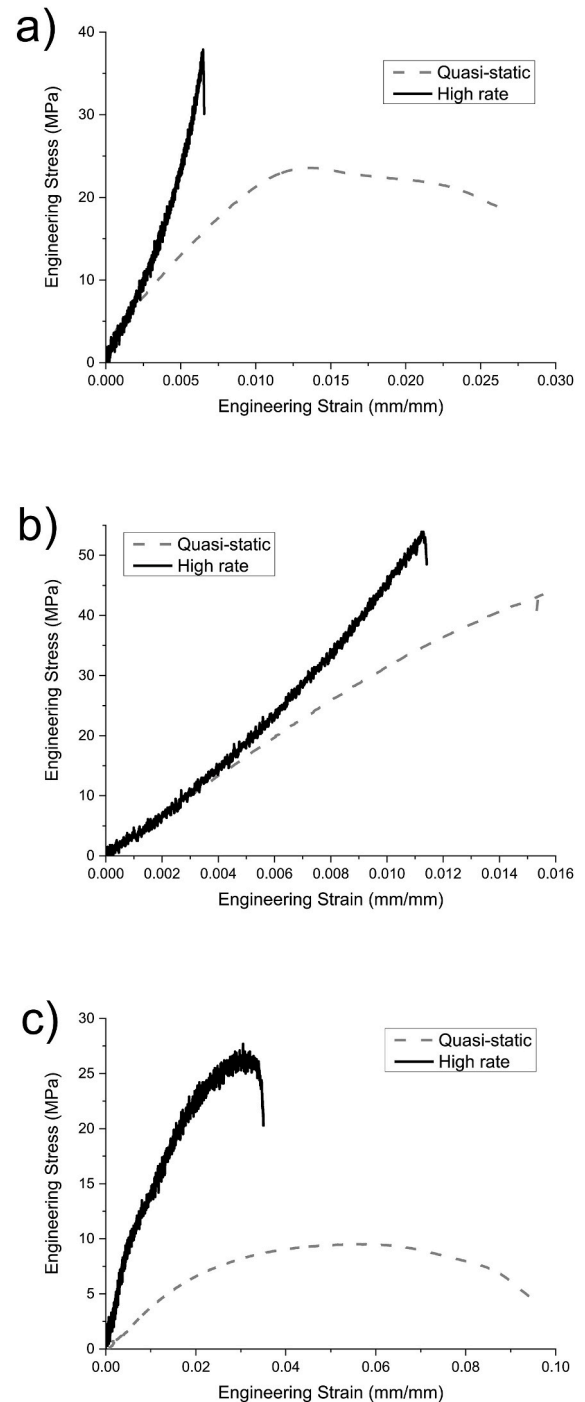


Fig. 19. Mechanical response measured at room temperature during quasi-static and high strain rate tension experiments: (a) Hytac® XTL syntactic foam (b) Hytac® C1R syntactic foam and, (c) Polyurethane syntactic foam.

trends identified by the quasi-static and high-rate datasets appear to diverge at sub-ambient temperatures.

5.4. High-rate tension

This section summarises the experimental results obtained during high-rate tension experiments at different temperatures on the materials under investigation.

Force equilibrium conditions are particularly difficult to achieve during dynamic tensile loading, due to the larger dimensions of the samples and to the generally lower strains to failure. Fig. 18 reports the

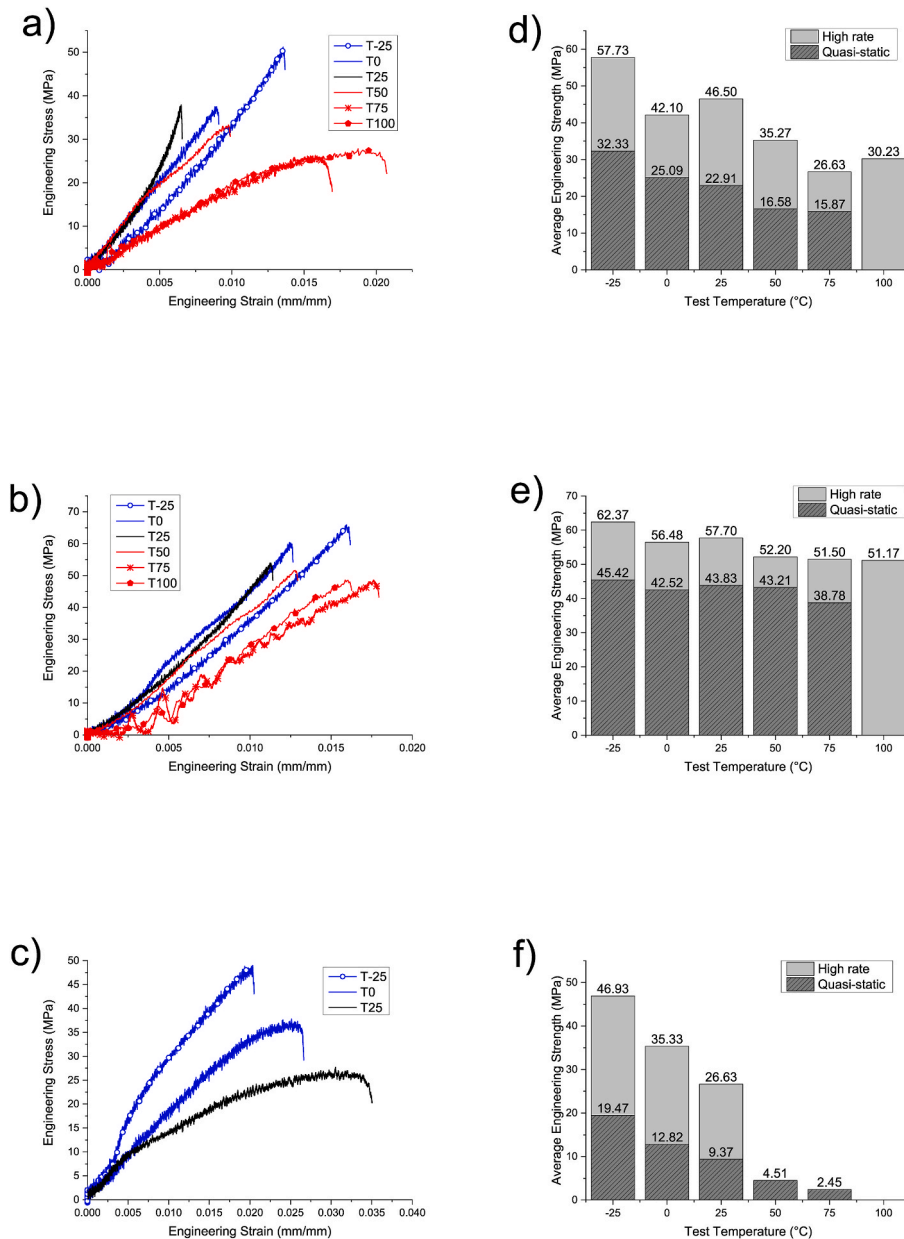


Fig. 20. Temperature dependence of the high-rate tensile response of: (a) Hytac® XTL syntactic foam (b) Hytac® C1R syntactic foam and, (c) Polyurethane syntactic foam. Rate dependence of the average tensile strength measured at different test temperatures: (d) Hytac® XTL syntactic foam (e) Hytac® C1R syntactic foam and, (f) Polyurethane syntactic foam.

time histories of the forces acting at the input and output interfaces of the sample during representative room temperature experiments, as well as the corresponding strain rate.

Dynamic equilibrium conditions were attained on all experiments reported in this study. It is noted, however, that the experimental results corresponding to experiments conducted at 50 °C, 75 °C and 100 °C on the polyurethane syntactic foam are not reported, as it was not possible to achieve satisfactory force equilibrium conditions at these temperatures.

Figs. 18a and b presents incident and transmitted forces measured during room temperature experiments on the Hytac® XTL and Hytac® C1R foams. It is evident that dynamic equilibrium conditions were attained at the very early stages of deformation. The time histories of the strain rate show that, to ensure the validity of the measured experimental results, the strain rates were limited to the range between 20 s^{-1} and 40 s^{-1} . This was achieved limiting the striker velocity and using 1

mm thick neoprene pulse shapers.

Fig. 18c presents the history of the forces acting on the two sides of the sample during a dynamic experiment on the polyurethane foam. The incident force oscillates around the transmitted force showing that force equilibrium was achieved during the room temperature dynamic experiments.

Fig. 19 reports the results of representative room temperature experiments conducted on the three materials during quasi-static and dynamic experiments. It is evident that the Hytac® XTL foam is characterised by a pronounced rate dependence of both the tensile strength and stiffness (Fig. 19a). The tensile strength increases from approximately 24 MPa in the quasi-static regime to 38 MPa in dynamic loading conditions. The Hytac C1R epoxy-based syntactic foam exhibit a more moderate rate dependence with the tensile strength increasing from 44 MPa to 54 MPa from quasi-static to dynamic loading conditions (Fig. 19b). The tensile stiffness also appears to be affected by the strain

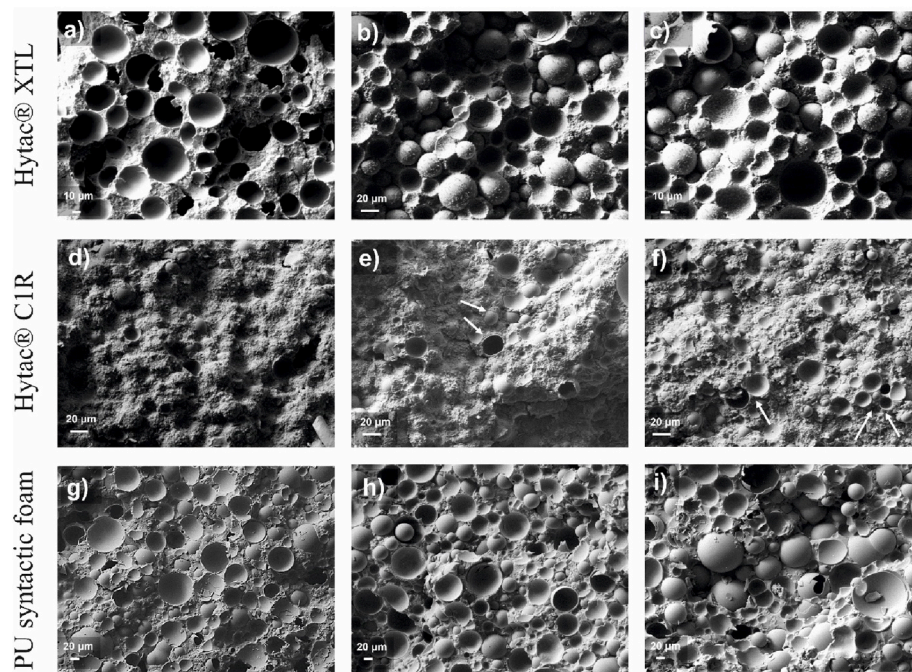


Fig. 21. Micrographs illustrating the failure surface of tensile samples tested in dynamic loading conditions. Failure surface of Hytac® XTL polyamide syntactic foam deformed at (a) -25 , (b) 25 °C and (c) 75 °C; failure surface of Hytac® C1R epoxy copolymer syntactic foam deformed at (d) -25 °C (e) 25 °C and (f) 75 °C; failure surface of polyurethane syntactic foam deformed at (g) -25 °C and (h), (i) 25 °C.

rate, although to a lesser extent when compared to the Hytac® XTL. Similarly to what observed in relation to the compression experiments, the polyurethane syntactic foam displays a very pronounced rate dependence, with the dynamic tensile strength increasing to approximately three times the value measured during low rate experiments and an apparent increase of stiffness (Fig. 19c).

Fig. 20 presents a summary of the temperature and rate dependence measured during tensile experiments on the three syntactic foams investigated in this work. Representative stress-strain curves measured during dynamic tensile experiments at different temperatures are reported in Fig. 20a–c. Fig. 20d–f outlines the average tensile strength measured during all experiments, categorised by temperature and loading rate. In this context, tensile strength is defined as the ultimate stress in the engineering stress-strain response.

The plots delineate an observable temperature dependence of the dynamic tensile response, particularly for the Hytac® XTL and for the polyurethane syntactic foam. The average dynamic tensile strength of the Hytac® XTL material (Fig. 20a,d) increases from approximately 46 MPa at room temperature to 58 MPa at -25 °C, while it reduces to approximately 30 MPa at temperatures between 75 °C and 100 °C. Differently from what observed in compression experiments, the rate dependence of the material does not appear to be affected significantly by the testing temperature, as the increment in strength measured in the dynamic regime does not vary significantly at different temperatures.

Fig. 20b,e provide a summary of the temperature and rate dependence of the tensile strength for the Hytac® C1R Foam. The dynamic tensile strength of the material is only mildly influenced by the testing temperature. The dynamic tensile strength decreases from approximately 58 MPa at room temperature to 51 MPa between 75 °C and 100 °C, while it increases to approximately 62 MPa at -25 °C. Similarly to what observed in compression experiments, the rate dependency of the material does not appear to be influenced by the testing temperature, as the increment in strength due to the strain rate appears to be approximately constant across all testing temperatures considered in this study.

The temperature and rate dependent tensile behaviour of the examined polyurethane foam is displayed in Fig. 20d,f. The effect of the testing temperature on the dynamic tensile response of the material is

conspicuous. The dynamic tensile strength of the PU foam increases from approximately 27 MPa at room temperature to around 35 MPa and 47 MPa at 0 °C and -25 °C respectively. The rate dependence is moderately influenced by the prescribed testing temperature as the difference in strength between dynamic and quasi-static conditions appears to increase slightly at lower temperatures. It must be noted that the dynamic tensile characteristics of this foam at temperatures above the room temperature are not reported as, although the images show substantially uniform deformations, dynamic equilibrium conditions could not be rigorously measured during the experiments.

Fig. 21 illustrates the failure surface of tensile samples deformed in dynamic loading conditions. The influence of strain rate and temperature on the failure mode of the Hytac® XTL is delineated in Fig. 21a–c. The increase in the strain rate in dynamic experiments leads to a noticeable rise in the number of fractured micro-balloons, particularly pronounced in samples tested at -25 °C (Fig. 21a). Additionally, the fracture surface of the polyamide matrix appears rugged. This distinct observation, unlike in quasi-static loading conditions, points to a brittle failure even in specimens tested at room temperature and 75 °C.

Fig. 21d–f outline the fracture surfaces of the Hytac® C1R tensile samples deformed during dynamic experiments conducted at different temperatures. The micrographs show a brittle failure of the matrix, where the majority of microspheres are embedded within it. Notably, the number of micro-balloons visible on the failure surface seems to decrease compared to quasi-static loading conditions. Lastly, the micrographs reveal several fractured microspheres, as highlighted by arrows in Fig. 21e and f. These fractures appear to increase slightly with rising temperatures, suggesting that the reduction in stiffness observed in samples tested at 75 °C could be attributed, at least in part, to micro-balloon fractures.

The failure surface of tensile samples made from the PU foam under investigation is displayed in Fig. 21g–i. The effect of the elevated strain rate becomes apparent through a distinct reduction in ductility, emphasised by the irregular surface of the failed matrix. Moreover, there is an increase in the number of micro-balloons exhibiting fractures, particularly noticeable in samples tested at -25 °C (Fig. 21g).

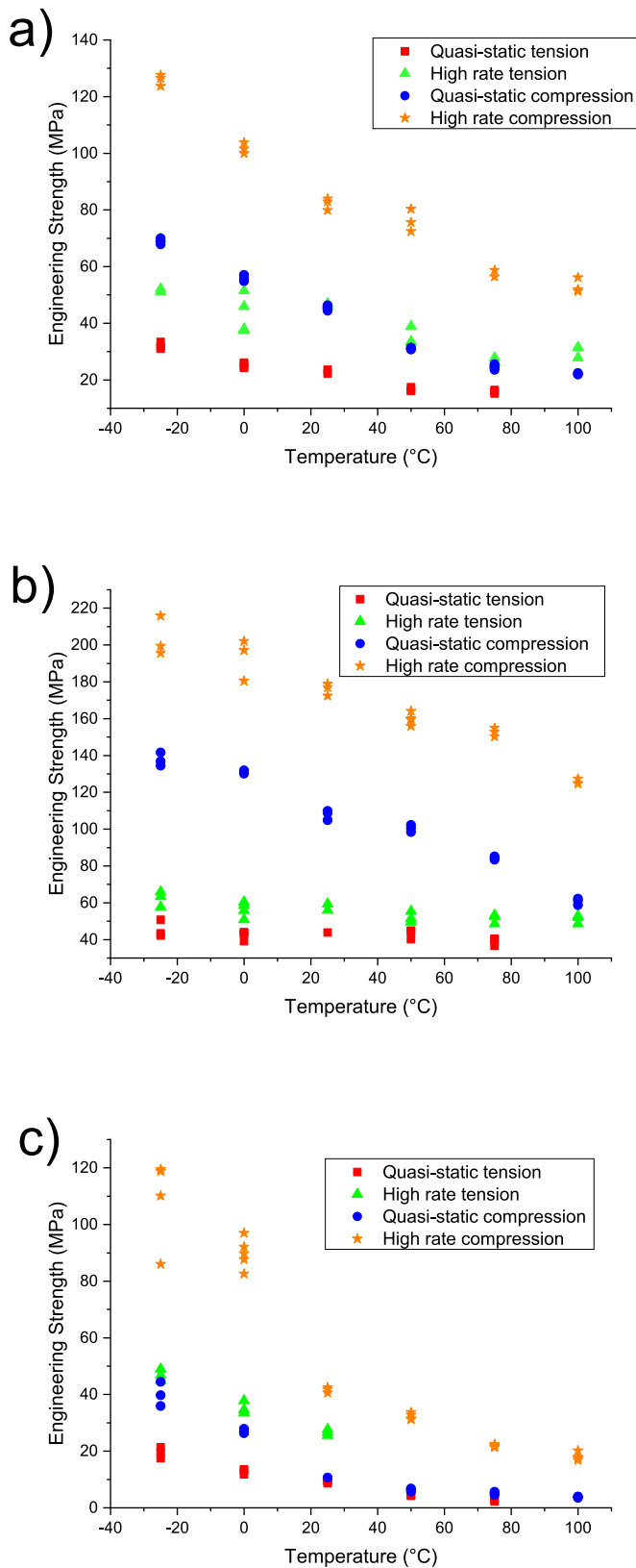


Fig. 22. Tensile and compressive strengths measured during quasi-static and dynamic loading experiments conducted at different temperatures on (a) Hytac® XTL syntactic foam (b) Hytac® C1R syntactic foam and, (c) Polyurethane syntactic foam.

5.5. Summary of experimental results

The entire set of results obtained in this study is grouped in Fig. 22 to provide a comprehensive summary of the experimental results and an assessment of their scatter. The figure displays the compressive collapse strength and the tensile strength in the different experimental conditions prescribed in this study. It can be observed that the variability of the strength data measured in different conditions is minor, particularly considering the nature of the materials under investigation, highly sensitive to environmental and loading rate conditions. It is also apparent that the three syntactic foams examined are characterised by a noticeable strength asymmetry, being the compressive collapse strength significantly higher than the tensile failure stress in all conditions.

It must be noted that, as indicated in Fig. 22, the strain rates measured during dynamic tension and compression experiments were considerably different. This is because force equilibrium conditions were achievable only at strain rates in the range of 20 s^{-1} – 100 s^{-1} in tension, while validity conditions were attained at strain rates in the order of $1\text{--}3 \cdot 10^3 \text{ s}^{-1}$ in compression.

Fig. 22a highlights the strong rate dependence of the Hytac® XTL polyamide foam, both in tension and compression. The material also exhibits an evident temperature dependence. The foam does not exhibit a noticeable interaction between the effects of temperature and strain rate, as the increase in strength measured in dynamic conditions does not vary significantly with the prescribed testing temperature.

The Hytac® C1R syntactic foam is characterised by a modest rate dependence in tension, while the effect of the loading rate is more pronounced in compression (Fig. 22b). The foam does not show any significant interaction between temperature and rate dependence, as demonstrated by the approximately constant effect of the strain rate across the range of temperatures examined in this study.

Fig. 22c shows that the mechanical response of the polyurethane foam examined in this study is strongly influenced by both temperature and strain rate. The plot highlights a noticeable coupling between the two effects, as the dynamic increment in strength measured at sub-ambient temperatures is significantly larger than at higher temperatures.

It is valuable to plot the ratio between the stress measured during high strain rate experiments to that measured during quasi static experiments at different strains levels, to assess the variations of the rate dependency during the deformation process. Fig. 23 displays the ratios of stress measurements for experiments conducted in tension and compression on the three materials under investigation. Notably, the ratio remains relatively stable for experiments in compression, whereas it exhibits variations in tension (Fig. 23b,f). These variations can be attributed to changes in stiffness at different strain rates and the relatively brittle failure of the materials in tension.

6. Conclusions

The present investigation details the temperature and strain rate dependent mechanical behaviour of three syntactic foams comprising of a polymer matrix and hollow glass micro-balloons. The three materials are a polyurethane, an epoxy and a nylon syntactic foams. Their behaviour was investigated under compressive and tensile loads. The main conclusions of this study are summarised below:

- The experimental results denote a noticeable temperature and rate dependent mechanical response of all investigated materials. The three syntactic foams examined are characterised by a noticeable strength asymmetry, with the compressive response significantly higher than the tensile.
- The densification strain of the polyamide and polyurethane syntactic foams does not show any appreciable rate dependence, while densification does not occur on the epoxy based syntactic foam.

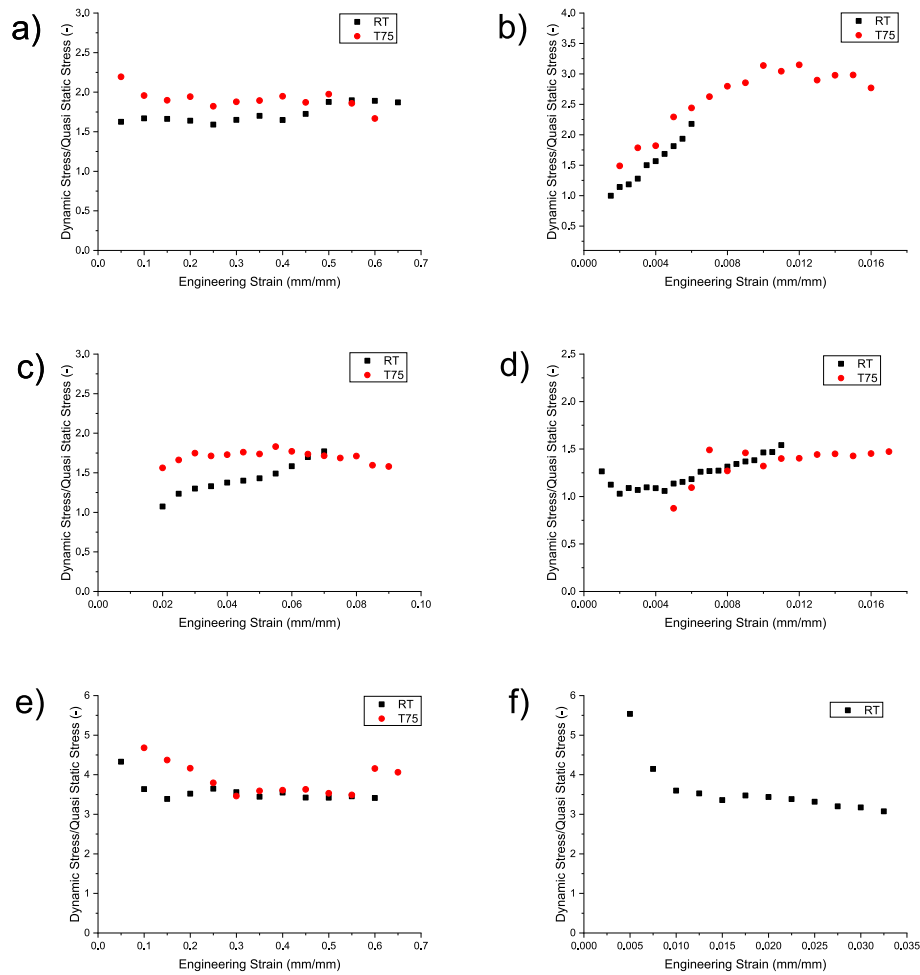


Fig. 23. Ratio between the dynamic and quasi static stress at different strains for experiments conducted at room temperature and 75 °C: (a) Hytac® XTL compression (b) Hytac® XTL tension (c) Hytac® C1R compression (d) Hytac® C1R tension (e) polyurethane foam compression (f) polyurethane foam tension.

- The experimental results, collected over a temperature range spanning from $-25\text{ }^{\circ}\text{C}$ to $100\text{ }^{\circ}\text{C}$ at low and high strain rates, elucidate a distinct interaction between the influences of temperature and strain rate on the mechanical characteristics of the materials being studied. The synergy between temperature and strain rate is observed to vary based on both the matrix material and the loading mode. More in detail:
 - o *Nylon syntactic foam.* In compression the rate dependency of the nylon syntactic foam is affected by the testing temperature, as the increment in strength measured in the dynamic regime appears to be slightly larger at low temperatures ($0\text{ }^{\circ}\text{C}$ and $-25\text{ }^{\circ}\text{C}$). Conversely in tension the rate dependency of the material does not appear to be affected significantly by the testing temperature.
 - o *Polyurethane syntactic foam.* In compression the rate dependency of the material appears to be considerably affected by the temperature, as the increment in strength observed at $0\text{ }^{\circ}\text{C}$ and $-25\text{ }^{\circ}\text{C}$ is substantially larger than at other temperatures. Differently, in tension the rate dependency of the material is only moderately influenced by the prescribed testing temperature, with the difference in strength between dynamic and quasi-static conditions showing a slight increase at lower temperatures.
 - o *Epoxy syntactic foam.* the rate dependency of the epoxy syntactic foam remains largely unaffected by the testing temperature both in tension and compression.
- The microstructure of the materials was analysed via optical microscopy and SEM micrographs. The analysis of the failure surfaces

of tensile samples subjected to quasi-static and dynamic deformation at various temperatures provided insights into the deformation and failure mechanisms of the matrix, micro-balloons, and their interfaces for the three materials under investigation. It is evident that the testing temperature visibly influences the deformation and failure of the matrix, while the strain rate impacts both the ductility of the matrix and the number of fractured micro-balloons.

Author contributions

Yue Chen: Investigation; Methodology; Visualization; Formal analysis; Writing - original draft. Gustavo Quino: Formal analysis; Conceptualization; Methodology; Validation; Writing - review & editing. Antonio Pellegrino: Supervision; Methodology; Conceptualization; Investigation; Funding acquisition; Writing - review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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