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Investigation on Tensile Properties of Plain and Nanoclay Reinforced Syntactic Foams

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Abstract Tensile properties of plain and nanoclay reinforced epoxy matrix syntactic foams with three different sizes of ceramic microballoons are investigated experimentally. Nine series of plain syntactic foams with 20, 40 and 60 volume fractions of microballoons are prepared and tested to study the volume fraction and size effects. Also nano syntactic foams specimens with six different weight fractions of nanoclay (0, 1, 2, 3, 5 & 7%) are tested and the effect of nanoclay content on the tensile properties is investigated. In addition to tensile tests, fracture modes of all syntactic foams are considered thoroughly by using scanning electron microscopy (SEM).

Keywords Syntactic foam, Ceramic microballoon, Nanoclay, Tensile properties, Experimental analysis

1. Introduction

Application of lightweight materials is widely increased in aeronautical, marine and civil structures. Among these materials, foams with a significant weight saving have an important role, but their applications are limited by their low strength and modulus. So, developing some methods for introducing porosity in materials without causing serious effects on mechanical properties is the topic of many researches during recent years. Syntactic foam is a kind of porous material with walled hollow particles (microballoon) in a matrix material [1]. Desired properties for this material can be obtained by choosing appropriate material for matrix and microballoons. Metal, polymer or ceramic can be selected for matrix and microballoons can be made of ceramic, glass, carbon or polymer. A wide range of possible volume percentages of microballoons gives opportunity to tune the desired properties [2]. Most of the studies performed have been on the mechanical and fracture properties of syntactic foams under compression [3-6], flexural [7-9] or thermal degradation [10, 11]. However few published works have been reported on tensile properties of syntactic foams. Gupta and Nagorny [12] investigated the effect of hollow glass microballoons volume fraction on the tensile properties and fracture modes of syntactic foams. They observed that the tensile strength decreases with an increase in the volume fraction of microballoons. The similar results have been reported by Wouterson et al. [13]. Gupta et al. [14] focused on the synthesis and characterization of vinyl ester/glass microballoon syntactic foams. They found that specific tensile strength and specific tensile modulus of foams are comparable with their neat resin and are higher for some samples. Gouhe and Demei [15] tested four types of hollow polymer particles as filler in UV-heat-cured epoxy resin. The tests show that tensile strength and specific properties of all foams decrease with increasing the particle content. Also the results showed that the mechanical properties of syntactic foams specially depend on microballoons' material. Among the published papers ceramic microballoons are considered in few cases. Ceramic microballoons are being developed for structural applications or electric appliances like piezoelectric transducers and low dielectric constant substrates [16, 17]. Mechanical properties are obviously the primary concern for ceramic microballoon filled syntactic foams. Interest for exploiting the benefit of low density of syntactic foams has made it necessary to characterize these materials for tensile loading and study various parameters affecting their properties.

It has been studied that use of nanoclay in the structure of neat polymers such as epoxy, increases tensile strength, tensile modulus and impact resistance [18-20]. Most of the published papers indicate that large number of interfaces that are created in a nanocomposite upon dispersion of nano particles results in increasing of strength of the composite matrix [21, 22]. Although, it is shown that nano-reinforcing of the matrix material of syntactic foams enhanced the mechanical properties of glass microballoon filled syntactic foams [23-27], the reports are few and the full potential of nano-reinforcing of the syntactic foams with different microballoons is not explored yet.

In this paper, the tensile properties of nine types of plain syntactic foams (three microballoon sizes and three

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microballoon volume fractions) and neat epoxy are obtained experimentally to study the effects of volume fraction and microballoon size and material. Also, nano syntactic foams of 40% volume fraction of microballoons of only one type is prepared with six weight fraction of nanoclay (0, 1, 2, 3, 5 and 7 wt.%) in epoxy matrix. They are tested to study the effects of the presence of nanoparticles on the tensile properties.

The fabricating method was evaluated by measuring density and porosity of fabricated foams. Also, extensive scanning electron microscopic observations are performed to establish the modes of failure and further to understand the mechanical properties.

2. Experimental Procedure

2.1. Materials

The ceramic microballoons of syntactic foams are from OMEGA MINERALS Germany GmbH with the trade names of SG, WM and W150. Some typical properties of these microballoons are shown in Table 1. Epoxy resin, with the trade name of EPIKOTE 828 from Resolution Performance Products LLC and TETA hardener from Akzo Nobel Functional Chemicals are used for matrix material. EPIKOTE 828 is a medium viscous epoxy resin based on Bisphenol-A. TETA hardener is a low viscous aliphatic amine used for room temperature curing. The resin to hardener ratio specified by the supplier was 10:1. Density of the pure cured epoxy is measured to 1.18 g/cm^3 according to ASTM C271 standard [28]. The nanoclay used is a natural montmorillonite modified with a quaternary ammonium salt that has the trade name Closite 30B, which is manufactured by Southern Clay Products, Inc. Its moisture is less than 2% and its density is about 1.98 g/cm^3.

Table 1. Properties of ceramic microballoons

Microballoon type	Outer diameter (μm)	Thickness to radius ratio (%)	True density (g/cm^3)
WM	170	10	0.7
SG	130	10	0.7
W150	80	10	0.7

2.2. Fabrication

Plain syntactic foams were fabricated by hand stirring mixing method. First, microballoons were added to resin gently and stirred with a wooden rod until a uniform distribution is ensured. Then the hardener was added to the mixture. For decreasing the undesirable phenomena effects, for 20 and 40 vol. % syntactic foams, final mixing last until the blend becomes a viscous paste. So, the floatation of the microballoons did not happen. On the other hand, for alleviating the matrix porosity of 60 vol. % syntactic foams, the uncured blend was degassed before moulding [5, 27, 29].

Nano syntactic foam samples consist of 40 vol.% of W150 microballoons. Five series of specimens with different nanoclay weight fraction in their matrix (1, 2, 3, 5, 7 wt.%) were prepared. For fabricating, the epoxy resin was preheated at 75˚C for approximately 24 hr in an oven to reduce the viscosity for better wetting of particles. Resin was then removed from the oven and nanoclay was incorporated in the required amount and the mixture was stirred by a mechanical shear mixture at 500 RPM for 30 minute. As the next step, the mixture was ultra-sonicated for 15 minutes with the cycle of 0.5 and the amplitude of 60% [30]. During this process, nanoclay clusters break into nanoclay platelets and the resin can penetrate into the nanoclay galleries. Sonication caused temperature increasing and a large number of micro-bubbles. So, degassing of the mixture was important. After cooling the mixture to room temperature, the microballoons were added and the mixture was hand stirred until a uniform distribution was insured. The hardener was added to the mixture as the last step.

Finally the blend, both plain and nano syntactic foam, was transferred to a stainless steel mould which was smeared with mould releasing agent. The mixture was cured for 48h at room temperature. The test specimens were cut from cured slabs by water-jet cutting machine.

2.3. Specimen Coding

Preventing from ambiguity, the following nomenclature for specimens has been used as: SF-XX-YY. SF is the abbreviation of syntactic foam. The name of microballoon is used instead of XX and volume percentage of microballoon in epoxy is used for YY. For example SF-SG-40 is the syntactic foam with 40 volume percentage of SG microballoons.

For nano syntactic forms the nomenclature of NSF-XX-Y has been used. Since the microballoon volume percentage of all the nano syntactic foam samples are 40%, the nomenclature has not included this value. XX show the type of microballoon and Y represents the nanoclay weight fraction of the samples. For example: NSF-W150-5 is a nanoclay syntactic foam with 40% volume fraction of microballoon and 5% weight of nanoclay in epoxy.

2.4. Test Procedure

ASTM standard C271 was applied to measure the density of all fabricated specimens. The weights and volumes of at least five pieces of 25×25×13 mm^3 size from each type were measured to calculate the foam density.

The tensile samples were cut according to ASTM standard D638 [31] into dog-bone test specimens. The test was carried out using Instron 5500 testing machine system (Figure 1) and at cross head speed of 1 mm/min. At least five specimens of each type of syntactic foams were tested. Strain data was collected by an extensometer with 25 mm gauge length.

It should be noted that because of the matrix porosity, some specimens failed prematurely. So, fractured surfaces of all specimens are examined for the presence of matrix porosity, and the test results of those had porosity on their surfaces were discarded.

Figure 1. Dog-bone test specimen and tensile testing machine

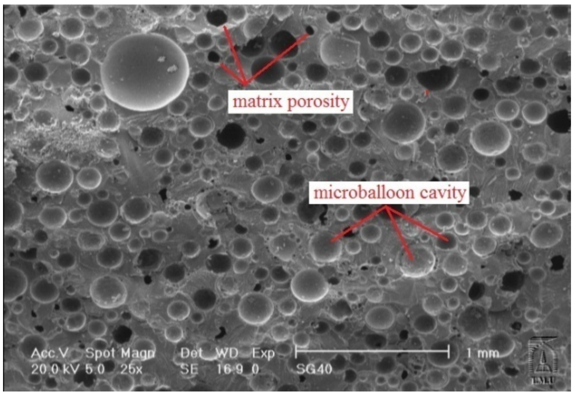

Figure 2. Matrix porosity in syntactic foam

The second type of porosity is the entrapment of air because of the mechanical mixing of the constituents (Figure 2). In all cases this type of porosity reduces the strength and modulus of the foam [7, 11, 12, 29]. Also if these cavities do not distributed well, the imperfections and strength reductions become considerable. However, it cannot be avoided in actual experiments; it is desired to maintain this type of porosity at a minimum level. As an estimate, matrix porosity can be derived by accounting for the difference between the theoretical density (ρ_{th}) and the actual density (ρ_{ac}):

$$\varphi_m = 1 - \rho_{ac} / \rho_{th} \tag{2}$$

The results are shown in Table 2. This shows that with increasing the volume fraction, the unwanted porosity was increased, too. But they were not proportional to each other. The calculated values for 20 % and 40 % foams are around 2 %. But for 60 % volume fraction the undesired porosity has increased to about 10%. Actually, the mechanical stirring has caused air entrapment in blend and this event has become more apparent with increasing viscosity, as it is seen for 60 vol. % syntactic foams. The results of the Table 2 show that the size of microballoons does not affect the matrix porosity content. Like previous studies the undesired porosity content is in the range of 2-11% in syntactic foams [12, 14].

As mentioned before, increasing the viscosity of the uncured blend causes air entrapment during mixing. In nano syntactic foams as the nanoclay content increased, the viscosity increased of the blend increased, too. So, the matrix porosity increased.

Table 2. Structural properties of neat epoxy, syntactic foams and nano syntactic foams

Sample Code	Measured Density (g/cm^3)	Theoretical Density (g/cm^3)	Matrix Porosity
Epoxy	1.18	--	--
SF-SG-20	1.068	1.088	1.8
SF-SG-40	0.969	0.991	2.2
SF-SG-60	0.795	0.894	11.1
SF-WM-20	1.062	1.088	2.4
SF-WM-40	0.962	0.991	2.9
SF-WM-60	0.806	0.894	9.8
SF-W150-20	1.046	1.088	3.8
SF-W150-40	0.954	0.991	3.7
SF-W150-60	0.810	0.894	9.4
NSF-W150-1	0.975	0.994	1.9
NSF-W150-2	0.974	0.997	2.3
NSF-W150-3	0.972	1.001	2.9
NSF-W150-5	0.970	1.006	3.6
NSF-W150-7	0.958	1.010	5.1

3.2. Effect of Volume Fraction

The results of tensile strength, elastic modulus, fracture strain, specific strength and specific modulus are shown in Table 3. The results state that with increasing the microballoon content of the foams, the strength of them decreased. It means that the strength of syntactic foam strongly depended on the content of matrix as the main constituent of load bearing. But it did not have a linear relation between increase of strength and volume fraction as shown in Figures 3 & 4. By adding 20% microballoons to epoxy resin the strength decreased about 33-35%. Actually adding microballoons created some imperfections such as unwanted porosity and weak interfacial surfaces between microballoons and matrix. These facilitated the crack propagation inside the foam. The SEM figures of the fracture surfaces of tested specimens validated this claim. Reviewing the fracture surfaces, both damage mechanisms of microballoon fracture and propagation of crack along the weak interface of microballoons and matrix can be seen (Figure 5). It was expected that in well prepared syntactic foams, bonds between microballoons and matrix remained intact and this will force the crack to propagate along the matrix or fracture the microballoons. In SEM figures of this work, debonding between microballoons and matrix was rarely observed and it can be deduced that the interfacial bonding is strong.

The extent of decrease in strength from neat resin to SF-SG-20 is not the same as for SF-SG-20 and SF-SG-40. In the former case the reduction is about 34 % but in the latter case the strength decreased about 20% as shown in Table 3.

Table 3. Tensile strength, fracture strain, elastic modulus, specific strength and specific modulus of syntactic foams and neat epoxy

Sample Name	Tensile Strength (MPa)	Fracture Strain (%)	Elastic Modulus (MPa)	Specific Strength (MPa.cm^3/g)	Specific Modulus (MPa.cm^3/g)
Neat Epoxy	36.8 ± 0.9	1.85 ± 0.05	2.30	31.1	1.94
SF-SG-20	24.2 ± 0.7	1.02 ± 0.07	3.55	22.7	3.32
SF-SG-40	19.4 ± 0.9	0.56 ± 0.02	4.01	20.0	4.14
SF-SG-60	11.2 ± 1.2	0.26 ± 0.04	4.31	14.1	5.42
SF-WM-20	23.9 ± 0.8	1.03 ± 0.05	3.49	22.5	3.29
SF-WM-40	19.0 ± 0.6	0.59 ± 0.03	3.94	19.8	4.10
SF-WM-60	9.5 ± 1.1	0.27 ± 0.05	4.17	11.8	5.17
SF-W150-20	24.8 ± 0.5	1.00 ± 0.02	3.63	23.7	3.47
SF-W150-40	20.2 ± 0.8	0.60 ± 0.05	4.07	21.1	4.27
SF-W150-60	13.6 ± 1.0	0.30 ± 0.04	4.49	16.8	5.54

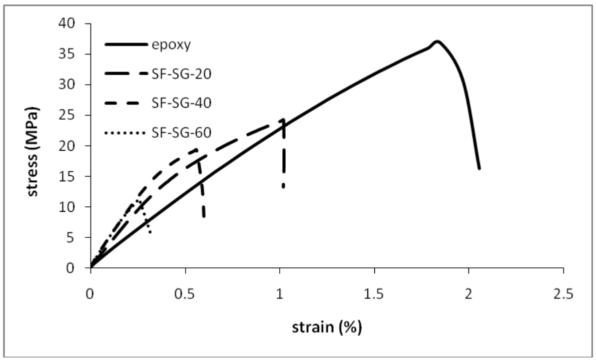

Figure 3. Stress – Strain curves for neat epoxy and SG microballoons syntactic foams with different volume fractions

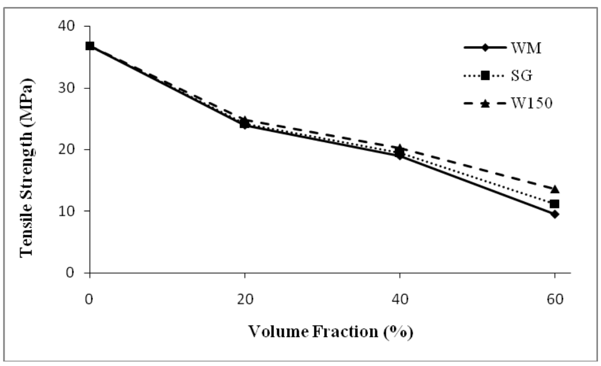

Figure 4. Effect of microballoon volume fraction on the tensile strength of syntactic foams

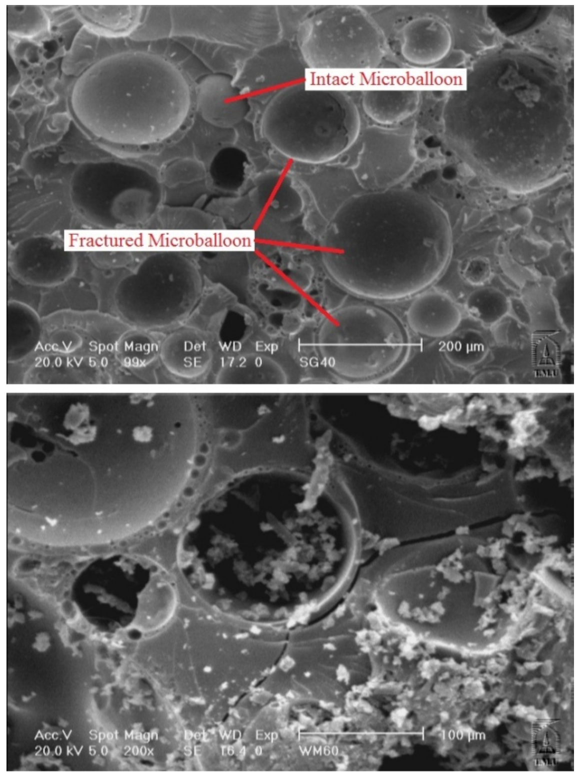

Figure 5. Crack paths through microballoon, matrix and interfacial surfaces between them in SEM figures from fractured surfaces

Although the weaker interfacial surfaces increased in syntactic foams of high microballoon volume fraction but there is also the possibility of microballoon fracture. These caused that the strength of high microballoon volume fraction syntactic foams reduced further. The strength of 60 volume fraction syntactic foams were about 32-50% of the strength of 40 volume fraction ones. In this case the matrix porosity was very high and this is the main reason for this low strength.

Changes of the fracture strains were the same as the tensile strength. So, as the microballoon volume fraction increases, the fracture strain decreases.

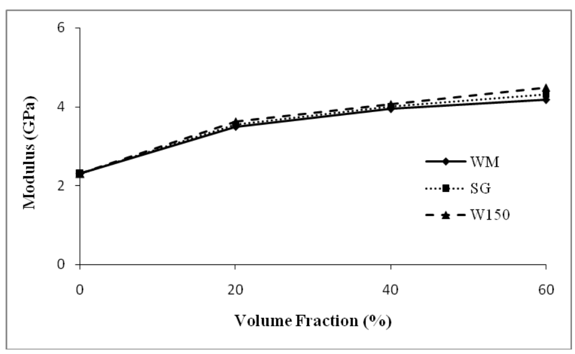

Figure 6. Effect of microballoon volume fraction on the tensile modulus of syntactic foams

On the contrary of the previous properties, the modulus increased significantly as the microballoon content became more, as shown in Figure 6. Actually, adding stiff ceramic microballoons to epoxy caused that the syntactic foam's deformation decreased.

By comparing the results of this work with the results of other investigators that use glass microballoons with epoxy matrix [12], it is found that the trend of changes in strength is similar but it is different for modulus. Gupta and Nagorny [12] tested some types of glass microballoon/epoxy matrix syntactic foams and conclude that the modulus of syntactic foam decreased while increasing the volume fraction of microballoons. This shows that the main effect of the microballoon material would be on the modulus of the syntactic foam. The main advantage of using syntactic foam in structures is its enhanced modulus, so the material of the syntactic foam should be chosen properly.

Also, it is reported that the matrix-microballoon interfacial bonding was not very strong and microballoon fracture was rarely occurred [12, 13]. So, many glass microballoons remain intact but this was not the case in the ceramic microballoon/epoxy syntactic foam. The observation shows that the most of microballoons in the specimens were fractured and few of them remain intact on the fractured surface. This showed that the bonding between epoxy matrix and ceramic microballoons is better than glass microballoons.

In most applications of syntactic foams, weight reduction is an important factor. For evaluating weight sensitivity, specific strength and specific modulus of tested specimens versus microballoons volume fraction are plotted in Figure 7. In fact the lower the difference between the specific strength of foam and its neat matrix, the foam would perform better.

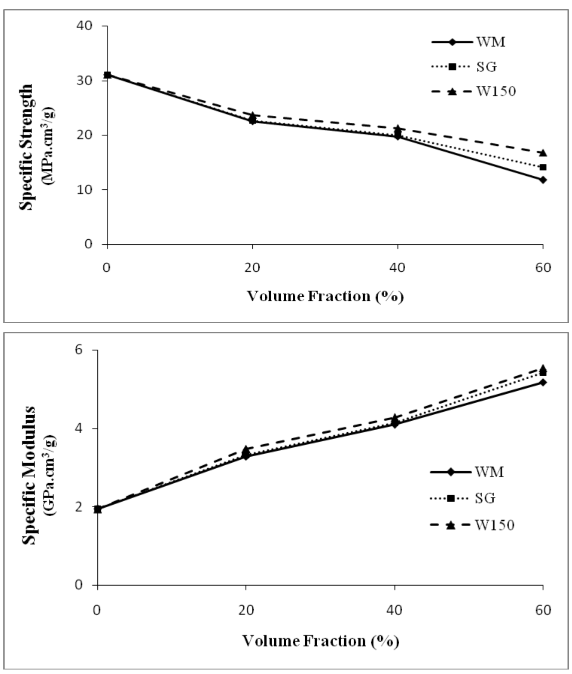

Figure 7. Effect of microballoon volume fraction on specific strength and specific modulus

One of the applications of syntactic foams is as a core material in the sandwich composites for structural applications [8]. The weight of components can be minimized if E/ρ, E/ρ^2 and E/ρ^3 are increased for the same axial stiffness of a beam, bending stiffness of a beam and bending stiffness of a plate, respectively. The results shown in Figure 8 indicate that using syntactic foams in structural application, result in substantial weight saving.

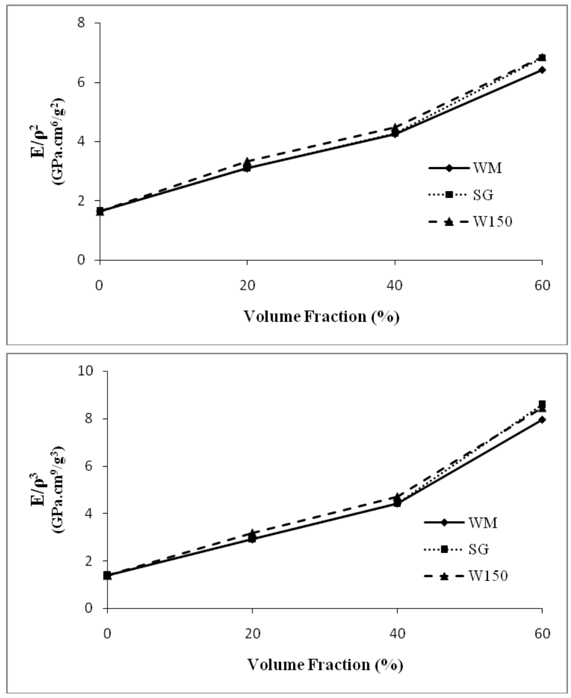

Figure 8. Effect of microballoon volume fraction on E/ρ^2 and E/ρ^3 of syntactic foams

3.3. Effect of Microballoon Size

The results of tensile tests for nine types of syntactic foams with the different microballoon volume fractions and different microballoon sizes are presented in Table 3 and Figures 9 & 10. The results show that the microballoons' size did not affect the fracture strain significantly. Of course its effect on the tensile modulus was about 4-7 %. Actually syntactic foams with smaller microballoons had higher modulus and as the microballoon content increased, the difference became more.

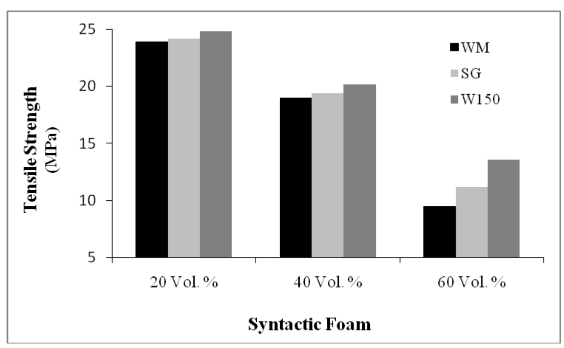

Figure 9. Comparison of the tensile strength of syntactic foams with different microballoon size

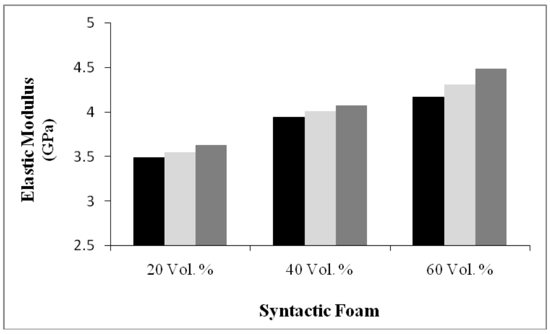

Figure 10. Comparison of the elastic modulus of syntactic foams with different microballoon size

The main changes of varying the microballoon size were observed in the tensile strengths. The strength of 20 vol. % syntactic foams increased about 4% by reducing the microballoon size to half. In 60 % volume fraction syntactic foams, the difference became much more about 30%.

In tensile loading, the crack in foam may propagate through matrix, interface of microballoon and matrix and also through the wall of microballoons. So the crack path is mainly depended on the strength of matrix material, microballoon material and interface bonding between microballoon and matrix. The SEM figures from fractured surface of tested syntactic foams show that many microballoons are fractured thoroughly and few of them remain intact. This shows that bonding between microballoon and matrix has a noticeable strength. In syntactic foams with the same volume fraction, but for smaller size microballoons, the number of fractured microballoons increases. This is partly due to increase in the interfacial surfaces and also the wall cross sectional of microballoons. Since the bonding is strong enough, the crack would propagate through the wall of microballoon and in this case the part of the load bearing of microballoons becomes higher. This phenomenon coupled with higher level of porosity for foam with bigger microballoons (i.e. SF-W150-60) caused SF-WM-60 foam having smaller microballoons to be relatively stronger.

3.4. Effect of Nanoclay Weight Fraction

As mentioned before, presence of limited amount of nanoclay in epoxy would enhance its mechanical properties. In this study, the effect of nanoclay in epoxy matrix of syntactic foam has been investigated. Six types of nano syntactic foams were prepared with W150 microballoons and 40 % volume fraction. Nanoclay weight fraction of epoxy matrix of these syntactic foams were 0%, 1%, 2%, 3%, 5% and 7%.

The stress - strain curves are illustrated in Figure 11. Also, the comparative results for tensile strength and modulus are in Figures 12 & 13. Generally, presence of nanoclay in epoxy matrix, made nano syntactic foams more brittle. As the nanoclay content increased, the fracture strain decreased and the tensile modulus increased. The tensile modulus of NSF-W150-5 was the most and nearly twice the tensile modulus of NSF-W150-0.

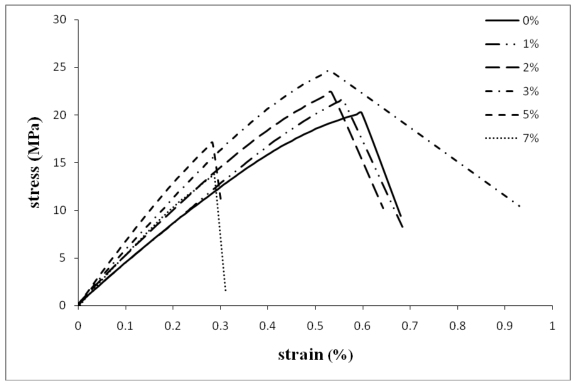

Figure 11. Stress – Strain curves for nano syntactic foams with 40% W150 microballoon volume fraction and different nanoclay content

The results show that the tensile strength was affected, too. It has been increased for nano syntactic foams of 1%, 2% and 3% weight fraction. But with more increase in nanoclay content, the tensile strength decreased. Overall, the improvement in tensile strength is about 22% with adding 3% weight fraction nanoclay to matrix.

In a well dispersed of nanoclay platelets into epoxy matrix syntactic foam, the good adherence of nano particles to matrix created a large number of strong interfaces. As these interfaces increased, the strength of nano syntactic foams increased, too. Actually the nano platelets are stronger and stiffer than epoxy material. So, when they intercalated in matrix material, load bearing capacity of the whole increased. On the other hand, with increasing the nanoclay content more than 3% weight fraction, the strength decreased. It indicated that the agglomeration of nanoclay clusters is occurred. In this situation, the modulus of nano syntactic foams increased because of stiff behavior of nanoclay platelets, but agglomerated clusters made locations with stress concentrations. So, the cracks would propagate easier from these locations and failure would happen in lower levels of stress.

Table 4. Tensile strength, fracture strain, elastic modulus, specific strength and specific modulus of nano syntactic foams

Sample Name	Tensile Strength (MPa)	Fracture Strain (%)	Elastic Modulus (MPa)	Specific Strength (MPa.cm^3/g)	Specific Modulus (MPa.cm^3/g)
SF-W150-0	20.2 ± 0.8	0.6 ± 0.05	4.07	21.1	4.27
NSF-W150-1	21.6 ± 0.9	0.56 ± 0.02	4.21	22.2	4.32
NSF-W150-2	22.4 ± 0.8	0.53 ± 0.04	4.55	23.0	4.67
NSF-W150-3	24.6 ± 0.5	0.53 ± 0.02	5.43	25.3	5.59
NSF-W150-5	17.1 ± 0.8	0.28 ± 0.05	6.05	17.6	6.24
NSF-W150-7	13.7 ± 0.9	0.29 ± 0.04	4.83	14.3	5.04

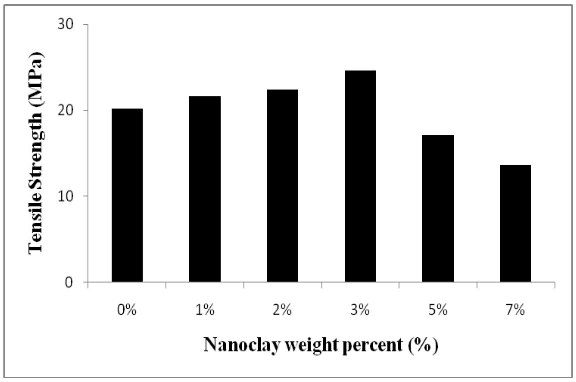

Figure 12. Effect of nanoclay weight fraction on the tensile strength of syntactic foams with 40% W150 microballoon volume fraction

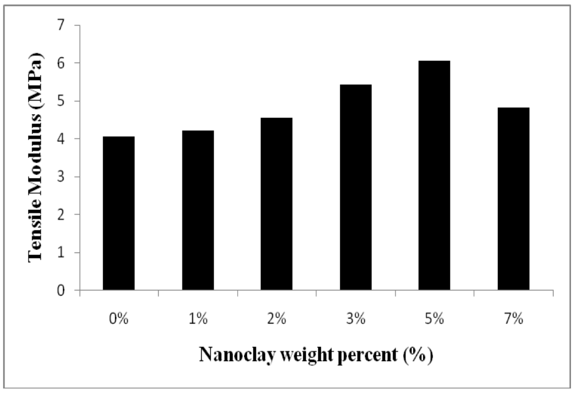

Figure 13. Effect of nanoclay weight fraction on the elastic modulus of syntactic foams with 40% W150 microballoon volume fraction

4. Conclusions

Tensile properties of ceramic microballoon/epoxy matrix syntactic foams with different volume fraction and microballoons' size were investigated in this work. The fracture strain and tensile strength of syntactic foams are significantly influenced by the microballoon content. It is found that both of them decreased as the volume fraction increased. Contrary to glass microballoons, introducing the ceramic microballoons in epoxy enhanced the tensile modulus as the volume fraction increased.

SEM images showed that the cracks propagated along the matrix and microballoon materials. Interfacial bonding of microballoon surface and matrix was strong enough that debonding rarely occurred.

Microballoons' size effect on the mechanical behavior is also investigated and results show that syntactic foam with bigger microballoons has less strength than syntactic foam with smaller microballoons. Also, it should be indicated that the fracture strain and the tensile modulus did not change significantly.

Tensile properties of nanoclay reinforced syntactic foams were studied, too. Adding nanoclay to the matrix of syntactic foams resulted in increasing the tensile modulus and decreasing the fracture strain. Increasing nanoclay content up to 3% enhanced the tensile strength but more nanoclay had a reverse effect and decreased it.

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