Mechanical behavior and failure mechanisms in polymeric syntactic foams

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NANYANG
TECHNOLOGICAL
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**MECHANICAL BEHAVIOUR AND FAILURE
MECHANISMS IN POLYMERIC SYNTACTIC FOAMS**

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SCHOOL OF MECHANICAL AND AEROSPACE ENGINEERING

NANYANG TECHNOLOGICAL UNIVERSITY

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Abstract

Syntactic foams have been increasingly used in load bearing structures in the low to high speed applications due to their excellent specific properties. The aim of this research is thus to fully understand the overall mechanical properties and the associated failure mechanisms under different strain rates.

The quasi-static (QS) compression tests and the finite element (FE) modeling of a representative elementary volume syntactic foam were first performed to investigate the elastic behavior and failure mechanism of the glass microballoon epoxy syntactic foams as well as the effect of glass microballoon volume fractions (V) and the radius ratio η . The elastic characteristics of the foam vary with both the V and η . The localized stresses concentrate in various zones within the foam. Dependent on the V , micro-cracks can propagate either in the preferred longitudinal or diagonal directions in the foam. To directly observe the internal microstructural change of the constituents during the failure process, the x-ray micro computed tomography (μ XT) with interrupted uniaxial compression was conducted on cenosphere epoxy syntactic foams. Moreover, to further investigate the failure micromechanisms of the foam, FE modelling of the full scale foam specimen was developed and experimentally validated to predict the localized stress, fracture of cenospheres and deformation in the matrix. The FE predictions were related to the μ XT observations to analyze the underlying mechanisms of internal 3D failure process in the plateau region of the foam. It was found that the internal compressive failure in microscopic scale consists of (1) the crushing of

hollow spheres and (2) the plastic deformation and fracture of the matrix. The failure mechanisms in the two constituents are determined by the localized stress state and the stress transfer between the constituents, and govern the different strain stages of bulk stress–strain behavior of the foam. The failure mode of the individual hollow sphere was vertical splitting. Finally, to investigate the strain rate dependent failure mechanisms of syntactic foam, the QS compressive tests and Split-Hopkinson pressure bar tests were controlled to stop the deformation of the foams at various strains. It was found that the failure mechanism is significantly affected by strain rates. At dynamic rates, macro-cracks form earlier in the matrix and can split hollow spheres. An empirical constitutive equation was used to quantitatively relate the damage to the strain and strain rate.

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Chapter 1

Introduction

1.1 Background

In many industries, there is an ongoing search for lightweight materials. One of the major driving factors for this development is the ever increasing oil price. Reducing the weight of transportation vehicles will result in increasing fuel efficiency and contribute to a better environment through the reduction of exhaust emissions.

Improving specific properties is vital for reducing the weight of structural applications. It can be achieved through an increase in the material properties or through a decrease in the density. As most traditional materials have been around for decades, it is not expected that research on improving material properties will lead to a dramatic reduction of the specific properties. Major improvement is certainly expected from the latter one. Thus more and more efforts have been made to investigate the low density materials, such as composites and foam materials. Among low density materials, syntactic foams are attractive because of the novel combinations of mechanical and physical properties, for example the capacity of undergoing large strains at almost constant stress allowing the significant energy

absorption without generating damaging peak stresses, the good insulation and ablation abilities with the lightweight and chemical stability, the low cost of the constituent materials and easy fabrication process etc. Therefore, syntactic foams have been used successfully in a large range of industries, the applications can be found in marine industry, oil extracting equipments, civil engineering, transportation vehicles, electrical and electronic industry, medical uses and sports requisites etc.

It is believed that with the development of design concept, manufactory technology and deeper investigations, the role of syntactic foam in various industries will be more and more important.

1.2 Problem statement and objectives

Numerous research activities have been performed in the past decades to investigate the bulk mechanical properties of syntactic foams such as compression, tension, impact resistance, flexural response and damping etc. The bulk behavior of porous materials is significantly determined by the deformation and failure process in the constituents in microscopic scale. However, very few studies focused on the microscopic failure mechanisms in syntactic foams. In the few literatures, the fracture surface of the foam after mechanical testing was characterized to explore the microstructural change in the final stage and analyze the possible failure process. However, the post-test examination overlooks the effects of elastic recovery, shrinkage and crack closure in polymer, and cannot reveal the real-time process of failure. Moreover, internal failure features under the fracture surface of

the foam are not characterized and thus unknown. Therefore, the microstructural evolution in the syntactic foam needs to be tracked to reveal the internal 3D deformation and failure process in microscopic scale.

It is a challenge to characterize the internal 3D morphology and the damage evolution in syntactic foams due to the increased complexity of multiple constituents. Recent developments in X-ray microtomography (μ XT) have allowed for the observation of internal microstructural features in composite and porous materials with a high resolution. The μ XT coupled with in situ mechanical tests has been proved a powerful tool that can provide a new insight to understand the internal morphology and the morphological change of materials subjected to loads. However, there is a lack of the data about the in situ mechanical tests with μ XT inspections conducting on syntactic foams.

To design the structural components using syntactic foams, reliable constitutive descriptions of the mechanical properties of the material are required. Some analytical approaches were proposed to calculate the bulk foam properties based on the known constitutive parameters of the microballoons and matrix. But they cannot be used to investigate the behaviors in micro scale. Some researchers developed the representative elementary volume (REV) finite element models to represent a small part of a full syntactic foam specimen, and the localized stress field can be analyzed. However, most REV models were created with the unrealistic assumptions, and were only used to simulate the elastic behaviors of syntactic foams. Moreover, the REV models cannot well represent the mechanical

behavior of a full syntactic foam specimen in which different stress states and deformation phenomena may occur in various parts of the specimen. Thus, a full-scale model of the syntactic foam is required to more accurately represent the foam and better simulate the behavior. In addition, the plastic deformation of the matrix and failure of the embedded hollow spheres need to be included in the FE model to simulate the phenomena in the plateau region of the foam.

The increasing usage of syntactic foams in transportation applications motivates the comprehensive studies on evaluating the mechanical response of syntactic foams under different strain rates. Though it has been found the mechanical properties of syntactic foam show the rate dependency, very little research has focused on the damage evolution and microscopic failure mechanism of syntactic foams subjected to dynamic loads.

The aim of the research is to solve the problems as highlighted above, including:

a) Get a better understanding of the relationship between the microstructure and mechanical behaviors of syntactic foam under quasi-static and dynamic strain rates.

b) Establish the reliable 3D finite element model that is close to the real material to simulate the mechanical behaviors and internal deformation of syntactic foam.

c) Find out the 3D damage evolution and associated mechanisms of

syntactic foam under different strain rates in various length scales.

1.3 Scope and layout of the study

In order to achieve the objectives, the work is divided into two parts: (1) experimental investigations on the mechanical behaviors and the internal deformation process of syntactic foam and (2) finite element modeling of the mechanical behavior of syntactic foams. The work is outlined in Figure 1. 1.

Figure 1. 1 Project scope.

The details of the three studies are:

a) The elastic behavior and failure mechanism in the glass microballoon epoxy syntactic foams as well as the effect of glass microballoon volume fractions and the radius ratio were investigated. A 3D cubic REV in the foam was generated, in which the microballoons with a statistic size distribution were randomly distributed. The FE model of the REV foam was then developed to predict the localised stress field in the elastic region during the compression of the foam.

b) The μ XT combined with interrupted compression was performed to directly observe the internal morphological change of the cenosphere and matrix during the failure process of the cenosphere epoxy syntactic foam. A 3D FE model of the full scale foam specimen was developed to predict the localized stress field and the fracture of cenospheres and the 3D deformation during the compression of the foam.

c) The compressive behavior and failure mechanism of cenosphere epoxy syntactic foams at different strain rates were investigated, and the evolution of damage was quantified by using an empirical constitutive equation

The layout of the study is including:

A brief introduction is presented in the first chapter. Both the problems and the research objectives are listed in Chapter 1.

Chapter 2 gives a detailed literature review in syntactic foams materials and

their material selection, applications, mechanical behaviors under different strain rates, the failure modes and constitutive descriptions. Then the μ XT applied to characterize the microstructure of foam materials, build the FE models and couple with in situ mechanical experiments are also discussed.

Chapter 3 introduces the elastic behavior and failure mechanism in glass microballoon epoxy syntactic foams as well as the effect of glass microballoon volume fractions and the radius ratio. A reasonable REV FE model was developed to analyze the internal stress field and predict the mechanical behavior the syntactic foam.

Chapter 4 presents the 3D internal deformation and damage evolution of cenosphere epoxy syntactic foams. A full scale FE model with the consideration of cenosphere crushing and the matrix plastic deformation was developed to simulate the compressive behavior of the syntactic foam in its elastic region and plateau region. Based on the experimental and FE investigations, the failure mechanism of the material is discussed from micro scale to macro scale.

Chapter 5 illustrates the compressive behavior and damage evolution of cenosphere epoxy syntactic foams subjected to quasi static and dynamic loading rate. An empirical constitutive equation was used to quantify the relation between the damage, the strain and the strain rate.

The main conclusions and the future work are summarized in Chapter 6.

Chapter 2

Literature review

2.1 Syntactic foams

2.1.1 Introduction of syntactic foams

Syntactic foams (the word “syntactic” comes from the Greek word “syntaktikos” meaning to “put together” [1]; the word “foams” is used due to the nature of the cellular structure of the materials) are a special kind of particulate composite material. They consist of a matrix, called the binder, and a filler of hollow spherical particles, called microspheres or microballoons, distributed within the binder. A representative schematic of syntactic foam is shown in Figure 2. 1.

Figure 2. 1 A representative schematic of syntactic foam.

Since the hollow spheres have continuous and close shells, the syntactic foams can be considered as a closed-cell foam. However, they differ from the conventional close-cell foams. Syntactic foams are not binary-phase but tertiary-phase systems because the filler and binder are usually made from different materials, and the gas-containing particles is thought of the reinforced component in syntactic foams [2]. Compared with the ordinary close-cell foams, two types of porosity exist within syntactic foams. The majority of cellular structure of syntactic foams is controlled by the size, quantity and distributive uniformity of the hollow fillers. The air voids entrapped within the matrix also contribute to the porosity of syntactic foams. Such air entrapments are introduced by the stirring during the fabrication process, and will reduce the young's modulus, strength and increase moisture absorption of syntactic foams [3]. Due to their negative impact on the properties, the presence of air voids must be limited.

Figure 2. 2 shows a scanning electron microscope (SEM) image of the fracture surface of glass microballoon epoxy syntactic foam, in which the three phases can be clearly identified. The microspheres are seen as round particles embedded in the matrix. The smaller irregular shape holes are considered as the air voids.

Figure 2. 2 A SEM image of the fracture surface of syntactic foam.

2.1.2 Material selection of syntactic foams

In the viewpoint of the manufacturing technology, syntactic foam is not fabricated chemically, but is mechanically filled the matrix with the hollow particles. Therefore the materials selection of the binder and the filler forming syntactic foams has a very large range.

2.1.2.1 Fillers

The fillers used in syntactic foams should meet the requirements, including spherical contour, non-cohesive, strong, intact, moisture and chemically resistant and hydrolytically stable [4, 5]. Various types of hollow particles can be found in previous studies, such as glass [6, 7], polymeric [8], carbon [9, 10], ceramic [11, 12] or metal [13, 14] microspheres. Depending on the types of the microspheres, the

hollow spheres have a diameter range between 1 μm and 600 μm , a thickness of 1 μm to 10 μm , and bulk density of 70 kg/m^3 to 750 kg/m^3 .

Glass microspheres are most widely used in syntactic foams because of the high strength of the glass, smoothness, good wettability and low manufactory cost [15]. Such microspheres are manufactured in vertical tube furnaces heated by gas. A uniformly dispersed powder containing glass and a chemical blowing agent is sprayed into the bottom of the tower. The blowing agent evolves gas at the melting point of the glass and inflates the partially fused monolithic particles. The microspheres thus formed are carried by the hot gas to the top of the tower where they are cooled. Generally, sodium and borosilicate glasses are used to make glass microspheres [16].

Polymeric microspheres, made of epoxy resin [17], phenolic [18], polystyrene [19] and so on are also common fillers in syntactic foams. They are manufactured by thermally treating sprayed solutions or emulsions. A solution of any film-forming polymer can be used. The solvent has to be evaporated first, the sprayed monolithic particles are then heated to expand the gas or vapor within the droplets, and finally microspheres will be hardened [2]. Compared with glass microspheres, syntactic foams with polymeric microspheres have lower density and better adhesion with polymeric binders [20], even the coupling agent is not used. However, it is known that polymer is usually weaker and softer than glass, and polymeric microspheres cannot withstand a hydrostatic pressure higher than 2.5 MPa, whereas glass microspheres can stand pressures up to 12 MPa [2]. Therefore,

syntactic foams with polymeric microspheres show worse mechanical properties than glass microspheres filled foams.

Carbon microspheres are a new type of hollow filler applied in syntactic foams. They can be derived from carbonizing phenolic microspheres at 900 °C in an inert atmosphere [21]. The advantages of carbon microspheres are very low density (130 kg/m³ to 150 kg/m³), bind well with the polymeric matrix and extremely good strength. Carbon microspheres, even with defects in their shells, are much stronger than glass microspheres. At a nitrogen pressure of 7 MPa, 43 percentage of glass spheres, but only 5 percentage of carbon microspheres are damaged [2]. Consequently, very good mechanical properties can be gained by the carbon microspheres filled syntactic foams. Regardless the cost, it is considered the carbon microspheres are the ideal fillers for syntactic foams.

Another kind of high performance fillers is made of ceramics, such as Al₂O₃ [22] and SiC [12]. A relative low performance type of ceramic microspheres can be collected from the fly ash. The coal combustion produces thousands tons of fly ash every year. Fly ash particles can be classified into two categories [23]: precipitator and cenosphere. A cenosphere particle has hollow structure with a density of about 600 kg/m³, and the chemical composition of a cenosphere is silica (50% to 60%), alumina (22% to 30%) and iron (1.5% to 5.0%). The very low cost and good mechanical properties make it a good choice for producing syntactic foams. But the imperfect spherical shape and the porosity in the shell limit the performance of cenosphere, thus affect the properties of syntactic foams with

cenospheres. Luong et al. [24] used a technique based on deposition to produce ceramic microspheres. A sacrificial polymer precursor is first applied to deposit the Al_2O_3 nano pellets over its surface, and then is followed by sintering in an inert atmosphere at 1650 °C, during the process the precursor melts away and hollow Al_2O_3 spheres are formed. Although the geometric profile and overall properties of the Al_2O_3 spheres produced by sintering are very good, the manufactory process is very complicated and inefficiency. Therefore they are not suitable for large scale components in industrial applications.

Recently metallic hollow spheres have been found in syntactic foam system [13, 25]. They are produced through a powder metallurgical technique. In this process, an organic sphere template is coated with metal powder slurry. The organic sphere and the binding agent are then removed, and the spheres can either be sintered separately or formed into net-shape components [26]. Besides the high young's modulus and strength, the excellent plasticity is the unique characteristic for metallic materials. Hence the tolerance and toughness of metallic hollow spheres filled syntactic foam are improved very much. The drawbacks of the metallic spheres are similar to the sintering Al_2O_3 spheres.

Figure 2. 3 shows the surface morphology of the different types of the microspheres. The intact, spherical and smooth surface can be observed for the glass, phenolic and metallic microspheres. In contrast, some irregular shape and rough surface microspheres can be found in Figure 2. 3 (c) and Figure 2. 3 (d), indicating the quality of carbon microspheres and cenospheres are not as good as

the other three types of microspheres in the viewpoint of appearance.

Figure 2. 3 SEM or optical microscopic images of the different types of microspheres: (a) glass, (b) phenolic [27], (c) carbon [27], (d) ceramic (cenosphere) and (e) metal [26].

2.1.2.2 Binder

As binders, the material used to make syntactic foams must satisfy the following specifications: a) low viscosities, b) easily controlled gelation times, c) small exothermal effects during curing, d) small curing shrinkages, e) good adhesions and wettability to the filler, and g) compatible with modifiers and fillers. A wide variety of polymer and metal matrix syntactic foams has been reported in the published studies, see for example [28-30].

Among polymeric matrix syntactic foams, epoxy resin based foams are widely investigated due to their excellent mechanical properties. Other advantages gained by epoxy resins as the binder in syntactic foams are including the low levels of volatiles, good adhesion, low shrinkage, good chemical and heat resistance, and ease of processing [31]. However, the disadvantages of most epoxies are obvious as well. They show high viscosity at room temperature and are brittle after curing. The high cost is another problem for applying epoxy based syntactic foams in industry. The first two problems can be effectively circumvented by using diluents and adding modifier [2]. In addition the implantation of modifiers also increases the thermal resistance, and enhance the stability to wet or corrosive media. To decrease the cost, researchers investigated some low price polymeric matrix.

Gupta et al. [29] reported the synthesis process and mechanical properties of vinyl ester matrix syntactic foams. It is found the low cost vinyl ester resins can result in syntactic foams with lower but comparable performance to epoxy matrix systems. Winkel et al. [32] used polyester to create a syntactic foam. But the

polyester have limited the applications of the final foam material because it shrink strongly during hardening and this results in decarbonation and cracking [2].

Except for the mentioned polymeric matrix, some other types of polymer based syntactic foams have also been reported. Kenig et al. [33] investigated the fabrication parameters, mechanical properties and fracture behavior of silicone based syntactic foams. Zhang and Ma [34] applied the phenolic matrix to fabricate syntactic foams, and investigated the effect of coupling agent on their mechanical properties. Adrien et al. [35] reported the work on polypropylene and polyurethane based syntactic foams. It was found the stiffness and strength of thermoplastic and elastomer based syntactic foam is not as good as that in epoxy matrix foams due to the nature of soft matrix. Gladysz et al. [36] developed a bismaleamid matrix syntactic foam which can be used at the high temperature applications. However, it should be noted that the majority studies and applications of syntactic foam involve the use of epoxies.

Because of the relatively low strength and stiffness, low temperature capability, high flammability and susceptibility to degradation in some environments of polymers, polymeric matrix syntactic foams may not be suitable for working under the tough environment [37]. Recently, metal matrix syntactic foams have attracted more and more attentions due to their good combinations mechanical properties. In the consideration of lightweight, pure Al, Al alloy, Mg alloy and Ti alloy have been found as the binder in syntactic foams until now [38-43]. Metal matrix syntactic foams can be fabricated by powder metallurgy [44,

45] and infiltration techniques [46, 47]. Due to the complicated fabrication process and high cost, the experimental investigations of the metal binder syntactic foams is still some way off their industrial applications.

2.1.3 Applications of syntactic foams

Although the use of syntactic foam has only increased sharply in the twenty-first century, it has been around for nearly half a century. Syntactic foams originate from the early 1960s [2]. Syntactic foams are a good fit for applications seeking weight reduction with little effect on performance or durability. Applications for syntactic foams cover a large number of industry fields. General industrial uses of syntactic foams include use as a tooling material, structural components and offering the workability of wood with the dimensional stability of metal. The material permits overall vehicle weight reduction, and is suitable for use where a high strength to density ratio is required.

The oldest and widest applications of syntactic foams are for marine industry due to their minimum liquid absorbance together with the low density and impressive compressive properties. Syntactic foam products are used in the marine and oceanographic industry for use as floats, buoys and as a void filler in submarines, diving suits, insulation of underwater pipes, underwater rescue apparatus and equipment for boats or deep-water submarines, and capable of withstanding depths below 7,000 feet [48-50]. A typical example is shown in Figure 2. 4. It is a remotely operated vehicles purposely designed for scientific research. A bright yellow flotation package made of syntactic foam provides the

buoyancy for the rest of the vehicle when it sinks in the water, and reduces the average density of the whole equipment to just a little less than the density of seawater. This makes the vehicles just slightly buoyant, so it will float on the surface, but a small force generated by the vertical thrusters can drive the vehicles up or down.

Figure 2. 4 A remotely operated vehicles mounted with syntactic foam flotation package [51].

Another important application of syntactic foams is in aerospace industry for their insulation and ablation abilities. It has been well known that sandwich structure with honeycomb composites panels are used for manufacturing aircrafts. As a replacement for existing honeycomb structures, syntactic foams are more suitable in the parts with the complex structures. XFLAMTM developed a syntactic foam core sandwich panel that leads the way in high performance thermal resistant panel systems with lightweight as shown in Figure 2. 5. The syntactic foam core has excellent mechanical strength, superior insulation, and low toxicity and is completely recyclable. In addition, more specific properties can be gained by

syntactic foams, such as good acoustical attenuation, outstanding vibration insulation, low radar cross-section and so on [52]. The combination of these specific features makes syntactic foam a novel material that has real potential applications in military.

Figure 2. 5 A sandwich panel with syntactic foam core [53].

Finally, syntactic foams have also been used successfully in a large range of other areas, the applications can be found in oil extracting industry [54], civil engineering [55], automotive applications [56], electrical and electronic industry [5], medical uses [57] and sports requisite [58] etc.

2.2 Mechanical behaviors of syntactic foams

The advantageous properties such as, good mechanical performance, low moisture absorption, good chemical stability, high specific strength, excellent damage tolerance and outstanding insulation and ablation abilities and so on, have been introduced in the previous section. As this study focuses on the mechanical behaviors of syntactic foams, the mechanical properties of syntactic foams will be further discussed in this section.

2.2.1 Mechanical properties of syntactic foams under quasi-static loading rate

The bulk mechanical properties of syntactic foams under quasi-static (QS) have been extensively investigated during the last decades [2, 59]. The following paragraphs will highlight the compressive, tensile and flexural properties of bulk syntactic foams.

2.2.1.1 Compressive properties

Syntactic foams are mainly used under compressive loading because of their excellent compressive resistance and tolerance [60]. Hence a large number of studies reported the compressive properties of different types of syntactic foams [3, 12, 50, 61-66]. Most syntactic foams have one thing in common: they can be divided into three sections as show in Figure 2. 6.

Figure 2. 6 A typical compression stress-strain curve of syntactic foam [50].

In the initial section, the foam exhibits elastic behavior. Providing the yield point is not reached, the foam will recover back to the original shape after the load is releasing. The yield point, corresponding with the compression yield strength, occurs at the transition between the elastic section and the collapse plateau, which is the second section of the curve. The compressive load remains fairly constant during the plateau section. In this region of the compression test the foam can withstand most of the compressive yield strength. In Bunn and Mottram's [50] report, the phenolic microsphere filled epoxy syntactic foam can withstand 95% of its compressive yield strength. During the process, the foam undergoes densification as a result of a large number of microspheres get compacted and crushed. The third, and last, section of the curve occurs after full densification has been achieved and the compression sample can resist higher loads. The three regions can be summarized as four parameters; they are Young's modulus (E),

compressive strength (σ_y), the strain at peak stress (ε_{cr}) and the plateau stress (σ_{pl}).

These parameters are very useful in understanding the overall mechanical behavior of the foam materials, not only for syntactic foams, but also for the metal foams.

2.2.1.2 Tensile properties

The tensile properties of syntactic foams were also investigated by many researchers [8, 29, 67]. Gupta et al. [29] reported the effect of the type and volume fraction of microballoon on the tensile characteristics of vinyl ester syntactic foam, as shown in Figure 2. 7.

Figure 2. 7 Comparison of tensile stress–strain curves of vinyl ester matrix syntactic foams with different types of microballoons and their content [29].

From Figure 2. 7, it is found that all curves show linear stress–strain relationship immediately followed by perfect brittle rupture. The tensile curves are noticeably different from the stress–strain curves subjected to compressive load, in which an elongated plateau region related to energy absorption capability can be observed. The tensile strength of syntactic foams is not reported here because it is generally highly affected by matrix porosity and microstructural inhomogeneities as earlier discussed for epoxy matrix systems, whilst the tensile modulus shows the dependency with the volume fraction of microballoons. In this study, the tensile modulus decreased with the increase in fillers content. This is because the microballoon used in this work were considered as the weaken phase which replace the strength epoxy. But if the hard microballoon were embedded in syntactic foams, the inverse results will be obtained. In Chapter 3, the phenomenon will be discussed in detail.

2.2.1.3 Flexural properties

Compared to the compressive and tensile properties, the studies on the flexural properties of syntactic foams are scarce. Usually the three-point bending test is used to test the flexural properties. Figure 2. 8 shows a typical flexure stress-strain curve of syntactic foam. Similar with the tensile behavior, a linear elastic loading curve followed by the fracture can be observed. The brittle failure was considered as the rupture mode of syntactic foam subject to bending [36]. In addition, it has been reported the failure strain and flexural strength decrease with the increase of the volume fraction of the microballoons [8, 68].

Figure 2. 8 A typical flexure stress-strain curve of the syntactic foam in a three-point bend configuration [36].

2.2.2 The effect of strain rate on mechanical properties

Recently, more research efforts have been made to explore the high strain rate response of the syntactic foams, and these studies reported significant strain rate sensitivity [69-72]. The rate dependency of syntactic foams is most likely caused by the rate sensitivity of the matrix material. Viscoelasticity of the polymer matrix stands the chance to result in the strain rate dependency of the matrix and consequently of the syntactic foam [69].

Li et al. [69] investigated the strain rate dependent compressive properties of glass microballoon epoxy syntactic foams. They found the tendency of the dynamic stress-strain curve is similar to that at quasi-static rates as shown in Figure 2. 9. But the peak stress and plateau stress increases with strain rate. They developed a reasonable empirical constitutive description to represent the relation between peak/plateau stresses and strain rate, the measured and predicted results

were shown in Figure 2. 10.

Figure 2. 9 The stress–strain curves of glass microballoon epoxy syntactic foams at different loading rates [69].

Figure 2. 10 The measured and predicted peak and plateau stresses as a function of strain rate [69].

Except for the peak and plateau stresses, the Young's modulus and failure stress of syntactic foams also show the strong strain rate dependency. Figure 2. 11 and Figure 2. 12 illustrate the modulus and failure stress increase with strain rates.

Figure 2. 11 Modulus versus microballoon volume fraction for glass microballoon epoxy syntactic foams at varying strain rates [73].

Figure 2. 12 Strain-rate effects on the failure stress for epoxy syntactic foam [74].

More recently Pellegrino et al. [75] reported the glass microballoon polyurethane syntactic foam subjected to tensile loading under high strain rate. In their work, the syntactic foam possessed higher strain rate sensitivity in tension compared to compression.

2.2.3 Failure mode of syntactic foams

Another research focus on syntactic foams is the failure mechanisms, which is necessary to determine actual limits and assess lifespan for the materials. In the viewpoint of reality applications, the failure of syntactic foams subjected to compression loading will be introduced.

Based on the experimental investigations, three failure modes under the quasi-static condition have been found in literatures. They are: 1) shear fracture in the diagonal of the specimen (nearly 45° angle to the loading axis), 2) longitudinal splitting failure along the compression direction (vertical cracks along the loading axis), and 3) layered crushing due to the failure of some weak transverse planes (perpendicular to the loading axis) [61, 64, 69, 76, 77]. Figure 2. 13 demonstrate the three failure modes of syntactic foams.

There are much fewer studies on the failure mechanisms of syntactic foams under dynamic strain rate. Shunmugasamy et al. [72] reported the failure initiated at one side of the specimen in the impact test. Then a crushed layer will be formed, from which the cracks originated and cut through the whole sample, rather than choosing a preferential path, the cracks propagate indiscriminately. Therefore, both

the shear and vertical cracks can be observed during the dynamic compression, as shown in Figure 2. 14.

Figure 2. 13 The failure modes of syntactic foams subjected to compressive loading under quasi-static strain rate: (a) shear cracking [55], (b) vertical cracking [68] and (c) layered crushing [64].

Figure 2. 14 A μ XT slice showing the cracks of the syntactic foam specimen after the impact test [72].

2.3 Constitutive descriptions of syntactic foams

The absence of the reliable constitutive descriptions for calculating and predicting the properties of the syntactic foams will restrict considerably their

applications. Hence for a proper design of components using syntactic foams, it is very useful to be able to have the accurate constitutive descriptions that can convey the constitutive parameters of the hollow spheres and the matrix to the bulk foam properties. The following paragraph will be focusing on the constitutive descriptions of syntactic foams.

2.3.1 Calculation of modulus and strength of syntactic foams

An approach by adopting a microstructural model of syntactic foam morphology to calculate the micromechanical behaviors of an epoxy foam with phenolic microspheres was reported by Shutov [2]. It assumed that the mechanical properties of the microsphere walls and the binder are the same, and the volume fraction of filler is substantially smaller than that of the matrix. The microstructural parameters of the syntactic foam are then defined in terms of the dimensions of the microspheres, and their displacements have the same nature as the deformations of the nodes and edges of an imaginary lattice, so that

$$E = E_0 \frac{\beta^2}{1 + \beta^2}$$

$$\sigma_c = \sigma_0 \frac{\beta^2}{1 + \beta^2}$$

where E and E_0 are the Young's modulus of the syntactic foam and the polymer matrix, respectively, σ_c is the nominal ultimate compression strength of the foam, σ_0 is the compression strength of the polymer matrix, and β is a parameter of the

cellular structure defined by the equation

$$\frac{\gamma}{\gamma_0} = \frac{3\beta^2 + \beta^3}{(1 + \beta)^3}$$

where γ is the apparent density of the foam, and γ_0 is the density of the polymer matrix.

2.3.2 Finite element modeling of syntactic foams

Though there are some calculations that can obtain the modulus and strength of syntactic foams from the properties of the constituent materials, such descriptions cannot reveal the microscopic behaviors and internal stress distributions. To solve the problem, finite element modelling has been employed to investigate the syntactic foams, and have been proved an effective tool to investigate the stress field and to characterize the constitutive and failure behavior of syntactic foams [35, 78].

Nguyena and Gupta [79] developed a unit cell model, containing one eighth of a hollow sphere embedded in the matrix, to estimate the elastic behavior of the syntactic foam as shown in Figure 2. 15. By using the model, the effective Young's modulus of fiber-reinforced syntactic foams was estimated, and the effect of wall thickness on the modulus of the bulk composites was studied. Moreover, the von-Mises stress distribution in matrix, microballoon and fiber was also analyzed. The model explained some experimental results, but may not represent the overall behavior because the syntactic foam is heterogeneous in microscopic scale.

Figure 2. 15 A unit cell model contains one eighth of a hollow sphere embedded in the matrix [79].

Hence, a FE model of syntactic foams should consist of a sufficient amount of hollow spheres to better characterise the foam. Bardella et al. [80] developed a 3D FE model consisting of a number of microballoons as shown in Figure 2. 16. In their study, it was demonstrated that such 3D FE model was more effective in predicting the mechanical properties of syntactic foams compared to the one eighth unit cell model. Similar investigations were also performed by other researchers [81, 82].

Figure 2. 16 A 3D FE model of syntactic foam consisting of a number of microballoons [80].

However, there are still some unrealistic assumptions in models created by Bardella et al. [80]. They assumed the simple uniform distribution in the size (equal outer diameter) or location (array arrangement) of microballoons or the both. In fact a 3D model close to the real syntactic foam needs to include the random location distribution of microballoons and the statistic distribution of their sizes. Taking the sizes and locations of microspheres into account, Yu et al. [78] developed a 3D syntactic foam model that is close to the real case (see Figure 2. 17). Moreover, the cohesive layer was applied to represent the bonding between the microspheres and the matrix. Therefore the tensile strength properties and the associated failure process of syntactic foams were well predicted.

Figure 2. 17 A 3D FE model that is close to the real syntactic foam (The sizes and locations of microspheres were taken into account) [78].

2.4 μ XT applied to investigate the foam materials

μ XT has appeared to be a very powerful tool allowing characterizing the microstructure and also the deformation modes of cellular materials. The tomography technique allows for instance to capture architectural differences between materials exhibiting identical relative densities. The knowledge of the actual structure and arrangement of the solid phase in the 3D has straightforwardly suggested using this new kind of information as an input for finite element models which describe better the peculiarities of the behavior of these materials. Recently, foam materials has be tested in situ coupling with the μ XT imaging, a better understanding of the internal morphology, as well as the morphological changes as a result of the test was derived. The next section will mainly discuss the uses of μ XT in the investigations of the foam materials.

2.4.1 Structural characterization of foam materials

According to specific scales, the structures of foam materials can be divided into two groups, namely mesostructure and microstructure [83].

2.4.1.1 Mesostructure

The mesoscale is the scale of the arrangement between different phases in foams. For this mesostructure (also called architecture), the material contains the solid and gas phases that are easily identified in the reconstructed volume due to the high attenuation contrast caused by the difference in densities of the two phases. Usually the resolution requirement to obtain the 3D images of the mesostructure by μ XT is not smaller than 50 μm , and a common laboratory μ XT system is sufficient for the 3D mesostructure observation.

Figure 2. 18 shows some typical μ XT images of different foam materials [83]. They are made of metals, polymers, or ceramics. Each image was processed by removing the voxels of the gas phase based on a threshold value of the grey level in the reconstructed image. To create a good representative mesostructure the investigation of both the mesostructure and the overall properties should be performed on adequately large specimens. As a rule of thumb, at least 10 cells across each dimension are acceptable. This implies the magnification of the μ XT scans should not be very high. Generally, this constraint simultaneously prevents the characterization of the internal microstructure, because the resolution used to capture the whole specimen image is not high enough to observe the

microstructure.

Figure 2. 18 Various μ XT images of the mesostructure of several highly porous solids: (a) Aluminum closed-cell foam, (b) Polymer close-cell foam, (c) Nickel open-cell foam, (d) Aluminum open-cell foam, (e) Aluminum closed-cell foam, and (f) Al_2O_3 open-cell foam [83].

2.4.1.2 Microstructure and local tomography

To understand the properties of foam materials, it is also important to analyze the microstructure of the solid phase. This can be achieved by increasing the magnification in μ XT. Such magnification requirement suggests the samples have to be cut into smaller pieces and placed very close to the X-ray source to allow a high resolution in the projection image. Figure 2. 19 (a) shows an example of microstructure analysis of a very small piece of aluminum foam sandwich at the interface between the foam and the skin [84]. As compared with the scale shown in Figure 2. 18, only a small region located at the neck of one of the struts seen at a larger magnification can be observed. Investigators have recently reconstructed a high quality image of syntactic foam in micro scale. Figure 2. 19 (b) shows glass phase in epoxy based syntactic foam [85]. The images were thresholded for a value of the grey level permitting to obtain the glass phase, from which a lot of information can be obtained, such as imperfections in the material, size distribution and the arrangement of glass spheres and so on, indicating the power of μ XT for investigating the microstructure of syntactic foam.

Figure 2. 19 Microstructure of foams: (a) Tomographic reconstruction at high resolution of the interface between the foam and the aluminum sheet in an aluminum foam sandwich [84], (b) Local glass phase in epoxy based syntactic foam [85].

2.4.2 Modeling based on μ XT imaging

The accurate FE geometrical model can be generated from μ XT imaging, because the actual shape of the beams, struts and shell walls of the architecture can be exactly reproduced. A first mesh of the interface between the different phases present in the material is produced [86]. This initial mesh is made of triangles. It is a marching cube fit of the solid-gas interface at the scale of the voxels. This surface is then simplified to reduce the number of triangles while preserving a good description of the surface. To achieve this simplification, small groups of triangles are replaced by equivalent larger triangles. On the basis of this surface mesh, a grid of solid tetrahedral elements is finally generated by an advanced front method. It has recently been implemented for many different engineering types of foams [87-90]. Figure 2. 20 shows some examples of such meshes published in the literatures. But so far, there is no syntactic foam finite element model developed from the μ XT data due to the complexity of the micro structure.

Figure 2. 20 Examples of finite element meshes produced from μ XT data: (a) A PU foam [86], (b) Initial FE mesh of a closed-cell an Al foam, (c) Von Mises stress for an open cell Al foam, (d) Shell elements mesh of a close-cell polymer foam [83].

2.4.3 In situ mechanical tests with μ XT observations

In order to optimize the microstructure of structural materials, it is necessary to fully understand the mechanisms that are controlling their mechanical response. For many years, research has focused on the post-mortem

characterization of the specimens in this field [91]. Usually, the fracture surface of the materials after mechanical testing can be characterised by various types of microscopy to explore the microstructural change in the final stage and analyse the possible deformation process and failure mechanisms [8, 69, 92]. However, the post-test examination overlooks the effects of elastic recovery, shrinkage and crack closure in polymer, and cannot reveal the real-time process of failure. Moreover the applicability of the surface findings to deduce the bulk behavior of the material remains questionable [91]. For example, in the field of damage development under load, some recent studies have clearly shown the differences between results obtained in the bulk and at the surface of strained samples [93, 94]. Therefore, the investigation of the structural evolution in the materials requires the in situ track of the 3D internal deformation.

As discussed in section 2.4.1, the powerful ability of μ XT providing the directly and non-destructive 3D images of internal structure materials has been widely realized. Therefore, it is believed the in situ tests with μ XT inspections can offer a new insight in scientific research [95-97]. The development of in situ experiments monitored by μ XT probably started with the observation of Guvenilir et al. [98]. After that, the number and the type of in situ experiments involving μ XT has also widened considerably and various experimental environments (temperature, stress, corrosive environment etc.) have been studied with not only synchrotron but also laboratory X-ray sources [91, 96, 99]. A good example was reported by Hufenbach et al. [100], he developed an in situ tensile or compressive loading tests rig for μ XT inspection. Figure 2. 21 (a) shows the schematics of the

system, as most in situ loading rig used in μ XT, the main components are the lifting gear and load transfer tube. During a test, the specimen subjected to a controlled load or strain together with the surrounding tube will be scanned, as a result the in situ 3D structural characterization of the material as a function of load can be achieved. The researched used this rig investigate the damage evolution in carbon fibre reinforced plastics (CFRP) specimen. Results showed the internal micro cracks and other forms of internal damage can be clearly observed.

Figure 2. 21 Test device concept: (a) Scheme, and (b) Adjustment in the CT [100].

Now, the potential of μ XT for investigating the mechanical behavior of materials through in situ experiments in various experimental conditions is still be exploited. With the development of X-ray CT techniques, 3D imaging softwares and other specific devices, the in situ experiments with μ XT inspections will play a more and more important role in material research.

2.5 Summary

In this chapter, a brief introduction of syntactic foams, including their constituent materials and the applications, were first reviewed. Then the mechanical behaviors, the strain rate dependency and failure modes of syntactic foams were highlighted. Subsequently, constitutive descriptions, including the calculations to obtain the modulus and strength of syntactic foams from the known constitutive parameters of the microballoons and matrix and the development of finite element modelling in syntactic foam, were introduced. Finally a detailed literature review in the μ XT applied to the characterization of foam materials and the in situ experiments coupling with μ XT inspections were discussed.

Chapter 3

Elastic behavior and failure mechanism in epoxy syntactic foams

3.1 Introduction

A number of research activities have been carried out in the past decades to investigate the bulk mechanical behavior of syntactic foams and the effect of various factors, such as the properties of microballoons and the matrix, microballoon volume fraction and geometry, and strain rate [79, 101-105]. The microballoon volume fraction V is one of the key methods to control the mechanical properties of syntactic foams. As discussed in Chapter 2, it has been reported that the volume fraction of microballoons affects the bulk compressive, tensile and flexural properties as well as fracture toughness and impact resistance of the foam [9, 106-108]. The analytical approaches to calculate the Young's modulus, strength and shear modulus of syntactic foams based on the known constitutive parameters of the microballoons and matrix were also introduced in Chapter 2. However, very few studies have focused on the internal stress evolution during the deformation of syntactic foams, which is important to understand the

microscopic mechanism determining the bulk behavior. Furthermore, the effect of microballoon volume fractions in the microscopic scale was rarely studied.

The failure mechanism in syntactic foams significantly determines the lifespan of a component made of the foam. Three macroscopic failure modes (shear fracture in the diagonal, longitudinal splitting failure along the compression direction, and layered crushing) in the syntactic foam subjected to compression have been reviewed in Chapter 2. The reports analyzed the damage evolution and related mechanism of each failure mode, and indicated that the failure modes vary with the properties of constituents, aspect ratio of the specimen and strain rates. However, the effect of microballoon volume fractions on the failure of syntactic foams is still not well understood especially in the multiple scales, and rarely related to the internal stress field.

In this chapter, the elastic behavior and failure mechanism in epoxy syntactic foams as well as the effect of glass microballoon volume fractions and the radius ratio were investigated. A 3D cubic REV in the foam was generated, in which the microballoons with a statistic size distribution were randomly distributed. The FE model of the REV foam was then developed to predict the localised stress field in the elastic region during the compression of the foam. Uniaxial compression experiments at quasi-static rates were performed on the syntactic foams with different microballoon volume fractions to validate the FE model and to characterise the stress–strain curves and failure process of the foam. The predicted localised stresses were related to explore the failure mechanism for

different microballoon volume fractions. Based on the FE predictions and experimental observations, the failure within the microballoons and matrix was proposed to understand the bulk failure process.

3.2 Experimental procedure

3.2.1 Materials and microstructure

The syntactic foams were fabricated by mechanically mixing the soda-lime-borosilicate glass microballoons (3MTM Scotchlite micro hollow spheres K25 and S60 were used as the fillers, the porosity is present in the closed microballoons.) with Epicote 1006 epoxy resin (matrix). The amount of glass microballoons was controlled to achieve the foams with the respective glass microballoon volume fractions $V = 0.1, 0.2, 0.3$ and 0.4 . The detailed preparation process is explained as follow, and schematic of the corresponding steps are shown in Figure 3. 1:

- 1) The hardener component was added to the epoxy resin and thoroughly mixed. The blending ratio is 10 parts of resin to 6 parts of hardener by weight.

- 2) The next step is adding the microballoons to the epoxy matrix while stirring the mixture. The microballoons were added in multiple steps to avoid agglomeration. The stir speed was slow to minimise both the damage of glass microballoons and the occurrence of air bubbles in the epoxy.

3) After the mixture became the uniform slurry, it was left in a vacuum oven for 10 min at room temperature to further reduce the air bubbles.

4) Subsequently, the mixture was again stirred slowly for another 20 min before it started to become the gel, because the glass microballoons (bulk density: K25 $\approx 250 \text{ kg/m}^3$ and S60 $\approx 600 \text{ kg/m}^3$) have the tendency to float to the surface of the epoxy matrix (density $\approx 1100 \text{ kg/m}^3$).

5) Then the syntactic foam was cast in an aluminium mould coated with the release agents and was subsequently cured for 24 hours at room temperature.

6) Finally, the specimens were took out from the mould and machined into the required dimensions.

Figure 3. 1 The detailed procedure of the syntactic foam preparation.

In this study, SFK25 and SFS60 were used to represent the syntactic foam filled with K25 and S60 glass microballoons respectively.

Figure 3. 2 illustrates the microstructure of SFK25 and SFS60 syntactic foams with the microballoon volume fraction $V = 0.3$ as characterised by SEM. The glass microballoons are uniformly distributed in the epoxy matrix, resulting in a homogeneous microstructure in the foam.

Figure 3. 2 The SEM images of SFK25 and SFS60 syntactic foams with a glass microballoon volume fraction $V = 0.3$ at different magnification.

The raw glass microballoons K25 and S60 were examined in SEM; the outer diameters of individual microballoons were quantified from the SEM images and statistically analysed. Figure 3. 3 shows the detailed procedure of the statistic work.

Figure 3. 3 The process of the statistical analysis of microballoons diameters.

1) Put the glass microballoons on the conductive adhesive tape, and coat the microballoons with thin gold layer. And then SEM was used to observe their outer diameters.

2) Black circles were used to represent the circle microballoons. Noted the small ones (S60: $D < 10 \mu\text{m}$, K25: $D < 20 \mu\text{m}$) were not considered. Because the small microballoons are very few (see Table 3.1.), and their influence on the bulk foam is very limited.

Table 3.1 Particle size distribution provided by the manufacturer

Microballoon type	Particle Size Distribution (μm)			Effective top size (μm)	η
	10 th %	50 th %	90 th %		
K25	25	55	90	105	0.96
S60	15	30	55	65	0.85

3) To make sure the sample size is big enough for the statistically analysis, a number of SEM images were used to extract the black circles.

4) All the black circles were import to ImageJ for the particle size analysis. A threshold grey level was first given to mark the circles. The watershed was then used to separate the overlap circles. The output is the area of each circle.

It was found that the outer diameter nearly follows a log-normal distribution for both K25 and S60 microballoons (Figure 3. 4). For the majority of microballoons by volume, the outer diameter (D) range of K25 and S60 are in 25 to 105 μm and 10 to 65 μm . This agrees with the material data provided by the manufacturer (Table 3.1).

Figure 3. 4 The log-normal distribution of outer diameter of K25 and S60 glass microballoons measured from SEM.

The SEM observations on the microballoon debris reveal that the wall thickness of K25 ($t = \sim 1 \text{ } \mu\text{m}$) and S60 ($t = \sim 2 \text{ } \mu\text{m}$) are nearly constant within individual microballoons and among them regardless of their outer diameters Figure 3. 2. To explain the difference of K25 and S60 microballoons caused by the variation of outer diameter and wall thickness, a parameter called radius ratio, η , was introduced and defined by the following equation:

$$\eta = \frac{r_i}{r_o} = \frac{r_o - t}{r_o}$$

Where t is the wall thickness of the microballoon, r_i is the internal radius and r_o is the outer radius of the microballoon (the radius ratio for K25 and S60 microballoons is listed in Table 3.1). Changing the value of η does not change any other parameters such as microballoon surface area and microballoon/matrix interfacial strength. However, change in η leads to a different wall thickness in the same size microballoons. Therefore, it affects the mechanical properties of the microballoons, such as the modulus and strength of the microballoons [61, 62].

3.2.2 Mechanical tests

Cylindrical specimens of $d = 10$ mm diameter and $l = 10$ mm length, thus an aspect ratio of $l/d = 1.0$, were machined from the fabricated syntactic foam rods. The specimen diameter was large enough to ensure that the specimen behavior is representative of the bulk foam. Uniaxial compression tests were conducted on the foam specimens in an INSTRON 5569 universal mechanical testing machine fitted with a 50 kN load cell at room temperature. In the displacement control mode, a cross-head speed of 0.01 mm/s was applied to achieve a quasi-static strain rate of 0.001 s^{-1} . To minimize the errors due to the machine deformation, an axial clip-on extensometer was used to accurately measure the displacement of the specimens. The deformation history of the specimens was recorded in a JAI BM-500 high resolution camera.

To quantify the Young's modulus of syntactic foams, the specimens with higher aspect ratio ($d = 5.0$ mm, $l = 10$ mm, and $l/d = 2.0$) were machined and then tested under quasi-static compression (0.001 s^{-1}). A pair of TML strain gauges was mounted on the opposite sides of the specimens to more accurately measure the longitudinal strain. Figure 3. 5 shows a pair of strain gauges mounted specimen and the schematic of the compressive test. Only the elastic region from 0 to 0.03 strains was measured as the strain gauge used in this study can is limited up to 0.03.

Figure 3. 5 The strain gauges mounted specimen with higher aspect ratio of 2.

All the specimens were carefully lubricated with the Castrol LMX grease to minimize the interfacial friction during compression. At least three specimens were tested under the same conditions. The compressive tests with various specimen dimensions are summarized in Table 3.2.

Table 3.2 The compressive tests with various specimen dimensions.

Specimen	SFK25 and SFS60	
Measured strain	0 - 0.02 (mm/mm)	0 - ~0.75 (mm/mm)
Specimen dimensions	$l = 10 \text{ mm} \times d = 5 \text{ mm}$	$l = 10 \text{ mm} \times d = 10 \text{ mm}$
Strain gauge	√	×
Output	Young's modulus (The accurate young's modulus can be used to calibrate the whole σ - ϵ curves.)	The whole σ - ϵ curves, peak stress σ_{pk} and critical stain ϵ_{cr} .

3.3 Finite element modelling

A 3D FE model of a cubic representative elementary volume in the syntactic foam was established using the implicit solver in ABAQUS to predict the localized stress field and deformation in the glass microballoons and epoxy matrix, which are then linked to explore the macroscopic behavior of the foam. Figure 3. 6 shows the procedure to generate the geometrical model of the microballoons randomly embedded in the REV of the matrix.

Figure 3. 6 The flowchart of generating the random location and outer diameter for a set of glass microballoons in a given REV of the epoxy matrix.

The SFK25 REV is used as the example to illustrate the details of the geometrical model generating. The out diameter of K25 microballoons in the FE models were assumed to range from 20 to 105 μm , and follow a log-normal distribution, based on the statistic information of the microballoon outer diameter as experimentally observed (Figure 3. 4) and provided by the manufacturer (Table. 1). For the target microballoon volume fraction (V can be any value lower than 0.45 by using the algorithm in Figure 3. 6. To compare with the experimental investigations, V fixed at 0.1, 0.2, 0.3 and 0.4 in the REV) in the defined $0.3 \times 0.3 \times 0.3 \text{ mm}^3$ REV (the elementary volume must be large enough to be representative of the overall behavior of the foam. It was reported that the volume size must be at least five times the mean cell size [86], hence the SFK25 REV size was selected as $0.3 \text{ mm} \times 0.3 \text{ mm} \times 0.3 \text{ mm}$ as the average diameter of K25 microballoon is 53 μm), the outer diameter for a set of microballoons was estimated according to the experimentally observed log-normal distribution. The location of each microballoon in the set was randomly generated as shown by the flowchart in Figure 3. 6. The location was evaluated such that no overlap between the microballoons can occur. The MATLAB software was used to code the algorithm to generate the random location and outer diameter of a series of microballoons in the REV foam (Figure 3. 7). Similarly, the SFS60 REV was also developed, but the volume size was selected as $0.2 \times 0.2 \times 0.2 \text{ mm}^3$ as the mean diameter of S60 microballoon is about 26 μm .

Figure 3. 7 The geometrical model of a REV in syntactic foam.

The geometry of syntactic foams in MATLAB was transferred to ABAQUS using Python scripts. The wall thickness of the glass microballoons K25 and S60 was assumed to be $t = 1 \mu\text{m}$ and $t = 2 \mu\text{m}$ in the FE model. Both the microballoons and matrix were meshed with tetrahedral elements (Figure 3. 8). As compared Figure 3. 2 and Figure 3. 8, the geometry features of the FE model are consistent with those observed in SEM image, for example, the volume (area in the 2D) of microballoons in the REV, the outer diameter distribution, and the randomly dispersed locations.

Figure 3. 8 The FE meshes in a cross section.

Only the elastic deformation was simulated for the REV syntactic foam. As no debonding between the glass microballoons and epoxy matrix arises in the elastic region and their deformation occurs simultaneously, a tie constraint in ABAQUS was applied to the interface between the microballoons and matrix. The FE model of the REV foam was subjected to symmetrical boundary conditions. The bottom surface of the REV was fixed whilst the top surface subjected to a prescribed velocity equivalent to a strain rate of 0.001/s. The elastic properties of the two constituents were specified in the FE model: the Young's modulus of $E_{gl} = 60$ GPa and Poisson ratio of $\nu_{gl} = 0.21$ for the glass, and $E_{ep} = 2.2$ GPa and $\nu_{ep} = 0.36$ for the epoxy resin [79].

3.4 Results and discussion

3.4.1 Bulk compressive response of syntactic foams

Figure 3. 9 and Figure 3. 10 show the representative stress–strain curves of SFK25 and SFS60 syntactic foams with various glass microballoon volume fractions ranging from $V = 0.1$ to 0.4 under compression at quasi-static rates of strain. Note that a good repeatability ($< 5\%$) was achieved for all the testing conditions. Similar to most porous materials [61, 109-111], three different regions can be identified in the curves regardless the volume fractions or the radius ratio of the glass microballoons.

I. Elastic Region: an initial portion, which is first approximately linear and then non-linear, corresponds to the elastic behavior of the syntactic foam (refer to Figure 3. 9 and Figure 3. 10). The elastic region is limited up to the critical strain $\varepsilon_{cr} = (4 \pm 0.5) \%$ in the foam where the peak stress (σ_{pk}) is reached. The Young's modulus of the foam can be calculated by the slope in the initial linear portion of the stress–strain curves for the specimens with $l/d = 2.0$.

II. Plateau Region: a drop in stress arises after the peak stress, implying that glass microballoons start to become crushed and the syntactic foam begins to yield. In the plateau region, the change of stress is not significant although it can slightly increase, decrease or maintain nearly constant depending on the microballoon volume fraction. This is mainly attributed to the simultaneous microstructural change: the crushing of microballoons and the propagation of micro-cracks and

macro-cracks in the epoxy [69]. The plateau region is also associated with the energy dissipation capability which can be defined by the average stress.

III. **Densification Region:** this region is characterised by a significant increase of stress in the stress–strain curves. When most microballoons are crushed and compacted, the syntactic foam is compressed and thus densified into a bulk material.

The elastic and plateau regions of syntactic foams are critical to most industrial applications, such as energy dissipation. Hence, further examination is required on both the macroscopic and microscopic behavior of the foam in these two regions.

Figure 3. 9 The measured stress - strain curves of SFK25 syntactic foams with different volume fractions of glass microballoons. Only the representative curve is shown for each volume fraction.

Figure 3. 10 The measured stress - strain curves of SFS60 syntactic foams with different volume fractions of glass microballoons. Only the representative curve is shown for each volume fraction.

3.4.2 Microscopic and macroscopic behavior in the elastic region

The deformation process and internal stress distribution of syntactic foams were simulated in the developed FE model. As compared in Figure 3. 11 and Figure 3. 12, the predicted stress–strain curves in the elastic region match those measured from the foam specimens with higher aspect ratios ($l/d = 2.0$) for various microballoon volume fractions. The agreement in the curves validates the FE model. It should be noted that the predicted Young’s modulus is about 10% higher than the measured one (Table 3.3). The minor difference from the experimental results is attributed to the approximation in the geometry of the FE model. For instance, the air bubbles were ignored in the model, although they still exist in the matrix in particular with high microballoon volume fractions.

Table 3.3 The comparison of the experimental and predicted Young’s modulus

V%	Young’s modulus of SFK25 (MPa)		Young’s modulus of SFS60 (MPa)	
	Experimental	Predicted	Experimental	Predicted
0.1	2038.98	2105.39	2281.70	2352.29
0.2	1801.87	1950.51	2334.02	2576.19
0.3	1679.31	1853.74	2457.86	2760.79
0.4	1618.46	1750.74	2660.73	2959.50

Figure 3. 11 The measured and predicted stress - strain curves in the elastic region of SFK25 syntactic foams with two volume fractions of glass microballoons.

Figure 3. 12 The measured and predicted stress - strain curves in the elastic region of SFS60 syntactic foams with two volume fractions of glass microballoons.

3.4.2.1 Localized stress distribution within syntactic foams

Figure 3. 13(a) and Figure 3. 13(c) demonstrate the maximum principal stress field in SFK25 and SFS60 syntactic foams with different microballoon volume fractions. The localised stresses in the microballoons are considerably higher compared to those in the matrix; this is the result of the comparatively higher Young's modulus of glass microballoons relative to the epoxy. Therefore, the microballoons are the constituents in the foam to carry the majority of the applied loads. In the foam, the stress in an individual microballoon increases with its outer diameter (see Figure 3. 14); the maximum stress in the entire REV foam occurs on the inner surface of the biggest microballoon. This indicates that the failure of microballoons is most likely to originate at the biggest ones.

Within an individual microballoon, the stress decreases with the distance from the equator and concentrates near the annular equatorial area. This is consistent with the findings reported on the stress distribution in glass and carbon microballoons [112, 113] as well as metallic hollow spheres [114]. The stress concentration near the equator suggests that a microballoon fractures through vertical splitting along the compressive loading axis, which has been experimentally observed on ceramic hollow spheres by Chung et al. [115].

Figure 3. 13 The maximum principal stress distribution in the glass microballoon filled epoxy matrix syntactic foams: (a) SFK25 and (c) SFS60, and the von Mises stress distribution in the epoxy matrix of syntactic foams: (b) SFK25 and (d) SFS60.

Figure 3. 14 The 3D maximum principle stress distribution in K25 and S60 microballoons.

As shown in Figure 3. 13(b) and Figure 3. 13(d), the stress in the epoxy matrix is non-uniformly distributed and concentrates in the following zones: the vicinity of the top and bottom of any individual microballoons, and the connection between adjacent microballoons. The increased microballoon volume fraction causes the reduced distance between microballoons and more stress concentration in the matrix. Micro-cracks will initiate at the stress concentration zones in the matrix.

3.4.2.2 Effect of volume fraction and radius ratio of glass microballoons on bulk elastic properties

The bulk elastic properties of syntactic foams, such as the Young's modulus E , critical strain ε_{cr} and peak stress σ_{pk} , vary with the glass microballoon volume fraction as illustrated in Figure 3. 15, Figure 3. 16 and Figure 3. 17. The critical strain ε_{cr} is corresponding to the strain value where the peak stress occurs. The peak stress is the maximum stress value at the transitional section between the elastic and plateau regions, it also indicates the initiation of the microballoon crushing. As mentioned in section 3.2.1, the dissimilarity of the microballoons can be attributed to the radius ratio. Therefore by comparing the elastic properties of SFK25 and SFS60, the effect of radius ratio of the microballoons on the bulk foam mechanical properties can also be found out.

Figure 3. 15 Effect of glass microballoon volume fractions on the Young's modulus E of the syntactic foams: (a) SFK25 and (b) SFS60.

Figure 3. 16 Effect of glass microballoon volume fractions on the critical strain ε_{cr} of the syntactic foams: (a) SFK25 and (b) SFS60.

Figure 3. 17 Effect of glass microballoon volume fractions on the peak stress σ_{pk} of the syntactic foams: (a) SFK25 and (b) SFS60.

SFK25 and SFS60 syntactic foams show the different trend in Young's modulus with the increase of the microballoon volume fractions (Figure 3. 15). The adding of higher radius ratio glass microballoon (K25, thin wall microballoons) reduced the young's modulus of the final syntactic foam (Figure 3. 15(a)), this is because the glass microballoon K25 can be considered a weakening phase due to its lower modulus than that of the epoxy matrix. According to the rule of mixture, the resultant Young's modulus of the syntactic foam will decrease with the volume fraction of the weakening phase (K25). Similar results were reported by Woldesenbet and Pete [103]. However SFS60 syntactic foams show the inverse effect in the bulk foam Young's modulus with the increase of the microballoon volume fractions. This is because the modulus of glass microballoon S60 is higher than that of the epoxy matrix due to the thick wall of the microballoons, the inclusion of S60 glass microballoons will harden the syntactic foam. With increase of the hardening phase content, the Young's modulus SFS60 syntactic foam will increase (Figure 3. 15(b)). This experimental result on the Young's modulus agrees with the previous numerical and analytical studies by Bardella and Genna [76].

The critical strain ε_{cr} decreases as more microballoons are added in both SFK25 and SFS60 syntactic foams (Figure 3. 16). In syntactic foams, the peak stress is largely determined by the onset of the fracture of the biggest microballoons. The increase of microballoon volume fractions results in more stress concentration in both the microballoons and epoxy matrix (refer to Figure 3. 13), and therefore can cause both the earlier crushing of the biggest microballoons and the earlier formation of micro-cracks in the matrix. The earlier fracture of the

microballoons leads to the smaller critical strain in the foam with higher microballoon volume fraction, and the phenomenon cannot be affected by the radius ratio of the microballoons. However, noted that Figure 3. 13(a) and (c) have demonstrated the stress in K25 microballoons is higher than that in the S60 microballoons subjected to the same strain for each microballoon volume fraction condition due to the thin wall thickness (Figure 3. 13(a) and (c)). It indicates the K25 microballoons are easier to be crushed under the same strain. On the other hand the K25 microballoons may be crushed at the earlier. Therefore, the critical strain ε_{cr} of SFK25 syntactic foams is comparatively smaller than that of SFS60 for the same microballoon volume fraction.

The weakening phase K25 microballoon not only affected the Young's modulus but also reduced the strength of the syntactic foams (Figure 3. 17(a)). As discussed in last paragraph, the increase in microballoon volume fractions results in more stress concentration in the microballoons, hence the stress value that can crush the K25 microballoons is easier to be reached in the high microballoon volume fraction syntactic foams. Due to the low strength, K25 microballoons behave more like the porosity rather than the inclusions. With increases of the K25 porosity, the bulk foam shows the decrease peak stress σ_{pk} with the increase of microballoon volume fraction in macro-scope. However, the peak stress of SFS60 syntactic foam is independent of the volume fraction (Figure 3. 17(b)). This is attributed the high strength of S60 microballoon caused by the high wall thickness and smaller diameters (low radius ratio of the microballoons).

3.4.3 Failure mechanism in the plateau region

3.4.3.1 Observations of macroscopic failure process in syntactic foams

The deformation series of SFK25 and SFS60 syntactic foams were recorded during the uniaxial compression experiments (Figure 3. 18 and Figure 3. 19). With the increase of microballoon volume fractions, the macroscopic cracks initiate at the lower strains, and the dominant cracking path on the specimen surface changes from the longitudinal to diagonal direction.

Figure 3. 18 Compressive deformation history of SFK25 syntactic foams with different glass microballoon volume fractions ($V = 0.1$ to 0.4 in (a) to (d)).

Figure 3. 19 Compressive deformation history of SFS60 syntactic foams with different glass microballoon volume fractions ($V = 0.1$ to 0.4 in (a) to (d)).

The change of the macro cracks orientation suggests that the failure mode of syntactic foams in macroscopic scale is significantly influenced by the microballoon volume fraction, but not sensitive to microballoon type. The foam with high volume fraction of microballoons tends to be brittle.

3.4.3.2 Microscopic failure mechanism

The failure of syntactic foams initiates from the fracture of glass microballoons and the formation of micro-cracks in the epoxy matrix. As the foam is compressed in the plateau region, more microballoons are crushed and more

micro-cracks form in the matrix. Figure 3. 20 demonstrates the proposed internal microstructural change in the syntactic foam subjected to uniaxial compression. A microballoon, when crushed, will not bear any loads; thus a void, originally occupied by the microballoon, forms in the foam. The void together with the adjoining micro-cracks in the matrix is likely to develop into a macro-crack. The macro-crack will propagate under the applied load and join the adjacent voids and cracks, thus causing the macroscopic cracking in the foam.

Figure 3. 20 Schematic of the microstructural change in syntactic foams at the elastic and plateau regions during uniaxial compression: (a) low and (b) high glass microballoon volume fractions.

3.4.3.3 Effect of glass microballoon volume fraction on failure mechanism

In the syntactic foam with low volume fraction of microballoons (e.g., $V \leq 0.2$), the distance between microballoons is long (Figure 3. 20(a)). The majority of micro-cracks in the matrix are near the top and bottom of the microballoons, but not the connection between the microballoons. The micro-cracks propagate almost

in the longitudinal direction (Figure 3. 20(a)) where the local stress in the epoxy concentrates (Figure 3. 13(a) and (c)). By joining the neighboring voids (crushed microballoons), the micro-cracks become a macroscopic longitudinal crack in the foam at the late stage of plateau deformation, which can be observed on the specimen surface (Figure 3. 18(a) and (b), Figure 3. 19(a) and (b)). It should be noted that the lateral tension caused by the barreling effect due to friction can also contribute to the formation of longitudinal macro-cracks on the surface.

There are more glass microballoons per unit volume matrix in the syntactic foam with high volume fraction of microballoons (e.g., $V \geq 0.3$, see Figure 3. 20(b)). Compared to low volume fraction, more voids arise in the matrix because more microballoons are crushed in the plateau region of compression. The stresses in the matrix concentrate not only near the top and bottom of a microballoon but also in the connection between microballoons. Therefore there are more micro-cracks forming in the matrix in all possible directions. As the foam is deformed, the micro-cracks join the adjacent voids to form a macro-crack in the preferred diagonal direction. The macro-crack propagates in the specimens and on the surface (Figure 3. 18(c) and (d), Figure 3. 19(c) and (d)).

3.5 Summary

A 3D finite element model of cubic REV syntactic foams was developed to predict the internal stress field in the glass microballoons and the epoxy matrix. The localised stresses were then related to investigate the elastic behavior and failure mechanism of the foam as well as the effect of the microballoon volume

fraction and the radius ratio of the microballoons. The conclusions were drawn as follows.

- The stress in glass microballoons is significantly higher than that in the matrix. Thus the microballoons are the key constituents to bear the majority of the applied loads. The stress in the matrix concentrates close to the top and bottom of microballoons; it is also considerably localised on the connection between microballoons for high microballoon volume fractions. Micro-cracks may originate at the stress concentration areas in the matrix.
- The stress in a glass microballoon concentrates on the equator. Thus the microballoons fail by the vertical splitting fracture along the compression direction, thus leaving the voids in the matrix after fracture. The voids joining the adjacent micro-cracks in the matrix can develop into a macro-crack.
- The failure of syntactic foams initiates due to the fracture of microballoons and the formation of micro-cracks in the matrix. The glass microballoon K25 weakens the foam whereas S60 is the hardening phase in the foam; the onset of microballoon fracture determines the peak stress. It was thus found that with the increased microballoon volume fraction: a) the Young's modulus, critical strain at peak stress and the peak stress decrease in SFK25 syntactic foam. However b) the Young's modulus increases but the critical strain at peak stress decreases, the peak stress nearly keep constant in SFS60 syntactic foam.

- Experimental observations reveal that the failure mode of syntactic foams was significantly affected by the microballoon volume fraction, but not sensitive to microballoon type. At low volume fraction, the glass microballoons are sparsely distributed in the foam. The micro-cracks tend to propagate towards the areas near the top and bottom of microballoons where the stress concentrates in the matrix, and thus forming a longitudinal macro-crack. However, at high volume fraction, the glass microballoons are more closely packed. The micro-cracks propagate in all possible directions in particular the connection between microballoons. They will form a macro-crack in the diagonal direction by joining the adjacent voids.

Chapter 4

X-ray micro-tomography and finite element modelling of compressive failure mechanism in cenosphere epoxy syntactic foams

4.1 Introduction

A cenosphere produced as the by-product of coal combustion is a hollow ceramic microsphere with small wall thickness to diameter ratio [116]. The cenosphere has become the ideal filler to be added into the polymer matrix for developing lightweight syntactic foams due to the low cost and the low bulk density [117, 118]. The application of cenosphere polymer syntactic foams in load bearing structures requires the full understanding of the deformation and failure mechanism in various length scales.

In this chapter, the underlying failure mechanism in cenosphere epoxy syntactic foams subjected to uniaxial compression was investigated. The μ XT combined with interrupted compression was performed to directly observe the internal morphological change of the cenospheres and matrix during the failure process of the foam. A 3D FE model of the full scale foam specimen was

developed to predict the localised stress field and the fracture of cenospheres during the compression of the foam. In addition, the failure of hollow spheres needs to be included in the FE model to simulate the phenomena in the plateau region of the foam. The FE predictions, experimentally validated, were linked to the μ XT observations to further analyse the microscopic failure mechanisms of the cenospheres and epoxy matrix in the foam as well as their relation to the bulk behavior of the foam.

4.2 Experimental details

4.2.1 Materials and specimens

The syntactic foams were fabricated by mechanically mixing the CENOSTAR ES200/600 cenospheres and Epicote 1006A epoxy resin. The detailed fabrication procedure was described in chapter 3. The volume fraction of cenospheres was controlled to be $V = 0.3$ in the foam. In this work, the syntactic foam rods were machined to the large (diameter $d = 10$ mm and length $l = 10$ mm) and small ($d = 3$ mm and $l = 3$ mm) cylindrical specimens, both with an aspect ratio of $l/d = 1.0$.

4.2.2 Uniaxial compression tests

To quantify the stress–strain curve, uniaxial compression experiments at the quasi-static strain rate of 0.001 s^{-1} were conducted on the syntactic foam specimens with $d = 10$ mm and $l = 10$ mm in an INSTRON mechanical testing machine with a 10kN load cell. At least five specimens were tested under the same condition.

4.2.3 Uniaxial compression tests combined with μ XT inspections

Yxlon X-ray CT system was applied to perform the tomography experiments in this study. The specimen was placed between X-ray source and detector. The source to specimen distance and detector to specimen distance are 23 mm and 680 mm respectively, giving a geometrical magnification of ~ 30 . X-ray source was sent to the specimen mounted on the rotator, working at 59 kV voltage and 45 μ A current. A set of 720 projections was taken within 360° (0.5° increment). The typical scan time is of the order of 80 min. The detector used to record the projections was a CCD camera with 1848×1480 sensitive elements. The projection images were then used by a reconstruction software to obtain the 3D distribution of the linear X-ray attenuation coefficient within the sample. This distribution forms a 3D image which elementary unit is called a voxel and can be viewed with the appropriate visualization software. The specimen in this study was reconstructed by Reconstruction Studio. Each reconstructed volume contains 1024^3 voxels. The reconstruction time (weighted filtered back projection, Feldkamp algorithm) on a Desktop computer with an Intel xeon quad core processor, 3.2 GHz CPU, 8 GBytes RAM, takes approximately 20 min for a 1024^3 voxels 3D image. The voxel size was approximately 5 μ m. The cross sectional image or slice can be produced from the reconstruction result, and visualized by Avizo Fire.

It should be noted that the μ XT scans were performed on the syntactic foam specimens with $d = 3$ mm and $l = 3$ mm (Figure 4. 1) in this work. In order to track the microstructural change during the uniaxial compression, the specimen

sandwiched with two plates was mounted into an in-house compressive loading rig (Figure 4. 1 (a)). Figure 4. 1 (b) shows the photograph of the loading rig, one of the end grips remained fixed to the central tube while the other can move to compress the specimen by driving the screw on the top of the rig. The displacement of the moving end grip can be controlled by the cycles of the screw rotated and interrupted at various levels. Some of the parts in the rig including the tube, plates compressing the specimen were made of polycarbonate to avoid the hiding of X-rays which may minimise x-ray attenuation by the rig. Polycarbonate is a suitable material to make the loading rig, because it is a kind of polymer has high strength and low density.

Figure 4. 1 The X-ray microtomography fitted with an in-house compressive loading rig for a syntactic foam specimen: (a) the schematic of the test, and (b) the photograph of the loading rig.

During the tests, the syntactic foam specimen was lubricated with the Castrol LMX grease to minimise the interfacial friction between its surface and the compression plates. The deformation of the syntactic foam was stopped at the strain value of $\epsilon = 0, 0.1, 0.2, 0.3, 0.4$ and 0.5 . In addition, μ XT scans were then conducted on the foam specimen compressed in the loading rig at each step, the typical X-ray projections of the specimen under the various strain are shown in

Figure 4. 2. It should be pointed out that to simplify the design of the in-house loading rig, no load cell was integrated to measure the load.

Figure 4. 2 The typical X-ray projections of the cenosphere epoxy syntactic foam specimen under the various strains.

4.2.4 Observations of microstructure in syntactic foams

Figure 4. 3 illustrates the morphology and distribution of cenospheres in the epoxy syntactic foams under no compression as characterised by the μ XT. The

cenospheres are uniformly dispersed within the epoxy matrix, thus leading to a homogeneous microstructure in the foam. The outer diameter for the majority of cenospheres ranges from 200 to 600 μm with an average of 350 to 400 μm , consistent with the data provided by the manufacturer. Therefore, the compressive behavior of the specimens ($d = 3 \text{ mm}$ and $l = 3 \text{ mm}$ as well as $d = 10 \text{ mm}$ and $l = 10 \text{ mm}$) is representative of the bulk syntactic foam.

Figure 4. 3 X-ray microtomography image of the microstructure of a cenosphere epoxy syntactic foam. (Noted: the diameter $d = 3\text{mm}$ and the length $l = 3 \text{ mm}$)

4.3 Finite element modelling

A full scale 3D FE model of the syntactic foam ($d = 3 \text{ mm}$ and $l = 3 \text{ mm}$) subjected to uniaxial compression was established in ABAQUS/Explicit to simulate the localised stress field, deformation and failure in the cenospheres and

epoxy matrix. An in-house MATLAB code as detailed in Chapter 3 was used to generate a set of cenospheres that are randomly distributed in the syntactic foam with a volume fraction of $V = 0.3$. The geometrical model of the foam was then created in ABAQUS using the Python scripts (Figure 4. 4).

Figure 4. 4 The full scale geometrical model of a cylindrical syntactic foam specimen with $d = 3\text{mm}$ and $l = 3\text{ mm}$.

The cenosphere outer diameter was assumed to range within 200 to 600 μm and follow a log-normal distribution as experimentally observed and statistically determined in Figure 4. 5. In the model, the cenosphere wall thickness was assumed to be 10 μm (Figure 4. 6). As compared in Figure 4. 3 and Figure 4. 4, the geometrical characteristics of the FE model agree well with the x-ray microtomographic observations. The tetrahedral elements were used to mesh both the cenospheres and the matrix (Figure 4. 7).

Figure 4. 5 The fitted log-normal distribution of the measured outer diameters of the cenosphere.

Figure 4. 6 SEM image showing the representative wall thickness in the cenosphere.

Figure 4. 7 The FE meshes in a cross section.

The symmetrical boundary conditions were applied to the FE model of the syntactic foam. The top surface was subjected to a prescribed velocity equivalent to a strain rate of 0.001 s^{-1} ; the bottom surface was fixed. The interface between the cenospheres and epoxy matrix was specified with the tie constraint in ABAQUS.

The constitutive data of the epoxy matrix in the foam were obtained by the uniaxial compression tests on the pure Epicote 1006A epoxy resin. The Young's modulus and Poisson's ratio are 934 MPa and 0.38, respectively. Figure 4. 8 shows the average true stress versus plastic strain curve of the epoxy resin as measured experimentally. This plastic behaviour was defined using the classic plasticity model in ABAQUS. Note that the fracture of the epoxy matrix was not considered in the FE model. The elastic constants of the cenosphere wall material were specified with Young's modulus 74 GPa and Poisson's ratio 0.18 [78]. As the main compositions in the wall material of cenospheres are silica and alumina, the brittle cracking criterion in ABAQUS was used to simulate the failure of the ceramic wall of the cenospheres. The brittle failure initiates if the maximum principal stress

exceeds the tensile strength (462 MPa) of the ceramic material [119]. The corresponding FE elements were deleted where the failure occurred in the model.

Figure 4. 8 The measured average true stress–strain curve of the epoxy resin under quasi-static compression.

4.4 Results

4.4.1 Bulk compressive response of syntactic foams

Figure 4. 9 shows the typical stress–strain curve of the syntactic foam at the quasi-static strain rate. It is found that the curve can be divided into the elastic, plateau and densification regions as well. The initial elastic region ($\varepsilon < \varepsilon_{cr}$) is characterised by an approximately linear stress–strain curve in which the foam deforms elastically. The peak value (σ_{pk}) corresponding to the critical strain (ε_{cr}) occurs where the elastic region transits to the plateau region. The plateau region ($\varepsilon_{cr} < \varepsilon < \varepsilon_D$) comprises a ~30% drop in stress up to the strain $\varepsilon \approx 0.15$ and the

subsequent slight increase of stress until the densification strain (ε_D) is reached. In the densification region ($\varepsilon > \varepsilon_D$), the stress significantly increases with strain and the syntactic foam is densified into a bulk material.

Figure 4. 9 Comparison between the measured and predicted stress-strain curves of syntactic foam. (Note: only the representative measured curve is shown.)

The densification strain (ε_D) is a key material parameter to determine the energy dissipation capacity of the foam. Various methodologies have been proposed to quantify the strain of densification [120, 121]. The strain can be defined at the intersection of the two tangent lines for the plateau and densification regions in the stress–strain curve [121]. As the selection of the tangent lines can be arbitrary, the strain of densification ($\varepsilon_D = 0.59$, refer to Figure 4. 9) for the syntactic foam was calculated using the energy dissipation efficiency methodology as detailed in the literature [120]. The densification occurs at the strain $\varepsilon_D = 0.59$

corresponding to the maximum energy dissipation efficiency in the efficiency versus strain curve that can be derived from the stress–strain response. Given the stress - strain curve of a specimen, its energy absorption efficiency $\eta(\varepsilon_a)$ is defined as the energy absorbed up to a given nominal strain ε_a normalised by the corresponding stress value according to:

$$\eta(\varepsilon_a) = [\sigma(\varepsilon)]_{\varepsilon=\varepsilon_a}^{-1} \int_0^{\varepsilon_a} \sigma(\varepsilon) d\varepsilon$$

the densification strain ε_D is the strain value corresponding to the stationary point in the efficiency - strain curve where the efficiency is a global maximum, i.e $[d\eta(\varepsilon)/d\varepsilon]_{\varepsilon=\varepsilon_D} = 0$ (see Figure 4. 10, $\varepsilon_D = 0.59$).

Figure 4. 10 The energy absorption efficiency versus nominal strain curve of the syntactic foam.

4.4.2 Validation of the FE model

The stress–strain curve of the syntactic foam was predicted in the developed FE model (Figure 4. 9), and the experimental and predicted stress values at the strategic strains are listed in Table 4.1 it is found that the predicted curve matches the measured from the specimens and consists of three regions. Another intuitive way to compare the measured and predicted results is to track the morphological change of constituents in the syntactic foam during the uniaxial compression. As shown in Figure 4. 11 and Figure 4. 12, the x-ray microtomographic slices reveal the microstructural change of the foam at different deformation stages ($\varepsilon = 0$ to 0.5). Figure 4. 13 demonstrates the FE predictions of the internal deformation process in a typical longitudinal slice of the foam at various strain stages ($\varepsilon = 0$ to 0.4). Both the measured and predicted results exhibit the collapse of cenospheres and the plastic deformation of epoxy matrix in the syntactic foam.

Table 4.1 The experimental and predicted stress values at the strategic strains

Strategic strain (mm/mm)	Experimental stress (MPa)	Predicted stress (MPa)	Difference
0.1	26.01	29.05	11.69%
0.2	25.65	29.63	15.52%
0.3	29.93	33.14	10.73%
0.4	36.58	41.54	13.56%

Figure 4. 11 X-ray microtomographic observation of the internal deformation and failure process of the syntactic foam at different strain stages during the uniaxial compression: the longitudinal central slices

Figure 4. 12 X-ray microtomographic observation of the internal deformation and failure process of the syntactic foam at different strain stages during the uniaxial compression: transverse cross-sectional slices at the center of the specimen

Figure 4. 13 Predicted deformation history of a syntactic foam at different strain stages during the uniaxial compression.

The FE model can be validated by the good quantitative (the stress–strain curve in Figure 4. 9) and qualitative (the internal deformation process in Figure 4. 11 and Figure 4. 13) agreement between the experiments and FE predictions. However, the stress level in the plateau region of the predicted stress–strain curve is $< 10\%$ higher than the stress in the measured curve. This slight difference from the experimental results can be attributed to some approximations of the geometrical model. For example, a perfect interfacial bond using the tie constraint was assumed between the ideal cynospheres and the matrix. The air bubbles may still exist in the syntactic foam specimen, but they were ignored in the matrix of the

FE model. It is noteworthy that macro-cracks in the epoxy matrix can be observed in the μ XT transverse slices (refer to $\varepsilon = 0.3$ to 0.5 in Figure 4. 12). Although the failure criterion for the epoxy matrix was not included in the FE model, the predicted localised stress state in the matrix can be used to elucidate the possible failure of the matrix as will be discussed in Section 4.4.3.3.

4.4.3 Failure process in syntactic foams

4.4.3.1 X-ray microtomographic observations of failure process

In order to understand the internal failure process of syntactic foams in more details, the five typical large cenospheres (1 to 5) as marked in Figure 4. 11 were selected from the various locations in a longitudinal μ XT slice of the specimen. The propagation of macro-cracks in the epoxy matrix was also examined in a middle transverse slice of the μ XT images (Figure 4. 12). The internal failure process of the foam can be associated with the morphological change of the marked cenospheres as well as the deformation and failure of the matrix.

At the low strain ($\varepsilon = 0.1$) corresponding to the stress drop in the plateau region, the failure of the foam initiates at the large cenospheres of the central part (e.g., 1 and 2 in Figure 4. 11). This is consistent with the findings reported for aluminium and polymer matrix syntactic foams [35, 46]. These cenospheres (1 and 2) are crushed; and the voids, originally occupied by the cenospheres, become oblate due to the compression. However, the adjoining small cenospheres as well as the large cenospheres in other locations (e.g., 4 and 5) remain intact.

At the strain of $\varepsilon = 0.2$ where the stress starts to increase in the plateau region, the voids left by the crushed cenospheres in the central part (e.g., 1 and 2 in Figure 4. 11) become more oblate owing to the further deformation. Almost no visible deformation can be found in ceramic cenospheres in the top and bottom of the foam specimen. The longitudinal μ XT slice at $\varepsilon = 0.2$ reveals that the cenospheres 3 to 5 are nearly undistorted.

At the high strain ($\varepsilon = 0.3$) in the plateau region, the crush of cenospheres 3, 4 and 5 (Figure 4. 11) indicates the severe deformation within the syntactic foam. The majority of cenospheres near the centre and the diagonal of the specimen are also crushed, thus forming the damage bands in the foam. The formation of macro-cracks in the epoxy matrix can be observed in the transverse μ XT slice at $\varepsilon = 0.3$ (Figure 4. 12). This suggests that micro-cracks may form in the matrix earlier at the lower strain (e.g., $\varepsilon = 0.2$ or even 0.1), although they are not detected by the μ XT due to their small dimensions and the relatively larger μ XT voxel size ($\sim 5 \mu\text{m}$).

At the even higher strains ($\varepsilon = 0.4$ and 0.5) prior to densification, the remaining cenospheres in the syntactic foam become gradually crushed (Figure 4. 11). The voids thereby left are subjected to considerable deformation. The macro-cracks propagate in the matrix by joining neighbouring voids and other cracks to become larger macro-cracks throughout the specimen (Figure 4. 12).

In summary, the μ XT slices reveal that the cenosphere collapse and inhomogeneous matrix deformation in the centre of the syntactic foam at lower strains. At higher strains, the damage evolves preferentially diagonally in the foam.

4.4.3.2 Fracture of individual cenospheres

Figure 4. 14 shows the evolution of the maximum principal stress distribution in syntactic foams as predicted by the FE model. Like the glass microballoons epoxy syntactic foam, the localised stresses in the cenospheres are also noticeably higher than those in the epoxy matrix, as a result of the higher Young's modulus of cenospheres than the epoxy. Hence, the cenospheres are the main load bearing constituents in the foam.

Figure 4. 14 Predicted maximum principle stress distribution in a syntactic foam at different strain stages during the uniaxial compression.

Figure 4. 15 illustrates the maximum principal stress distribution and the failure process in four adjacent cenospheres at different strain stages. The stresses

in individual intact cenospheres increase with their diameter. Therefore, the failure of cenospheres is most likely to originate in the largest ones; this is experimentally validated by the μ XT observations (refer to $\varepsilon = 0.1$ in Figure 4. 11).

Figure 4. 15 Predicted maximum principle stress distribution and failure process in the representative individual cenospheres in a cenosphere epoxy syntactic foam during the uniaxial compression.

The stress within any individual cenosphere concentrates near the annular equatorial area and decreases with the distance from the equator (Figure 4. 15). The similar findings have been reported on glass [7], carbon [113] and metal [114] hollow spheres. Under the compressive load, the equator expansion results in the tensile maximum principal stress state near the equator. When the tensile stress exceeds the strength of the ceramic wall material, the cenosphere will fracture along the external loading direction. As demonstrated in Figure 4. 15, the largest cenosphere cracks vertically at the increased strain stage and the stress in it is released. However, the stresses in other adjacent cenospheres increase after the largest cenosphere collapses into fragments.

The morphology of a typical cenosphere in the μ XT images of the foam was tracked and visualised at various strain stages to validate the FE predictions on the cenosphere fracture (see Figure 4. 16). As the strain increases, the vertical cracks occur in the cenosphere that then splits into fragments. The cenosphere may not be completely crushed immediately after the crack initiation, because it is well adhered to the surrounding matrix. The localised tensile stress in the cenosphere causes the cracks to propagate towards the top and bottom, thus leading to the vertical splitting failure mode (i.e., failure along the external compressive loading direction).

Figure 4. 16 X-ray microtomographic observation of the failure process in a representative cenosphere in a syntactic foam during the uniaxial compression.

4.4.3.3 Deformation and failure of the epoxy matrix

As shown in Figure 4. 17, the stress distribution is non-uniform in the epoxy matrix. The stress in the matrix concentrates in the vicinity of the top and bottom of any individual cenospheres as well as the connection between adjacent cenospheres. In the syntactic foam with a cenosphere volume fraction $V = 0.3$, the dominant

stress concentration focuses on the connection zones (Figure 4. 17) especially after the neighbouring cenospheres are crushed (refer to Figure 4. 14). Micro-cracks therefore tend to initiate at the connection zones of the cenospheres in the matrix. The micro-cracks then propagate and join other micro-cracks and the voids left by crushed cenospheres to form the macro-cracks preferentially along the diagonal of the specimen. The μ XT slices reveal the occurrence of macro-cracks at the strains $\varepsilon = 0.3$ and 0.4 in the connection zones (refer to the arrows in Figure 4. 18) after the cenospheres collapse. Note that prior to the formation of macro-cracks, micro-cracks arise in the connection zone, but they are not resolved in the μ XT images because of the limited μ XT resolution.

Figure 4. 17 Predicted von Mises stress field in the epoxy matrix at different strain.

$\varepsilon = 0.2$

$\varepsilon = 0.3$

$\varepsilon = 0.4$

Figure 4. 18 X-ray microtomographic slices showing the failure of the epoxy matrix in a syntactic foam as indicated by the two arrows.

4.5 Discussion

4.5.1 Failure mechanism in syntactic foams

Based on the x-ray microtomographic observations and the FE results, the different failure mechanisms dominate in the low and high strains of the plateau region in cenosphere epoxy syntactic foams.

The initial lower strain portion of the plateau region, where the stress drops, is characterised by the crushing of large cenospheres in the centre of the foam specimen (Figure 4. 11). After the collapse, the load initially carried by these cenospheres is transferred to the adjacent cenospheres and the connection zone in the matrix preferentially in the lateral and diagonal directions (see Figure 4. 14, Figure 4. 15 and Figure 4. 17). As a result, these adjacent cenospheres become crushed and micro-cracks arise in the connection zone. Under compression, this failure process continues until the majority of large cenospheres near the specimen centre are crushed.

At the subsequent higher strain portion of the plateau region where the stress increases slightly, the prevalent microscopic failure includes the crushing of cenospheres as well as the significant plastic deformation and fracture of the matrix. As a result of the transfer of stress concentration from the fractured cenospheres to the neighbouring intact materials (Figure 4. 14, Figure 4. 15 and Figure 4. 17), the collapse of cenospheres sequentially extends diagonally (Figure 4. 11). The new micro-cracks arise in the matrix connection zone between crushed cenospheres and

grow to join the neighbouring micro-cracks and voids left by the crushed cenospheres to become macro-cracks (Figure 4. 12 and Figure 4. 18). At the late stage of the plateau region, almost all the cenospheres are crushed and hence no more voids can be produced and then consumed by the matrix. Therefore, further compression leads to the lateral expansion of the specimen and subsequently the densification region.

4.5.2 Relation between failure mechanism and bulk behavior

Combined the bulk stress – strain curve (Figure 4. 9) and the internal deformation history (Figure 4. 11 - Figure 4. 13), the relation between failure mechanism and bulk behaviour can be summarized. The initial elastic deformation of the cenospheres and epoxy matrix is associated with the linear elastic portion of the stress–strain curve of the syntactic foam. The peak stress corresponds to the onset of the breakage in the large cenospheres. The subsequent drop in stress is attributed to the collapse of the large cenospheres as well as their adjacent ones in the centre of the foam specimen (Figure 4. 11 and Figure 4. 13, $\varepsilon = 0.1$). The subsequent plateau regime can be easily related to the dispersion of cenospheres collapse. In this process, the cenospheres fragmentation will extend laterally and diagonally (Figure 4. 11 - Figure 4. 13, $\varepsilon = 0.2 - 0.4$). During the process, the crushing of cenospheres and the formation of micro-cracks and macro-cracks in the matrix can reduce the overall load bearing capacity. However, the hardening behaviour of the stress–strain curve of the epoxy matrix as experimentally observed increases the load bearing capacity of the foam. The

simultaneous deformation and failure mechanisms in the constituents result in the slight increase of stress in the late stage of the plateau region in the syntactic foam (Figure 4. 11 and Figure 4. 12, $\varepsilon = 0.5$). Finally the densification is the main mechanical response, the stress increase rapidly with the strain ($\varepsilon > 0.59$).

4.6 Summary

The compressive failure mechanism of cenosphere epoxy syntactic foams was investigated using full scale FE modelling and x-ray microtomography coupled with interrupted compression. The predicted localised stress, fracture of cenospheres and deformation in the matrix were related to the μ XT observations to further analyse the underlying failure mechanism especially in the plateau region of the foam. The conclusions were drawn as follows.

- The compressive failure process of syntactic foams in microscopic scale is characterised by two simultaneous phenomena: (1) the crushing of cenospheres and (2) the plastic deformation and fracture of the matrix. These two phenomena are driven and determined by the localised stress state in the constituents.
- The stress concentration in cenospheres is considerably higher than that in the epoxy matrix, thus making the cenospheres the main load bearing constituents in the foam. The failure of cenospheres originates in the largest ones because the localised stress in individual intact cenospheres increases with their diameter. The tensile stress concentration near the annular equatorial area causes the cenosphere to fracture along the external

compressive loading direction and then split into fragments. After the fracture of cenospheres, the stress concentration is transferred to the neighbouring cenospheres and the matrix material.

- The stress in the epoxy matrix mainly concentrates in the connection zone between adjacent cenospheres. After these cenospheres become crushed, the increased stress concentration in the connection zone leads to the formation of micro-cracks. The micro-cracks propagate and join other micro-cracks as well as the voids left by crushed cenospheres to form the macro-cracks preferentially along the diagonal of the foam specimen.
- The failure mechanisms in the two constituents dominate the different strain stages of the bulk stress–strain behavior of the syntactic foam. At the lower strain of the plateau region, the crushing of large cenospheres is prevalent in the centre of the foam specimen. In addition to the cenosphere collapse, the plastic deformation and fracture process in the matrix dominates the higher strain stage.

Chapter 5

X-ray micro tomographic characterization of strain rate dependent compressive behavior and failure mechanism in cenosphere epoxy syntactic foams

5.1 Introduction

It is well known that the mechanical behavior of the materials is significantly affected by the applied strain rate. Recently, more research efforts have been made to explore the high strain rate response of syntactic foams. As discussed in Chapter 2, the compressive dynamic properties of syntactic foams are affected by many factors, such as the geometry and volume fraction of hollow microspheres, and the pre-treatment of the hollow microspheres [71, 103, 122, 123]. However, very little research has focused on the microscopic failure mechanism of syntactic foams subjected to dynamic loads. The post-test SEM [69, 124, 125] and μ XT [126] examination on deformed specimens was commonly used to characterise dynamic failure features. Nevertheless, the mechanism of high rate failure process in the foam was rarely studied.

In this chapter, the compressive behavior and failure mechanism of cenosphere epoxy syntactic foams at different strain rates were investigated, and the evolution of damage (failure in the foam) was quantified. The syntactic foam specimens were deformed to different strain stages in both quasi-static (0.003 s^{-1}) and dynamic (3000 s^{-1}) compression experiments. To our best knowledge, no one has performed the interrupted compressive tests on the syntactic foams under the dynamic strain rate. The μ XT observations were then performed to characterise the internal microstructural change of the foams. Finally an empirical constitutive equation was developed to quantitatively relate the damage to the strain and strain rate.

5.2 Experimental details

5.2.1 Materials and specimens

The Epicote 1006A epoxy resin (matrix) and CENOSTAR ES200/600 cenospheres (fillers) were used to fabricate the syntactic foams. The detailed fabrication procedure was described in chapter 3. The volume fraction of cenospheres was controlled to be $V = 0.3$ in the foam. In this work, the cylindrical specimens of $d = 5 \text{ mm}$ diameter and $l = 5 \text{ mm}$ length with an aspect ratio of $l/d = 1.0$ were finally machined from the syntactic foam rods.

5.2.2 Uniaxial compression tests under quasi-static and dynamic rates

The quasi-static uniaxial compression experiments were conducted on the syntactic foam specimens in an INSTRON 5569 mechanical testing machine with a

10kN load cell at the strain rate of 0.003 s^{-1} . At least five specimens were tested under the same condition. A Split-Hopkinson pressures bar (SHPB) system was used to perform the dynamic compression tests at the strain rate of 3000 s^{-1} .

The quasi-static uniaxial compression experiments are very common and widely used in mechanical testing. In contrast, little attention has been directed towards material behavior under high rates of strain. As the most popular dynamic compression testing, the SHPB system test will be detailed in the next section.

5.2.2.1 Split-Hopkinson pressure bar operation

In compression SHPB tests, the sample is sandwiched between an elastic incident and a transmitted bar. A striker bar is then propelled into the incident bar generating an incident wave. The elastic displacement measurements of these bars are utilized to determine the stress-strain variation at the sample interface. The schematic of a SHPB system is shown in Figure 5. 1.

Figure 5. 1 The schematic of a split-Hopkinson pressure bar (SHPB) system [72].

For accurate operation, the bars must be aligned along the same axis, straight in nature and free to move without any constraints. Such specific alignment

is crucial in ensuring uniform and one dimensional wave propagation within the bars and uniaxial compression of the sample. Constraining the bars motion violates the necessary boundary condition to achieve one dimensional wave propagation in an infinite cylinder and concurrently introduces noise on the wave forms [127].

The length L and diameter D of the pressure bars are chosen to meet a number of criteria for test validity as well as the maximum strain rate and strain level desired in the specimen [127]. The criteria are listed as follow:

- 1) The length of the pressure bars must first ensure one-dimensional wave propagation for a given pulse length; for experimental measurements on most engineering materials, this propagation requires approximately 10 times of the bar diameters.

- 2) To readily allow separation of the incident and reflected waves for data reduction, each bar should exceed a length-to-diameter (L/D) ratio of ~ 20 .

- 3) Higher strain rate specimens demand smaller diameter bars.

- 4) The pressure bar must be at least twice as long as the incident wave if the incident and reflected waves are to be recorded without interference.

Similar to the incident and transmitted bar, the striker bar is constructed from the same material and has the same diameter. Length however, is dictated by the total desired strain rate and strain in the sample. The incident pulse wave length can be altered by varying the striker bar length as the pulse is twice the length of

the striker bar. The impact velocity of the striker bar also determines the amplitude of the incident pulse [73].

Besides the pressure bars, bearing and alignment fixtures to hold the bar in alignment are necessary. Some form of a propulsion device such as compressed gas launcher is needed to launch the striker bar. Strain gauges are essential to record strain variations in the bars and ultimately generate the data needed to obtain the stress-strain response of sample. Strain gauge signal conditioners must have a frequency response of at least 1MHz for adequate data resolution. Gain settings higher than 500 is also needed for high resolution measurement in low stress flow materials such as foam [127]. Instrumentation and data acquisition systems are needed to analyse the strain data.

To conductive a split Hopkinson Pressure Test effectively, the specimen size should be design carefully as well. This includes:

1) The maximum sample diameter d should be less than $D\sqrt{1 - \varepsilon_d}$, where D is the given pressure bar diameter, ε_d is the desired specimen strain [128].

2) An aspect ratio l/d of 0.5 to 1 to ensure minimal inertial and frictional effects ensuring optimal area matching of the sample to the bar [127], such a ratio would also prevent the formation of fragments near cracks which might disrupt uniform compression and prevent barrelling effects during compression [69]. Hence the height of the sample should be in the range of $0.5d$ and d .

In this study the solid aluminium alloy 6061 was used to make the pressure bars, because a lower-modulus material increases the signal-to-noise level, the selection of a bar material with lower strength and lower elastic modulus material for the bars is sometimes desirable to facilitate high-resolution dynamic testing of low-strength materials such as polymers or foams. The length of the incident, transmitted and striker bar was designed as 120 cm, 120 cm and 50 cm respectively, and all the bars have the same diameter (12.7 mm).

When the diameter of the pressure bars was fixed, the specimen dimension can be designed as well. The desired specimen strain was 0.7 (totally failure strain at quasi-static strain rate) to obtain the whole stress-strain curve of the syntactic foam subjected to the dynamic strain rate. Hence the specimen diameter should be less than $12.7 \text{ mm} \times \sqrt{1 - 0.7} = 6.96 \text{ mm}$.

5.2.2.2 Interpretation of Stress-Strain Behavior

The use of long elastic bars for the study of high strain rate mechanical behavior is possible via elastic bar measurements based on sample response due to the well predicted wave propagation behavior. As such it is possible to determine either the strain or the stress at any point along the split Hopkinson pressure test set-up through the measurement of the elastic wave as it moves along the bars.

The data from the strain gauge recordings indicate the strain variations within the incident and transmitted bar in voltage against time. The shape of the incident wave which is different from a typical trapezoidal wave form in SHPB

experimentation can be explained by the use of pulse shaping. The sudden sharp rise in the reflected wave is indicative of the specimen's loss of load bearing ability in turn causing an increase in incident specimen interface velocity [55].

To convert this data into a stress-strain curve, mathematical formulation and manipulation are necessary. Figure 5. 2 shows the incident, reflected and transmitted stress wave directions.

Figure 5. 2 Expanded view of incident bar / specimen / transmitted bar region.

The solution to the wave equation can be described as:

$$\frac{\partial^2 u}{\partial x^2} = \frac{1}{c_b^2} \times \frac{\partial^2 u}{\partial t^2} \quad 5.1$$

or
$$u = f(x - C_b t) + g(x + C_b t) = u_i + u_r \quad 5.2$$

where u is the displacement of the specimen at the incident and transmitted interface, using subscripts i and r to denote the incident and transmitted bar end of the specimen. For the incident bar, f and g describe the incident and reflected

wave shape and C_b refers to the longitudinal wave speed, which can be approximated by:

$$C_b = \left(\frac{E}{\rho}\right)^{\frac{1}{2}} \quad 5.3$$

where E refers to the young's modulus of the bars and ρ is the bar density.

Differentiating the wave equation with respect to x , the strain of the interface between the specimen and the incident bar $\epsilon_{Incident}$ can be determined to be:

$$\epsilon_{Incident} = \epsilon_i + \epsilon_r \quad 5.4$$

ϵ_i , ϵ_r and ϵ_t denotes the incident, reflected and transmitted strains respectively.

Differentiating the wave equation with respect to time, the velocity of the interface between the specimen and the incident bar $\dot{u}_1$ can be determined to be:

$$\dot{u}_1 = C_b (-f' + g') = C_b (-\epsilon_i + \epsilon_r) \quad 5.5$$

Differentiating displacement in the transmitted bar with respect to time yields:

$$\dot{u}_2 = -C_b \epsilon_t \quad 5.6$$

where $\dot{u}_2$ is the velocity of the interface between the specimen and the transmitted bar.

Therefore, the specimen strain rate $\dot{\epsilon}$ can be seen to be:

$$\dot{\epsilon} = \frac{\dot{u}_1 - \dot{u}_2}{L_s} \quad 5.7$$

where L_s refers to the specimen instantaneous length. Combining the above mentioned equations, the specimen strain rate can be resolved as:

$$\dot{\epsilon} = \frac{C_b}{L_s} (-\epsilon_i + \epsilon_r + \epsilon_t) \quad 5.8$$

The forces in the incident and transmitted bar can be described as:

$$F_1 = AE(\epsilon_i + \epsilon_r) \text{ and } F_2 = AE\epsilon_t \quad 5.9$$

where A is the cross sectional area of the bars.

One of the basic assumption is that the specimen is in force equilibrium ($F_1 = F_2$) and is deforming uniformly, hence:

$$\epsilon_i + \epsilon_r = \epsilon_t \quad 5.10$$

Substitute Equation 5.10 into Equation 5.8 yields:

$$\dot{\epsilon} = \frac{2C_b\epsilon_r}{L_s} \quad 5.11$$

From which the nominal strain of the specimen can be obtained by integration the strain rate $\dot{\epsilon}$ with respect to time:

$$\varepsilon = \int \dot{\varepsilon} dt \quad 5.12$$

The nominal stress σ can be calculated from strain gauge signal measurement of transmitted force divided by instantaneous cross sectional area A_s of the specimen:

$$\sigma = \frac{AE\varepsilon_t}{A_s} \quad 5.13$$

The instantaneous area here is deduced from the reflected strain signal in the incident bar and is based on the assumption of volume conservation of the specimen.

5.2.2.3 Interrupted uniaxial compression tests under quasi-static and dynamic rates

In some of the quasi-static compression tests, the deformation of the syntactic foam specimens was controlled to stop at various strains. In quasi-static compression, the different specimens were deformed to the strain $\varepsilon = 0.1, 0.2, 0.3, 0.4, 0.5$ and 0.6 .

In the SHPB tests, as the conventional SHPB has difficulty in controlling the strain, a ring-shaped aluminium stopper was additionally placed around the foam specimen and between the input and output bars to prevent the further deformation of the specimen during the dynamic compression. As shown in Figure 5. 3(a), the sleeves were made of aluminium 6061-T6, the same material as the bars.

A section view may give a better illustration (see Figure 5. 3(b)). The sleeve was put on the interface between the sample and the transmission bar, the left side of the sleeve is like a ring. The ring is shorter than the sample height, so the load from the incident bar will be carried by the ring after the sample deform to the length of L_r . With the stoppers of different thicknesses L_r , the final deformation of the foam specimens in some of the SHPB tests can be controlled, the stop strain $\varepsilon = 0.1, 0.2, 0.3, 0.4$ and 0.5 in this study.

As the stoppers were applied at the end of the transmitted bar, the maximum allowable specimen diameter during deformation is the inner diameter of the stopper ring (8 mm) to ensure the ring will not restrict the lateral expansion of the specimen. The desired strain is 0.5 in the strain controlled SHPB test, so the specimen diameter should be less than $8 \text{ mm} \times \sqrt{1 - 0.5} = 5.66 \text{ mm}$. Combined the discussion in section 5.2.2.1, the dimension of the cylindrical specimen was designed as $d = 5 \text{ mm}$ diameter and $l = 5 \text{ mm}$ length with an aspect ratio of $l/d = 1.0$ were used in all the dynamic tests.

Figure 5. 3 The schematic of the stopper: (a) the isometric view and (b) the cross-section view.

5.2.4 X-ray micro tomography observations of the syntactic foam specimens

The syntactic foam specimens deformed at different strains were then subjected to slow elastic recovery. Subsequently the μ XT at 80 kV and 18 μ A was performed to scan these specimens. The effective voxel size was approximately 8 μ m. The AVIZO/FIRE software was used to visualise the internal morphology of the foam.

5.3 Results

5.3.1 Bulk compressive behavior at different strain rates

Figure 5. 4 shows the typical compressive stress–strain curves of cenosphere epoxy syntactic foams at the quasi-static and dynamic strain rates. A good repeatability in stress was achieved in the tests. The bulk stress–strain curves are similar in shape at both the low and high strain rates, and can be categorised into three regions (Figure 5. 4). The initial, approximately linear region corresponds to the elastic deformation. The peak stress (σ_{pk}) then occurs and is followed by a drop in stress. The subsequent plateau region is characterised by the nearly constant stress (i.e., plateau stress σ_{pl}). Both the peak and plateau stresses are the main characteristics of the foam [7, 69]. In the densification region, the foam is compressed into the bulk material and thus the stress increases rapidly. As also demonstrated in Figure 5. 4, the bulk stress of the syntactic foam is affected by the strain rate despite the similarity in the shape of low and high rate stress–strain curves. For instance, the peak and plateau stresses at dynamic rates are two to three times higher than those at the quasi-static rates. As summarised by Li et al. [69], a number of microscopic factors may contribute to the strain rate dependency of bulk behavior in syntactic foams. The strain rate sensitivity of the epoxy matrix thanks to the viscoelasticity [125] is likely one of the key factors to determine the bulk rate dependency of the foam.

Figure 5. 4 Representative stress–strain curves of cenosphere epoxy syntactic foams at the quasi-static and dynamic strain rates.

5.3.2 Quasi-static failure process

Figure 5. 5 illustrates the internal microstructural change of the syntactic foams that were deformed to different strains in quasi-static compression experiments and then recovered. The six strain stages were in the plateau region of the compressive response. At the low strain $\varepsilon = 0.1$, while some large cenospheres fail in the central portion of the specimen, the fragments are adhered to the epoxy matrix walls. After the compressive load is released, the failed cenospheres seem intact due to the recovery of the matrix. Therefore, no obvious internal failure was observed in the foam at the $\varepsilon = 0.1$ in Fig. 2. At the increased $\varepsilon = 0.2$, the cenospheres continue to fracture in the central part where the hydrostatic stress is considerable [69]; and the fragments debond off the matrix walls. The spaces left by the crushed cenospheres become oblate while the top and bottom portions of the foam specimen seem unchanged (Figure 5. 5). At the high strain $\varepsilon = 0.3$, the crushing of cenospheres occur along the diagonal of the specimen, in addition to the central part. At the even higher strains $\varepsilon = 0.4$ to 0.6 , the majority of the remaining cenospheres are crushed in the foam. Meanwhile, micro-cracks of the epoxy matrix form and propagate throughout the specimen, causing the separation of the foam specimen (see $\varepsilon = 0.6$ in Figure 5. 5). The deformation history is similar with the result obtained by in situ compression combined with the μ XT detailed in Chapter 4.

Figure 5. 5 The μ XT longitudinal slices of the internal deformation and failure of the syntactic foams at different strain stages of the quasi-static compression.

5.3.3 Dynamic failure process

Figure 5. 6 shows the μ XT observations on the internal failure process of the syntactic foams, which were deformed to different strains in the SHPB tests and then recovered. At the low strain stage ($\varepsilon = 0.1$) of the SHPB compression, the elastic deformation dominates within the foam. However, the fragments of fractured cenospheres can be observed in one side of the foam specimen in the bottom zone of Figure 5. 6 ($\varepsilon = 0.1$). At the increased strains ($\varepsilon = 0.2$ to 0.4) in the plateau region, the two distinct top and bottom zones, as roughly separated by the dashed lines, become more apparent in the foam (Figure 5. 6). Note that the initial failed ends of the specimens are shown in the bottom of the μ XT slices in Figure 5. 6; the failed ends can be either near the input or output bars of the SHPB system. In the bottom zone, the majority of cenospheres are crushed and the macro-cracks evolve in the epoxy matrix. In contrast, most of the cenospheres and the matrix in the top zone remain intact. This implies that the bottom zone deforms plastically whilst the top zone elastically. When the strain increases, the bottom plastic zone enlarges whereas the top elastic zone reduces (Figure 5. 6). At the high strain $\varepsilon = 0.5$, most cenospheres in the entire foam become crushed; the macro-cracks develop throughout the specimen almost in any directions rather than along a preferential path.

Figure 5. 6 The μ XT longitudinal slices of the internal deformation and failure of the syntactic foams at different strain stages of the dynamic compression. (Note: the initial failed end of the foams, which is located at the bottom of each figure, can be near either the input or output bar during the SHPB tests.

5.4 Discussion

5.4.1 Effect of strain rate on failure mechanism

The failure process of cenosphere epoxy syntactic foams subjected to quasi-static and dynamic uniaxial compression is dominated by (1) the crushing of cenospheres and (2) the plastic deformation and cracking (micro- and macro-cracking) of the epoxy matrix. These two internal failure processes can be considered the damage in the bulk syntactic foam. The microscopic mechanism in the failure process (i.e., damage evolution) is affected by the strain rate (compare Figure 5. 5 and Figure 5. 6). At low strain rates, the damage initiates from the central portion of the foam specimen and then evolves diagonally (Figure 5. 5). However, under dynamic compression, the damage originates in one end of the specimen and develops towards the other end (Figure 5. 6). Furthermore, compared to quasi-static compression, macro-cracking of the matrix occurs at a lower strain in the syntactic foam subjected to dynamic loads.

At high strain rates, the increased strength of the strain rate sensitive epoxy matrix suggests that the load and stress transfer between the two constituents in the syntactic foam is different from that in quasi-static experiments. As indicated by the arrows in Figure 5. 6, the macro-cracks can split the cenospheres ahead and propagate through cenospheres in the SHPB experiments of the foams. However, splitting of cenospheres by matrix macro-cracking can hardly be observed in the foam subjected to quasi-static compression, where the macro-cracking in the matrix tends to pass around the cenospheres or debond the cenosphere/matrix interface.

The similar phenomena observed by the post-test SEM examination was reported in the previous work [69, 103]. However, the present study directly characterised the splitting of cenospheres at various strain stages in the SHPB compression.

5.4.2 Quantification of damage evolution in syntactic foams

The damage in the syntactic foam evolves as a function of both the strain and strain rate. An empirical constitutive equation [55] was used to quantify the relation between the damage (D), the strain (ε) and the strain rate ($\dot{\varepsilon}$):

$$D = d \times \dot{\varepsilon}^{(\lambda-1)} \times (\varepsilon - \varepsilon_{th})^n \quad 5.14$$

where d , λ and n are the material constants, and ε_{th} is the threshold strain for damage initiation. The $\varepsilon_{th} = 0.04$ is estimated based on the measured stress–strain curves (Figure 5. 4). The damage scalar D was defined as the ratio of damaged volume to the whole specimen volume. The damaged volume includes the failure of cenospheres, the matrix connecting the collapsed cenospheres and the macro cracks in the specimen, because all these factors weaken the stiffness of the original foam. The damages can be detected from the 2D μ XT slices, e.g. Figure 5. 7 shows the schematic of the intact and damaged area of the specimen at the strain $\varepsilon = 30\%$. Compared the μ XT slices (Figure 5. 5 and Figure 5. 6, strain $\varepsilon = 30\%$), the cenosphere in the marked area were crushed at the quasi static strain rate, whilst the macro crack occurred except the cenosphere collapse at the dynamic strain rate. Note that not all the cenosphere were crushed in the marked area at the dynamic strain rate (Figure 5. 7, dynamic), there were still some unchanged cenosphere

between the macro cracks at the left bottom corner of the specimen (see Figure 5. 6, strain $\varepsilon = 30\%$). This is because the load around the intact cenospheres was released due to the macro cracks propagation. Though there were a few intact cenosphere, the left bottom corner between the macro cracks were almost separated from the specimen, and on longer contributed in the stiffness to the whole specimen. Thus the separated parts from the specimen were also considered as the damaged volume. When all the 2D μ XT slices were identified as the intact and damaged area, the 3D damaged volume and the whole specimen volume can be obtained.

Figure 5. 7 The schematic of the intact and damaged area of the specimen at the strain $\varepsilon = 30\%$.

Figure 5. 8 shows the calculated damage scalar D for the deformed foam specimens at various strains and strain rates. The constants d , λ and n (refer to Table 5.1) were fitted with a good correlation coefficient ($R^2 = 0.9859$). At a given strain (Figure 5. 8), the dynamic damage in the syntactic foam is more severe than the quasi-static damage. Therefore, more energy can be dissipated by the foam under impact condition.

Table 5.1 Parameters in Equation 5.14 to describe the damage as a function of strains and strain rates.

ε_{th}	d	λ	n
0.04	1.805	1.016	0.8947

Figure 5. 8 The damage in the syntactic foam as a function of strains at the quasi-static and dynamic strain rates.

5.5 Summary

X-ray microtomography was conducted to investigate the failure (damage) mechanism in cenosphere epoxy syntactic foams at quasi-static and dynamic strain rates. It was found that the microscopic mechanism in the failure process of the foam is significantly influenced by the strain rate. At high rates, macro-cracks in the matrix can split the cenospheres ahead and propagate through them; however, in quasi-static compression the matrix macro-crack propagates around a cenosphere and does not break it. Moreover, macro-cracking of the matrix occurs in the earlier strain stage of dynamic compression compared to quasi-static loading condition. The damage evolution in the foam can be quantified as a function of strain and strain rate using an empirical constitutive equation.

Chapter 6

Conclusions and future work

The objective of the work is to better understand the mechanical behaviour and the associated failure mechanisms of syntactic foams, and find out the relationship between the micro-structure and the mechanical response of the bulk syntactic foam. To achieve the goal, the compressive tests under different strain rates and the observations of the internal deformation were conducted. The unit cubic cell and full scale FE models were established to simulate the mechanical behaviour of the syntactic foam in its elastic and plateau regions. The main conclusions are drawn first, and then several recommendations are given for the future work.

6.1 Conclusions for the present work

6.1.1 Elastic behavior and failure mechanism in glass bubble epoxy syntactic foams

The glass microballoons filled epoxy syntactic foams were first developed. The elastic behavior and failure mechanism in the epoxy syntactic foams as well as the effect of glass microballoon volume fractions and the radius ratio were investigated. A 3D cubic REV in the foam was generated, in which the

microballoons with a statistic size distribution were randomly distributed. The FE model of the REV foam was then developed to predict the localised stress field in the elastic region during the compression of the foam. It was found:

1) The stress in glass microballoons is significantly higher than that in the matrix. Thus the microballoons are the key constituents to bear the majority of the applied loads. The stress in the matrix concentrates close to the top and bottom of microballoons; it is also considerably localised on the connection areas between microballoons for high microballoon volume fractions. Micro-cracks may originate at the stress concentration areas in the matrix.

2) The stress in a glass microballoon concentrates on the equator. Thus the microballoons fail by the vertical splitting fracture along the compression direction, thus leaving the voids in the matrix after fracture. The voids joining the adjacent micro-cracks in the matrix can develop into a macro-crack.

3) The failure of syntactic foams initiates due to the fracture of microballoons and the formation of micro-cracks in the matrix. The glass microballoon K25 is the weakening phase whereas S60 shows the inverse effect in the foam; the onset of microballoon fracture determines the peak stress. It was thus found that with the increased microballoon volume fraction: a) the Young's modulus, critical strain at peak stress and the peak stress decrease in SFK25 syntactic foam. However b) the Young's modulus increases but the critical strain at peak stress decreases, the peak stress nearly keep constant in SFS60 syntactic foam.

4) Experimental observations reveal that the failure mode of syntactic foams was significantly affected by the microballoon volume fraction, but not sensitive to microballoon type. At low volume fraction, the glass microballoons are widely distributed in the foam. The micro-cracks tend to propagate towards the areas near the top and bottom of microballoons where the stress concentrates in the matrix, and thus forming a longitudinal macro-crack. However, at high volume fraction, the glass microballoons are more closely packed. The micro-cracks propagate in all possible directions in particular the connection between microballoons. They will form a macro-crack in the diagonal direction by joining the adjacent voids.

6.1.2 μ XT and FE modelling of compressive failure mechanism in cenosphere epoxy syntactic foams

The compressive failure mechanism of cenosphere epoxy syntactic foams was investigated using full scale FE modelling and x-ray microtomography coupled with interrupted compression. The predicted localised stress, fracture of cenospheres and deformation in the matrix were related to the μ XT observations to further analyse the underlying failure mechanism especially in the plateau region of the foam. The conclusions were drawn as follows.

1) The internal compressive failure process of the cenosphere epoxy syntactic foams in microscopic scale is characterised by two simultaneous phenomena: a) the crushing of cenospheres and b) the plastic deformation and

fracture of the matrix. These two phenomena are driven and determined by the localised stress state in the constituents.

2) The failure mechanisms in the two constituents dominate the different strain stages of the bulk stress–strain behavior of the syntactic foam. At the lower strain of the plateau region, the crushing of large cenospheres is prevalent in the centre of the foam specimen. In addition to the cenosphere collapse, the plastic deformation and fracture process in the matrix dominates the higher strain stage.

3) The tensile stress concentration near the annular equatorial area causes the cenosphere to fracture along the external compressive loading direction and then split into fragments. After the fracture of cenospheres, the stress concentration is transferred to the neighbouring cenospheres and the matrix material.

6.1.3 μ XT characterization of strain rate dependent compressive behavior and failure mechanism in cenosphere epoxy syntactic foams

The strain rates effect on the compressive behavior and failure mechanism of cenosphere epoxy syntactic foams were finally investigated. The conclusions were drawn as follows.

1) The developed syntactic foam shows clear stain rate dependency. At dynamic strain rate, E , σ_{pk} and σ_{pl} are 2 or 3 times higher than those at the quasi-static strain rate. But ε_{Cr} doesn't show the rate dependency.

2) The microscopic mechanism in the failure process of the foam is also significantly influenced by the strain rate. At high rates, macro-cracks in the matrix can split the cenospheres ahead and propagate through them; however, in quasi-static compression the matrix macro-crack propagates around a cenosphere and does not break it. Moreover, macro-cracking of the matrix occurs in the earlier strain stage of dynamic compression compared to quasi-static loading condition.

3) The damage evolution in the foam can be quantified as a function of strain and strain rate using an empirical constitutive equation.

6.2 Recommendation for the future work

6.2.1 Optimization of the finite element modeling

Though the reasonable REV and full scale FE models of syntactic foam have been established, the models still can be improved in the following three aspects:

1) Some constitutive parameters of the micro spheres used in the models, such as the young's modulus and the strength, were obtained from the reported literatures. In fact, the constitutive properties should be measured from the experiments directly. Nano-indentation with the flat indenter may be an applicable method to obtain such parameters.

2) From the μ XT slices discussed in Chapter 4 and 5, the macro matrix cracks can be clearly observed at the late deformation stage under both quasi static

and dynamic strain rate. However, the matrix cracks haven't been considered in the models until now. As the epoxy used in this study shows brittle when it is subjected to tensile loading, the brittle cracking may be a good description for the failure of epoxy resin.

3) The mechanical behaviour of syntactic foam under dynamic strain rate has not been predicted. Due to the viscoelasticity of epoxy matrix, it is necessary to find a suitable constructive description for epoxy under high strain rate.

To make the models closer to the real syntactic foams, the three improvements should be facilitated.

6.2.2 High velocity impact tests on the syntactic foams

As the increasing usage of syntactic foam in automobile and aerospace transportation applications, future work requires the comprehensive studies on evaluating the mechanical response of syntactic foams under the high strain rate. Though the performance and associated mechanisms of syntactic foam under quasi-static or low velocity impact (e.g., $< 30\text{m/s}$) have been investigated in this study and also investigated by a number of other researchers, the impact behavior and related energy absorption capability under high velocity loading (e.g., $> 100\text{m/s}$) are still unknown. In fact, the high velocity loading is the condition that syntactic foams need to face when they are applied in transportation vehicles as the structural components. Therefore the high velocity impact tests on syntactic foams or the sandwich structure with syntactic foam cores should be conducted. The

experiments can be designed based on the high velocity impact Hopkinson pressure bar techniques, Taylor impact tests or ballistic impact experiments. Moreover, the large scale syntactic foam plate specimens should also be produced to fit the experiments.

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