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## Effect of Crumb Rubber on Mechanical Properties of Multi-phase

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## **Syntactic Foams**

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## 8 Abstract

9 Syntactic foam is a lightweight and strong material which can be used in marine and 10 aeronautical applications. However, the brittleness of the material limits its application to a broader range. Adding crumb rubber to the syntactic foam can increase its energy absorption 11 capacity. The effect of crumb rubber on the fracture toughness and energy absorption capacity 12 of 2-phase and 3-phase syntactic foam is evaluated under both static and impact loads. The 13 experimental results have shown that the fracture toughness of the 2-phase rubberized 14 syntactic foam increased by 8% while an increase of 22% of its fracture energy was observed. 15 Under quasi-static loads, the 3-phase rubberized syntactic foam showed decreases in the 16 compressive strength and elastic modulus but an increase in the energy absorption capacity as 17 compared to the syntactic foam without crumb rubber. In addition, the impact energy 18 19 absorption of the 3-phase rubberized syntactic foam increased by 24% as compared to that of the 3-phase syntactic foam without crumb rubber. 20

Keywords: Syntactic foam; Impact behaviour, Energy absorption; Fracture toughness; Crumb
rubber.

#### 23 **1. Introduction**

Syntactic foam is a type of lightweight and rigid composite material, which consists of binder 24 and fillers. The binder matrix can be made of polymeric resin, metal and ceramic [1] and the 25 26 fillers are in forms of microsphere and macro-sphere, which are made from rigid materials 27 such as glass, carbon, metal, ceramic, cenosphere and polymeric materials [2]. The syntactic foams are commonly categorized as 2-phase and 3-phase [3]. It is noted that this classification 28 29 is based on the main compositions of the material regardless the additive (e.g. crumb rubber). 30 Traditional foam is mainly made of binder matrix with relatively low compressive strength. 31 Therefore, microspheres are mixed with binder matrix to form the 2-phase syntactic foam. 3-32 phase syntactic foam is made of microspheres mixed binder matrix dispersed with macro-33 spheres, which can be gaseous voids or hollow spheres [4]. The macro-sphere, as reinforcing filler of syntactic foam, can be made of spheres coated with fibre reinforced epoxy. For 34 35 instance, Wu et al. [3] developed a macro-sphere by coating Expanded Polystyrene (EPS) beads with carbon fibre reinforced epoxy using rolling ball method. 36

37 The superior mechanical properties of the syntactic material can be obtained through the composite action [5-7]. It should be noted that the effect of the volume fraction of 38 39 microsphere on the mechanical properties of syntactic foam is not well understood [8, 9]. 40 Swetha and Kumar [2] found that the strength of the foam decreased with the increase of 41 microsphere content. Its energy absorption capacity kept increasing with the rising content of 42 microsphere up to 40% and then decreased. Kim and Khamis [10] observed that the 43 increasing volume fraction of the microsphere in the microsphere epoxy resin composites 44 improved its impact performance while decreased the fracture toughness and flexural strength. 45 However, Wouterson et al. [11] reported the opposite testing observations, i.e., the presence of microsphere increased the fracture toughness but decreased the impact resistance capacity 46

47 of syntactic foam. The strain rate effect of syntactic epoxy foam material has been investigated under ambient temperature [12-14]. The strain rate effect of expanded 48 49 polystyrene foam under high temperature has been also investigated [15]. The failure strength of polyurethane foam exhibited nonlinear strain-rate dependency [16, 17]. To improve the 50 51 mechanical properties, crumb rubber has been added into syntactic foam [18-20]. The rubber 52 can enhance impact energy absorption through elastic deformation of rubber and preventing micro-cracks from developing into macro-cracks. It is noted that replacing the microspheres 53 54 by the crumb rubber can increase the energy absorption capacity but slightly decrease the compressive strength [21]. Bagheri and Pearson [21] found that using 10% of crumb rubber is 55 the optimal value. Further volume fraction of crumb rubber (e.g. 15%) showed a reduction in 56 57 the fracture toughness. Maharsia et al. [18] found the presence of 2% rubber particles (40-75 μm) by volume quantity increased the flexural strength and energy absorption of syntactic 58 59 foam. Bagheri et al. [22] conducted a critical review of the effect of crumb rubber on the fracture toughness of 2-phase syntactic foam and found that the optimal value of the crumb 60 61 rubber volume fraction ranges between 10% and 20%.

The syntactic material can find applications owing to its characteristics of thermal efficiency, 62 63 lightweight and high compressive strength and toughness [23, 24]. The syntactic foam material has been intensively employed for marine applications including deep-water 64 exploration, which needs to withstand enormous water pressure while provide sufficient 65 buoyancy [3, 25]. Sandwich structure made of two thin stiff face-sheets and various thick 66 67 cores is used to absorb energy and resist loads. The cores can be made of lightweight 68 materials such as metal foam, polymer foam and lattice materials etc. For instance, the 69 syntactic foam material with aluminium matrix can be used as protection system in military 70 vehicles to withstand blast and impact loads and protect the passengers [26]. The material has 71 the potential for infrastructural protection of vehicle roadside barrier as energy absorption

device, which can effectively reduce the impact force [27]. In addition, the lightweight material can be used to protect the offshore structure against ship impact and underwater impact of the pipeline caused by dropping objects. By considering its great potential for impact applications, the behaviours of syntactic foam under impact are worth studying. However, the research on the dynamic response of syntactic material subjected to impact loading is limited and some contradicted findings pertaining to the presence and volume fraction of microsphere on the mechanical properties of syntactic foam were reported.

This study experimentally investigates the behaviours of four types of syntactic materials, associated with/without crumb rubber and with (3-phase)/without (2-phase) macro-spheres, subjected to quasi-static and impact loads. The fracture toughness and static/impact energy absorption of the syntactic foams are experimentally investigated.

#### 83 **2. Production of the syntactic foam**

#### 84 2.1. Composition and properties of materials

Four types of syntactic foam are investigated in this study. They are classified into 2-phase and 3-phase syntactic foams and which are further divided into two types of white (without crumb rubber) and black (with crumb rubber) materials. The 2-phase syntactic foam includes epoxy and glass microspheres (~ 50  $\mu$ m diameter) with/without crumb rubber. The syntactic foam without crumb rubber is named as white material while the one with crumb rubber is called black material. Carbon fibre reinforced macro-spheres (~ 10 mm diameter) were added to the 2-phase syntactic foam to form 3-phase syntactic foam.

92 The carbon fibre reinforced macro-spheres had the diameter of 10 mm as presented in Fig. 1.
93 The macro-spheres were coated with carbon fibre to significantly improve their compressive
94 strength. The macro-sphere production is usually a commercial secret of a marine equipment

95 production company. The epoxy and glass microspheres were supplied by Matrix [28]. The glass microspheres appear as free flowing white powder to naked eyes. They were made of 96 Borosilicate glass with the density ranging from 100 kg/m<sup>3</sup> to over 1000 kg/m<sup>3</sup>. The average 97 diameter of the glass microspheres is approximately 50 µm. The crumb rubber was produced 98 99 from recycled car tyres so that it was a mixture of different blends of rubbers and fillers. A 100 laser diffraction particle size test was conducted on the crumb rubber according to ISO 13320 101 [29]. The distribution of crumb rubber particle size is presented in Fig. 2. The composition of 102 these component materials is presented in Table 1. The volume fraction of the crumb rubber 103 of 15% was decided after conducting a review of its optimal value as presented in the previous study [22]. The compressive strength and modulus of the Matrix epoxy were 100 104 105 MPa and 2750 MPa, respectively. The density of the white and black 2-phase syntactic foam 106 was 770 and 920 kg/m<sup>3</sup>, respectively.

#### 107 2.2. Production of samples

108 The production of the 2-phase syntactic foam is well presented in the previous study [22] so that this section does not repeat the production process and only describes the procedure of 109 110 manufacturing the 3-phase syntactic foam. The required amount of carbon fibre reinforced macro-spheres (60% packing density) was put into a steel mould with the size of 100 mm x 111 112 200 mm. It is noted that a random packing of spheres is based on the previous study by He et 113 al. [30]. If the close random packing is applied for equal particles, the packing density approaches 63% [30]. Due to a high surface-area-to-volume ratio of these specimens, the 114 115 packing density in this study was approximately 60%. The mixture of the binder was prepared 116 from the Matrix epoxy blend and Matrix glass microspheres with/without crumb rubber. There were two types of the binder used in this study, including the white and black binders. 117 118 The black binder contained crumb rubber while the white binder did not. The binder was then

injected into the mould in a vacuum condition. The mixture was cured at 80°C overnight, followed by a post cure at 130°C for 2 hours. The production process of the samples is presented in Fig. 3. The density of the 3-phase syntactic foam for the black and white specimens was approximately 501 kg/m<sup>3</sup> and 419 kg/m<sup>3</sup>, respectively.

123 2.3. Microstructure

124 To check the syntactic foam structure, two different techniques were carried out, which included a Nikon SMZ800 stereomicroscope with a Schott KL1500LCD light source and 125 scanning electron microscope (SEM). The Toupcam UCMOS 14000KPA digital camera 126 associated with ToupView 3.7 software was used to capture the images. The microstructure of 127 the 2-phase syntactic foam is presented in Fig. 4. There was only micro-sphere particles in the 128 129 white specimen as shown in the left picture. Meanwhile, there were two types of particles in 130 the black material including glass micro-spheres and rubbers as shown in the right picture. The twinkling particles represent glass micro-spheres and the grey particles stand for crumb 131 132 rubber. The composition of the particles and the even distribution of the crumb rubber and 133 other particles are shown in Fig. 4. The bonding between particles and binder plays an essential role in the structural performance of the material. Thus, the rough surface of the 134 135 macro-sphere is to increase the bonding between the binder and the macro-spheres as shown in Fig. 5. The roughness of the surface is found to be significant for improving the bonding 136 between binder and particles [8, 31, 32]. The interface bonding between the macro-spheres 137 138 and the binder is examined after tests and presented in the later part.

- 139 **3. Fracture Toughness Testing**
- 140 3.1. Specimens and testing apparatus

141 The fracture toughness tests were conducted on the 2-phase syntactic foam including white 142 (without crumb rubber) and black (with crumb rubber) materials. Two slabs were prepared 143 using a PTFE lined steel mould with dimension of 300 mm long x 200 mm wide x 45 mm 144 deep. Ten notched beams were prepared for each type of material as presented in Fig. 6. Each 145 face of the specimens was milled square and parallel to create 20 mm x 40 mm x 180 mm cuboids, followed by a second milling process to form the slot. The apparatus and testing 146 procedure comply with ASTM D5045 [33]. All specimens were stored in the laboratory to 147 148 equilibrate to standard laboratory conditions for at least 3 weeks. Immediately prior to testing, 149 a final "sharp" crack was formed at the tip of the slot by resting the cutting edge of a "box" cutter" knife along the length of the slot and applying a moderate pressure by hand. It is noted 150 151 that there was no cutting or scoring motions were applied. An indentation test was carried out 152 for each material to identify the compliance of the test apparatus and the proportion of the 153 strain energy developed in each test that could be attributed to the indentation by the rollers.

#### 154 3.2. Results and discussions

The experimental results from the fracture toughness tests are respectively presented in Tables 2 and 3 for the white and black materials, respectively. It is noted that the energy correction due to the indentation of the rollers was measured as 0.019 and 0.034 J for the white and black materials, respectively. In general, the specimens failed at a cross head deflection of 0.75 mm with the loading rate of 10 mm/min [33]. To ensure the validity of the tests, the size of the specimen is chosen so that the yield stress must be greater than the minimum yield stress.

162 
$$\sigma_{\min} = K_{Ic} \sqrt{\frac{2.5}{W-a}} = 9.7 MPa \quad for white material$$
(1)
$$= 10.4 MPa \quad for \ black \ material$$

where  $\sigma_{\min}$  is the minimum yield stress,  $K_{Ic}$  is the plane-strain fracture toughness, and W and aare the dimensions of the specimen presented in Fig. 6. The fracture toughness or critical stress intensity factor ( $K_{Ic}$ ) and the critical strain energy release rate ( $G_{Ic}$ ) are determined as follows [33]:

167 
$$K_{Ic} = \frac{P_Q}{B\sqrt{W}} f(x)$$
(2)

(3)

 $G_{Ic} = \frac{U}{BW\omega}$ 

169 where  $P_Q$  is the force determined based on ASTM D5045 [33], *B* is the thickness of the 170 specimen shown in Fig. 6, f(x) is the function of the ratio between the crack length and the 171 specimen depth,  $\varphi$  is the energy calibration factor, and *U* is the corrected energy. The 172 corrected energy (*U*) results from subtracting the energy-to-peak by the energy caused by the 173 indentation displacement.

As presented in Tables 2-3, the average critical stress intensity factors or fracture toughness 174  $(K_{\rm Ic})$  for the white and black materials are 0.864 and 0.933 MPa  $\sqrt{m}$ , respectively. The 175 increase of the critical stress intensity factor of the black material was about 8% as compared 176 to the white material. The mean values of the critical strain energy release rate ( $G_{Ic}$ ) of the 177 white and black material are 0.332 and 0.404 kJ/m<sup>2</sup>, respectively. It results in an increase by 178 179 22% in the critical strain energy release rate of the black material as compared to the white one. As expected, replacing the microspheres by crumb rubber increases the fracture 180 181 toughness and the critical strain energy release rate of the materials. The results are consistent 182 with previous studies [2, 10].

183 It is worth mentioning that micro-length scale damage is more beneficial to the energy 184 absorption than macro-length scale damage. For instance, several micro-cracks may absorb 185 the same amount of energy as one macro-crack. The macro-crack may fragment the material

186 while the micro-cracks may only degrade the structural capacity and modulus. Therefore, 187 preventing the development of the micro-cracks into the macro-cracks is the key factor to 188 improve the fracture toughness and ductility of the material. Adding crumb rubber was found to result in the crack bridging phenomenon which improves the fracture toughness [18]. 189 190 However, the increase of the fracture toughness in this study is not significant. As presented 191 in the study by Bagheri et al. [22], the optimal volume fraction of crumb rubber needs to be identified and the authors recommended the searching range was from 10% to 20% of the 192 193 volume fraction. More trial fracture toughness tests should be conducted to determine the 194 optimal value of the volume fraction of the crumb rubber suggested between 10% and 15%.

In addition, there are many uncertainties introduced in this procedure so that it needs to be verified. ASTM D5045 [33] recommended the value of  $E/(1-v^2)$  derived by two different methods can be cross checked:

198 
$$\frac{E}{(1-\nu^2)} = \frac{K_{lc}^2}{G_{lc}}$$
(4)

199 
$$\frac{E}{(1-v^2)} = \frac{\psi}{B(C_Q - C_i)}$$
(5)

where  $\nu$  and E are the Poisson's ratio and tensile modulus of the material, respectively;  $C_Q$ and  $C_i$  are the compliance from the fracture tests and the indentation test, respectively; and  $\Psi$ is the calibration factor. As recommended by ASTM D5045 [33], the value estimated from Eq. 4 should be larger, and the difference is recommended to be less than 15%. As calculated, the differences determined by two different methods are 7.6% and 16.7% for the white and black materials, respectively.

The findings in this study are consistent with those from the previous study [18]. Maharsia et al. [18] observed that there was no significant difference in the modulus and stiffness of the

208 rubberized syntactic foam (adding 2% crumb rubber) and the plain syntactic foam. The 209 authors found an approximately 18% increase in the fracture strain with the addition of crumb 210 rubber.

#### 211 **4.** Compressive behaviour under quasi-static loads

The 3-phase syntactic foam was created to have light weight and relatively high strength and 212 energy absorption capacity, thus, its compressive behaviour under static and impact loads was 213 investigated. The Baldwin machine with the capacity of 600 kN was used to carry out the 214 215 compression tests on the 3-phase syntactic foam. The loading rate was maintained at 0.5 mm/min until the specimens failed. The loading rate was carried out to comply with AS 216 1012.9 [34] for compressive strength tests. The specimens were machined at two ends to 217 218 ensure full contact between the loading heads and the specimens. The cylindrical specimens 219 were 100 mm in diameter and 200 mm in height. The applied load and displacement of the specimens were measured by the embedded load cell and linear variable differential 220 221 transformer in the machine.

The tested specimens failed by some major cracks as presented in Fig. 7. The arbitrary failure 222 223 surface of the tested specimens was observed rather than an approximately typical 45° failure 224 surface in normal concrete. This figure shows the failure surfaces intersected carbon fiber reinforced macro-spheres. Failure of the interface between the binder and the macro-spheres 225 226 was found at only one macro-sphere. In general, the interface bonding was sufficient. All the macro-spheres in the failure surface were damaged. As a result, the failure of the macro-227 spheres also governs the failure of the specimens. This failure mode was also observed in the 228 229 previous study [3]. Since the binder and the macro-sphere govern the failure, adding crumb 230 rubber in the binder, which is not a huge volume fraction, may not considerably affect the

strength and energy absorption of the material. As a result, the behaviour of the white andblack materials did not show a significant difference.

233 The stress-strain curves of the tested cylinders are presented in Fig. 8. The stress-strain curves of the syntactic foam increase linearly up to about 12 MPa with the corresponding strain of 234 235 1.7% The stress-strain curves of the tested specimens dropped along with the specimens failure, showing a very brittle manner, which is different from those reported by Wu et al. 236 237 [3]. The 3-phase syntactic foam in the study by Wu et al. [3] failed in a more ductile manner, where the specimens reached the maximum stress at the strain of 5% and then the stress 238 239 significantly dropped. As shown in Fig. 8, the slope of these curves started to decrease after 240 reaching 12 MPa and the white and black specimens reached the maximum stresses of 16.5 241 and 15.5 MPa, respectively. Adding crumb rubber led to 6% reduction in the compressive strength of the rubberized syntactic foam as compared to the white one. The axial strains 242 243 corresponding to the maximum stresses of the white and black specimens were 2.9% and 3.1%, respectively. The energy absorption, defined by the area under the force-displacement 244 curves of the two specimens are 0.37 and 0.39 kN·m for the white and black materials, 245 respectively. The energy absorption of the black specimen was about 5.4% higher than that of 246 247 the white material. The two specimens did not show a considerable difference in the static 248 behaviour. It shows that replacing 15% volume fraction of the glass microspheres by the crumb rubber does not significantly change the static behaviour of the specimens. The elastic 249 250 modulus of the syntactic foam for the white and black specimens is 748 and 627 MPa, 251 respectively.

In addition, the microstructure of the material was examined after the compression tests and presented in Fig. 9. Observation of cracks in the black material specimens was difficult so that cracks in the white material specimen are presented herein. Two types of damage modes were

found in the specimen. A separation at the interface between the binder and the macro-sphere is presented in Fig. 9a. This crack is connected with another one cutting through the binder as shown in Fig. 9b. The crack in the binder shows both damages of the epoxy resin and glass micro-sphere. The crack in the binder was stopped at the macro-sphere. The failure mechanism helps to improve the ductility and thus the energy absorption performance, which demonstrated the effectiveness of coating macro-spheres. This mechanism was also observed in the previous study for micro-spheres [20].

262 5. Compressive behaviour under impact loads

#### 263 5.1. Test setup and data acquisition system

The impact testing apparatus as shown in Fig. 10 was used to carry out drop-weight impact 264 tests. A cylindrical steel projectile weighing 97.5 kg was dropped from 3 m height onto the 265 top of the tested cylinders. This drop generated a kinetic energy of 2.87 kJ. The projectile was 266 267 designed to have a smooth flat bottom with a radius r = 50 mm. A plastic guiding tube was 268 utilized to ensure the projectile falling vertically to the targets. A load cell was placed at the 269 bottom of the specimens to measure the impact force. A high-speed camera which was set to 270 capture 20000 frames per second was used to monitor the failure process. The data acquisition 271 system controlled by a computer was used to record signals from the load cell. The data acquisition system recorded data at a sampling rate of 1 MHz. This sampling rate was adopted 272 273 according to the recommendation from a previous study [35]. Pham and Hao [35] investigated 274 the sampling rate on the results of the axial impact tests. The authors recommended that the 275 sampling rate smaller than 100 kHz may not be able to capture peak impact load and 276 responses.

#### 277 5.2. Test results and discussions

278 The high speed camera was used to monitor the failure while the image analysis was utilized to derive the displacement of the specimens. The both white and black cylinders failed in an 279 280 explosive manner when the top half of the specimens was smashed. This failure mode is different from that of concrete cylinders in which the splitting failure was observed [35] (see 281 282 Fig. 11). This is because the tensile properties of the syntactic foam are governed by the epoxy resin which has much higher tensile strength than that of concrete. As a result, the 283 284 syntactic foam failed by crushing of the compressive material rather than splitting as in 285 concrete. The progressive failure of the white specimen is shown in Fig. 12. Spalling of the 286 white syntactic foam at the top started at about 0.8 ms after the impact. A vertical crack initiated at the impacted end propagated downward and became visible at 1 ms. The white 287 288 specimen was severely smashed in a very brittle manner at 2.35 ms. Meanwhile, the black 289 specimen did not exhibit damage up to 1 ms after the impact. At 1.45 ms, the black specimen 290 showed significant damage in the top half. The failure modes of the two specimens showed similar manner, which indicates using 15% volume fraction crumb rubber did not 291 292 considerably reduce the brittleness of the material.

293 The impact force time histories of the two specimens are presented in Figs. 13-14. During the 294 impact event, the projectile may impact the specimens one or multiple times depending on the projectile-specimen interaction. It is noted that the time scale of the impact force measured by 295 a load cell and those from the high speed camera are not synchronized. For the white 296 297 specimen, the projectile first impacted the specimen so that the projectile and the top surface 298 of the specimen moved with two different velocities in the same direction. They then lost 299 contact in a very short period of approximate 35 ms before being in contact again as shown in 300 Fig. 13. The impact force of the white specimen reached the maximum value at the second 301 peak of 372 kN. The impulses of the first and second impacts of the white specimen are 136 302 and 602 kN·ms, respectively. The duration of the first and the second impacts of the white

303 specimen was 1 ms and 5 ms, respectively. The impact force of the black specimen reached 304 the peak of 231 kN with the corresponding duration of 1 ms, resulting in the impulse of 138 305 kN·ms (Table 4). It can be concluded that the white material can withstand higher impact 306 force and impulse than the black material. The impulse of the impact force is defined as a 307 measure of the impact resistance capacity of the tested specimens. The replacement of glass 308 microsphere by crumb rubber decreased the impact impulse capacity of syntactic foam.

309 In this study, the impact energy absorption is estimated based on the energy conservation law 310 in which the impact velocity and the residual velocity of the projectile were traced from the 311 image processing technique. As shown in Fig. 15, the two specimens had similar impact 312 velocities (6.76 m/s) but the black specimen exhibited a lower residual velocity than that of 313 the white specimen (5.96 m/s vs 6.33 m/s). As a result, the black specimen absorbed 1136 J while the energy absorption of the while specimen was 915 J as shown in Fig. 16. The impact 314 315 energy absorption of the material was about 3 times its energy absorption under static loads as 316 shown in Table 4. In this study, replacing glass microspheres by crumb rubber leads to 24% 317 increase of the impact energy absorption while the energy absorption enhancement under 318 static load was only 5%. This increase agrees with the testing results by Li and John [20] in 319 which they found that rubberized syntactic foam with 20% volume fraction had a higher capacity to absorb impact energy and resist bending strength via the positive composite action 320 321 between glass microsphere and crumb rubber. The co-existence of stiff particles (i.e. glass 322 microsphere) and soft particles (i.e. crumb rubber) can adjust and reduce stress concentration. 323 As reported, the initiation energy (i.e. elastic strain energy absorption) increased by replacing 324 a portion of glass microspheres by crumb rubber, which proved the positive effect of adding 325 crumb rubber.

326 In addition, the specific energy absorption of these specimens were observed as  $583 \text{ kJ/m}^3$  for 327 the white foam and 723 kJ/m<sup>3</sup> for the black foam. The specific energy of the syntactic foam in

328 this study was about 10 times smaller than that of the hollow glass microsphere/epoxy based syntactic foam (6-15 MJ/m<sup>3</sup>) reported by Swetha and Kumar [2]. This difference is reasonable 329 330 since the compressive strength of the syntactic foam in the study by Swetha and Kumar [2] was approximately 6 times stronger than that of the syntactic foam in this study. Meanwhile, 331 332 Walter et al. [36] reported the similar energy absorption of epoxy-based syntactic foam ranging from  $200 - 2000 \text{ kJ/m}^3$  when materials with similar strength were used. Therefore, 333 334 the glass microspheres partially replaced by the crumb rubber yielded higher energy 335 absorption but lower impact impulse capacity. Adding crumb rubber to the syntactic foam can 336 increase the energy absorption but reduce the compressive strength of the material. This may be due to the fact that at a high volume fraction of crumb rubber, not much epoxy is available 337 for bonding the matrix and transferring stresses prior to fracture. In addition to examine the 338 dynamic increase factor, the compressive stresses of the specimens are presented in Figs. 17-339 340 18. As shown in these figures, the material is strain rate sensitive as the compressive stress of these specimens was approximately double their static strengths. This increase reasonably 341 342 agrees with the experimental results reported by Zhang et al. [6] in which split Hopkinson 343 pressure bar was used to investigate the dynamic properties of syntactic foam with hollow glass spheres. The authors observed the dynamic increase factor from 1.2 to 2.2 in varied 344 strain rates from  $0.01s^{-1}$  to 2750 s<sup>-1</sup>. It is noted that accurate strain measure could not be 345 346 achieved with the drop-weight tests so that better equipment (e.g. split Hopkinson pressure 347 bar) should be used to further investigate the strain rate effect on the mechanical properties of the syntactic foam. 348

349 5.3. Microstructure investigation

Unlike the specimens under the static tests, macro-spheres coated by fibre became brittleunder impact loads, which was also observed in the previous study [35]. Pham and Hao [35]

352 presented that carbon and glass fibre show very brittle behaviour under impact loads, and 353 glass fibre performs much better than carbon fibre due to its high rupture strain. Fig. 19 shows 354 the failure of the white specimen at the interface and a macro-sphere while this failure was not 355 seen in the rubberized specimen. The macro-sphere was broken in the plane at an angle to the 356 failure surface during the impact event, indicating the brittleness of macro-spheres. This 357 failure indicates the white binder can transfer a sufficiently higher stress to a macro-sphere as compared to the black binder. It is noted that the coated macro-spheres for the rubberized 358 359 specimen did not show damage in the angled plane respect to the failure surface. In general, adding crumb rubber did not show a significant difference of the material properties under 360 static loads but it resulted in a considerable change under impact loads as shown in Figs. 13-361 14, indicating it is sensitive to impact loads. This phenomenon also indicates that the white 362 specimen is able to absorb more energy than the rubberized specimen due to more damage 363 364 occurred in macro-spheres as evident in the impact tests.

In addition, the quality of the bonding between crumb rubbers and the epoxy is very important to the material performance. Kaynak et al. [37] observed poor bonding and separation between the crumb rubbers from waste tyres while Maharsia et al. [18] found a good bonding between the crumb rubbers and the epoxy. A sound bonding between the crumb rubber and epoxy was found in this study.

#### 370 6. Conclusions

371 Replacing 15% volume fraction of the glass microsphere by the crumb rubber slightly 372 increases energy absorption capacity of the syntactic foam under quasi-static loads but not 373 impact loads. The density of the 3-phase syntactic foam is about 25% of that of normal 374 strength concrete but it has a similar fracture toughness.

For the 2-phase syntactic foam, introducing 15% volume fraction of the crumb rubber slightly increases the fracture toughness (8%) and the fracture energy (22%). More trial fracture toughness tests should be conducted to determine the optimal value of the volume fraction of the crumb rubber between 10% and 15%.

For the 3-phase syntactic foam under quasi-static loads, the compressive strength and elastic modulus of the rubberized syntactic foam reduces by 6% and 16.1%, respectively. The energy absorption capacity of the black material increases by 5.1% as compared to the white material. The volume fraction of the crumb rubber is recommended to be reduced for a better performance on the energy absorption of the 3-phase syntactic foam.

The impact impulse resistance of the 3-phase rubberized syntactic foam is inferior as compared to the 3-phase syntactic foam without crumb rubber. However, the impact energy absorption of the 3-phase rubberized syntactic foam increased by 24% as compared to that of the syntactic foam without crumb rubber.

In summary, the optimal volume fraction of crumb rubber may fall between 10% and 15%. When 3-phase syntactic foam is introduced, it should have lower volume fraction of crumb rubber as compared to that in 2-phase syntactic foam. The optimal values depend on 2-phase or 3-phase syntactic foam and static or dynamic loading conditions.

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Chillip Martin

Motorial		Vo	lume fraction of	Macro spheres	
	Material	Epoxy blend	Glass microspheres	Crumb rubber (75 µm)	(packing density)
) mhasa	White syntactic foam	64	36	nil	nil
∠-pnase	Black syntactic foam	64	21	15	nil
2 mb aga	White syntactic foam	64	36	nil	60%
3-pnase	Black syntactic foam	64	21	15	60%
				S	

## 1 Table 1. Composition of component materials

2

No.	Ligament	Width	Crack	Peak	$K_{\rm Ic}$ <sup>a</sup>	Energy	Corrected	$G_{Ic}$ b
	width	(mm)	length	force	(MPa	to peak	energy	$(kJ/m^2)$
	(mm)		(mm)	(N)	$\sqrt{m}$ )	(Nm)	(Nm)	
1	20.1	40.2	20.10	304	0.809	0.073	0.054	0.272
2	20.2	40.0	19.80	331	0.873	0.086	0.067	0.337
3	20.2	39.9	19.70	311	0.81	0.071	0.052	0.260
4	20.2	40.1	19.90	338	0.876	0.093	0.074	0.363
5	20.1	40.0	19.90	317	0.839	0.084	0.065	0.325
6	20.1	40.2	20.10	336	0.894	0.098	0.079	0.395
7	20.1	40.2	20.10	349	0.928	0.098	0.079	0.397
8	20.1	40.1	20.00	331	0.869	0.081	0.062	0.307
9	20.2	40.1	19.90	349	0.909	0.096	0.077	0.382
10	20.1	40.0	19.90	313	0.828	0.075	0.056	0.283
Mean		40.1	19.9	328	0.864	0.086	0.067	0.332
SD		0.1	0.14	15.9	0.041	0.010	0.010	0.051

## 1 Table 2. Experimental results of fracture toughness tests for the white material

2 <sup>a</sup> Critical stress intensity factor

3 <sup>b</sup>Critical strain energy release rate

No.	Ligament	Width	Crack	Peak	$K_{\rm Ic}$ <sup>a</sup>	Energy	Corrected	$G_{Ic}$ b
	width	(mm)	length	force	(MPa	to peak	energy	$(kJ/m^2)$
	(mm)		(mm)	(N)	$\sqrt{m}$ )	(Nm)	(Nm)	
1	20.2	40.2	20.0	342	0.889	0.117	0.083	0.408
2	20.1	40.2	20.1	379	1.013	0.126	0.092	0.464
3	20.0	40.2	20.2	354	0.935	0.107	0.073	0.362
4	20.3	40.2	19.9	370	0.954	0.124	0.090	0.443
5	20.1	40.2	20.1	347	0.919	0.109	0.075	0.377
6	20.2	40.2	20.0	327	0.850	0.097	0.063	0.312
7	20.1	40.1	20.0	369	0.974	0.124	0.090	0.447
8	20.1	40.1	20.0	345	0.920	0.113	0.079	0.397
$9^*$	20.2	40.3	20.1	362	0.944	0.122	0.088	0.432
Mean		40.2	20.0	355	0.933	0.115	0.081	0.404
SD		0.1	0.1	16.4	0.047	0.010	0.010	0.048

1 Table 3. Experimental results of fracture toughness tests for the black material

- 2 \* Data of specimen no. 10 was lost
- 3 <sup>a</sup> Critical stress intensity factor
- 4 <sup>b</sup>Critical strain energy release rate

Testing condition	Characteristic	White foam	Black foam
	Compressive strength (MPa)	16.5	15.5
Static load	Elastic modulus (MPa)	748	627
	Energy absorption (kJ)	0.37	0.39
	Peak impact force (kN)	295/372 <sup>*</sup>	231
	Impact duration (ms)	$1/5^{*}$	1
Impact load	Impact impulse (kN.ms)	136/602*	138
	Energy absorption (kJ)	0.92	1.14
	Specific energy (kJ/m <sup>3</sup> )	583	723

## 1 Table 4. Experimental results of 3-phase syntactic foam

2 \* Results corresponding to the first and second peaks



Glass spheres (D50 µm)



Carbon fiber reinforced macro-spheres (D10 mm)







Crushing failure of syntactic foam

Splitting failure of concrete























Syntactic foam without crumb rubber Syntactic foam with crumb rubber







White syntactic foam



Black syntactic foam





## HIGHLIGHTS

- Fracture toughness of multiphase syntactic foam
- Impact behavior of multiphase syntactic foam
- Effect of rubber content on mechanical properties
- Impact testing of syntactic foam
- Dynamic properties
- Coating with epoxy resin