

Thermal and Mechanical Characterization of Expancel nanocomposite

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Abstract - A novel sonochemical method is developed to infuse nanoparticles (SiC, SiO₂) into expandable thermoplastic micro-spheres (Expancel) containing acrylonitrile and methylacrylonitrile polymer. The dry composite powder is foamed in a rectangular stainless steel mold by heating to ~190°C at a heating rate of 10°C for 30 min using a MTP-14 programmable compression molding under a pressure of ~ 3000lbs. The test coupons were cut precisely from the panels to carry out thermal, morphological and mechanical characterizations. The as-prepared nanophased foam samples were characterized by scanning electron microscopy (SEM), thermo gravimetric analysis (TGA) and differential scanning calorimetry (DSC). Compression tests have been carried out for both nanophased and neat foams systems. Test results show a significant increase in compressive strength and modulus of the nanophased foams over the neat system. This enhancement in compressive properties has been observed repeatedly for multiple batches. Details of the synthesis procedure, thermal and mechanical characterization are presented in this paper.

Key words: Expancel, Sonochemical, Nanocomposite foam

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INTRODUCTION

Polymeric foam materials are widely used in many industrial applications for their properties of light weight, excellent strength to weight ratio, superior insulating abilities, energy absorbing properties, low thermal conductivity, high sound absorption, large compressive strains. The main applications include sandwich structures, airframes, transportation vehicles, boat hulls, radar systems, and space structures [1-4]. High-performance structural foam materials are fabricated using a blowing agent (surfactants, hydrocarbons) in liquid polymers to expand and form rigid, low-density foams. Some of the leading thermoplastic foams made in this way are polymethacrylimide (PMI) and partly cross-linked polyvinyl chloride (PVC), with trade names Rohacell [2], Divinycell [1] and Expancel [5]. The hollow thermoplastic microspheres produced by Expancel, Inc., under the trade name Expancel® these microspheres are small, spherical plastic particles

consisting of a polymer shell encapsulating a hydrocarbon gas. When the gas inside the shell is heated, it increases in pressure and the thermoplastic shell softens, resulting in a dramatic increase in the volume of the microspheres. Researcher used these microspheres for various applications such as car protection (corrosion resistance, acoustic insulation, gap fillers, underbody coatings) [6], Young-wook and his coworkers developed a closed-cell silicon oxycarbide foams with cell densities greater than 10⁹ cells/cm³ and cells smaller than 30 μm were obtained from a preceramic polymer using expandable microspheres [7]. Lev et al studied the reinforcement of microspheres in PVC along with the aramid fibers and reported the improved mechanical properties [8].

Recently researchers have shown interests in improving polymeric materials physical, mechanical, thermal and chemical properties using nanoparticles as filler materials. Nanoparticles embedded in polymer matrix have attracted increasing interest because of the unique properties displayed by nanoparticles. Due to nanometer size of these particles, their physicochemical characteristics differ significantly from those of molecular and bulk materials [9, 10]. Nanoparticle-polymer nanocomposites synergistically combine the properties of both the host polymer matrix and the discrete nanoparticles there in. Such nanocomposite materials are expected to have novel thermal and mechanical properties [11, 12]. In the present research we have chosen SiC as a filler material because of its wide range of industrial applications and superior mechanical, chemical, electronic and thermal properties [13-16]. We study the morphological and mechanical properties of thermoplastic polymeric foam materials using SiC as fillers.

EXPERIMENTAL

Expancel-092-DU-120 is unexpanded thermoplastic polymer (particles sizes 28-38 μm) was received from Expancel Inc, Silicon Carbide (SiC ~30nm diameter) was purchased from MTI Corporation, and Silicon dioxide (SiO₂ ~15nm diameter) was purchased from Nanostructured & Amorphous Materials, Inc. Expancel polymeric powder and the known percentage of SiC or SiO₂ was dispersed in n-hexane using a high intensity ultrasonic horn (Ti-horn, 20 kHz, 100 W/cm²) at room temperature for 1 hour. The mixture was then dried in a vacuum for 12hs and remaining n-hexane was removed by heating the sample to 60°C for 1 hour. The Dry polymeric-SiC

or polymer-SiO₂ mixture was transferred to a rectangular aluminum mold (4''X 4''X1/2''). The mold is heated to ~190°C at a heating rate of 10°C/min for 30 min using a MTP-14 programmable compression molding under a pressure of ~3000lbs. The samples were cut precisely and used for the morphological and mechanical testing.

Thermo gravimetric analysis (TGA) of various specimens was carried out under nitrogen gas atmosphere on a Mettler Toledo TGA/SDTA 851° apparatus. The samples were cut into small pieces 10-20mg using a surgical blade. The TGA measurements were carried out for these samples from 30°C to 800° C at a heating rate of 10° C/min under nitrogen atmosphere. Differential scanning calorimetry (DSC) experiments were carried out using a Mettler Toledo DSC 822° from 30°C to 400°C at a heating rate of 10°C/min under nitrogen atmosphere. The morphological analysis was carried out using JEOL JSM 5800 Scanning electron microscopy (SEM). The sample were precisely cut in to small pieces and placed on a double sided carbon tape and coated with gold/palladium to prevent charge buildup by the electron absorption by the specimen.

In order to investigate the compression response, the specimens were tested in the thickness direction using Zwick/Roell Material Testing Machine. An ASTM C365-57 was followed for this compression test and the size of the specimen is 12.7 mm x 25 mm x 25 mm respectively. The capacity of the Zwick/Roell machine is approximately 2.5 k N. The test is carried out in displacement control mode and the cross-head speed was 1.27 mm/min. In order to maintain evenly distributed compressive loading, each specimen was sanded and polished with high accuracy so that the opposite faces were parallel to one another. A software TestXpert was used to develop a program which controls the test conditions and records both the load and cross-head displacement data. The load-deflection data recorded by the data acquisition system was converted to stress-strain curve after dividing load by specimen cross-sectional area and deflection by specimen thickness.

RESULTS AND DISCUSSION

Thermo gravimetric analysis (TGA) measurements were also carried out to obtain information on the thermal stability of the nanophased expancel foam. The TGA results of foam materials including neat and the nanophased expancel foam are presented in Table 1. TGA results clearly show that the foam disintegrates in three major steps: first step is corresponds to the loss of organic impurities and the second major weight loss is corresponds to the rupture of the microspheres and finally the third weight loss is corresponds to the decomposition of the polymer it self. The residual carbon content was estimated as ~40% for all the sample including neat and nanophased expancel foam. There was no significant improvement was observed in thermal properties.

Table 1: TGA results of neat and nanophased expancel foam

Sample	First	Second	Third	Total residue
Neat Expancel	0.6 % / 209	34% / 296	23% / 421	41%
1%-SiC-Expancel	1.2% / 196	33% / 300	21% / 434	45%
1%-SiO ₂ -Expancel	1.2% / 197	30% / 297	23% / 467	45%

The glass transition temperature (*T_g*) of the expancel was obtained from the DSC curves, and the scans were carried out at a heating rate of 10°C/min in a nitrogen atmosphere. The *T_g* measured for neat is ~ 110°C. These results are consistent with the manufactures data sheet. The nanophased foam samples show an increase of ~5°C in *T_g*.

COMPRESSION TEST

To understand the effect of SiC and SiO₂ infusion in polymeric foam the compression tests were carried for neat and nanophased foams. Two types of nanophased (1%

SiC and 1% SiO₂ by wt) samples and the neat samples were tested. Stress-strain curves for neat and nanophased samples are shown in Fig. 2. compression data is presented in Table 2.

Table 2: Compression properties of various samples.

Material	Compressive Strength [KPa]	Gain/Loss [%]	Compression Modulus [MPa]	Gain/Loss [%]
Neat Foam	1200±50	-	30±3	-
1%SiC Nanophased	2230±40	+78	34.1±4.5	+20
1%SiO ₂ Nanophased	1418±45	+14	28.6±5.5	-1.4

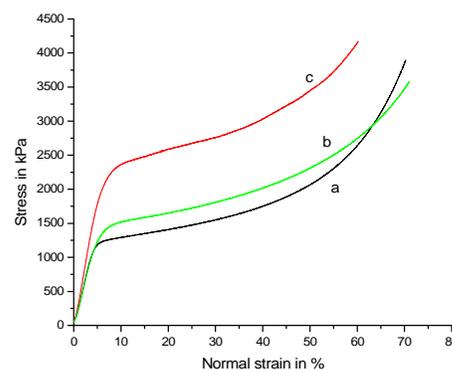
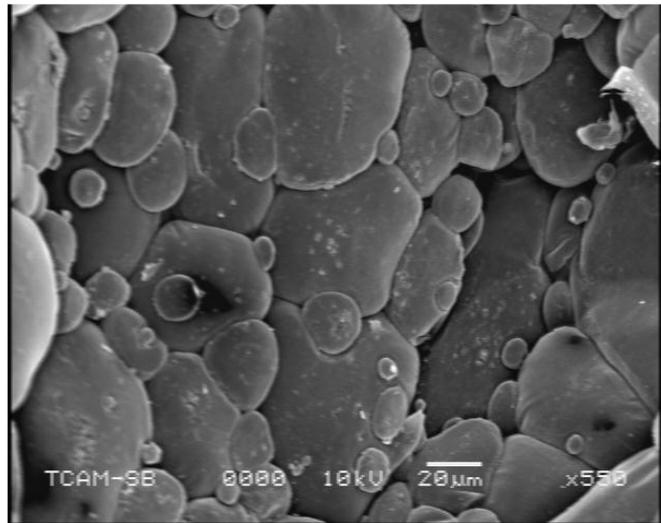


Figure 1: Compressive stress-strain curves are a) neat,

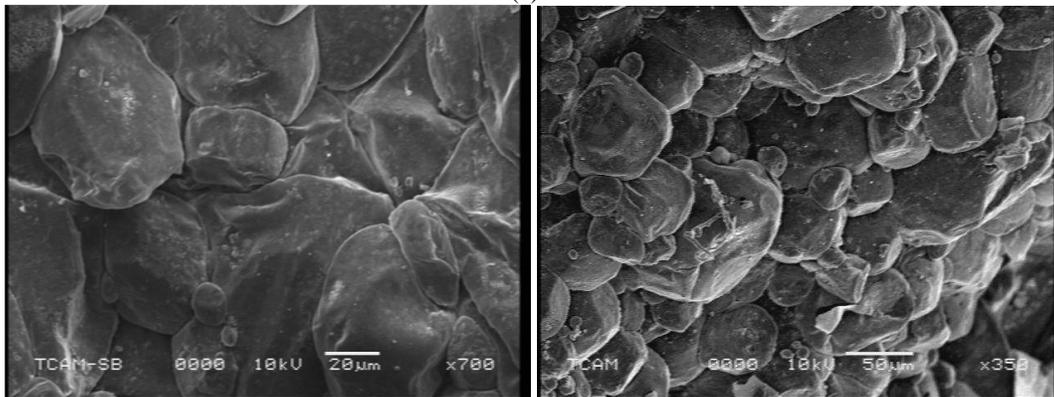
b)1% SiC and c) 1% SiO₂ nanophased expancel foam

It is observed in Fig. 1 that the compressive stress of the 1% SiC and 1% SiO₂ nano systems are about 78% and 14% respectively increased as compared with the neat expancel foam samples. The 1% SiC nanocomposite also shows 20% improvement in compressive modulus. This improvement may be the result of increasing the interfacial bond between the nanoparticles and polymeric matrix.

SEM analysis has been carried out to understand the morphology and dispersion of SiC and SiO₂ in expancel foam before and after the compression test. Figure 2(a) show that all the microspheres are expanded and typical sizes measured are about 40-100µm. Expanded nanophased microspheres are shown in Fig 2(b) these results show that the microspheres are slightly higher (60-120µm) than the neat microspheres and more uniformly expanded where as the 1%SiO₂ microspheres sizes are close to the neat microspheres. The reason for this may be due to the high conductivity of SiC helped in local heating of the microspheres to expand larger and more uniform compare to the neat and 1% SiO₂ samples.

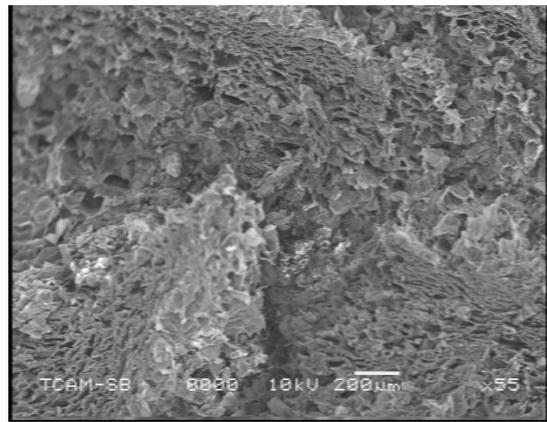


(a)

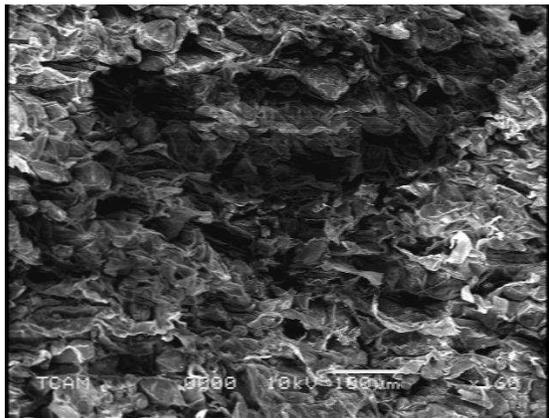


(b)

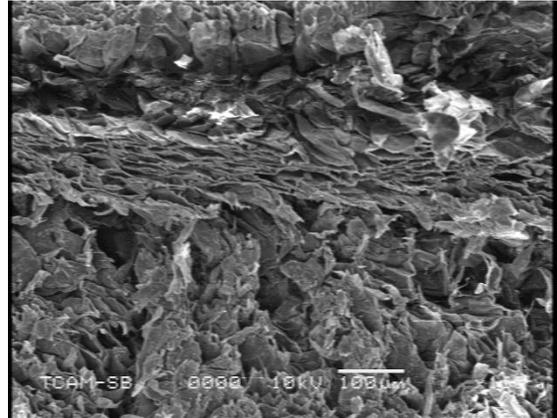
(c)



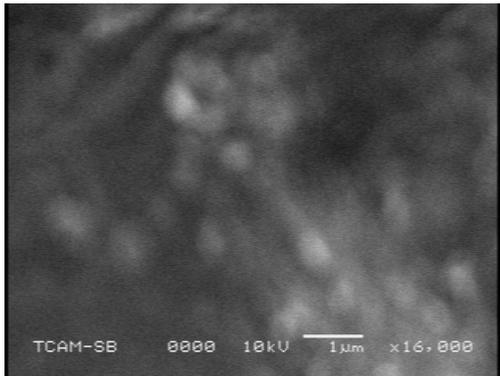
(d)



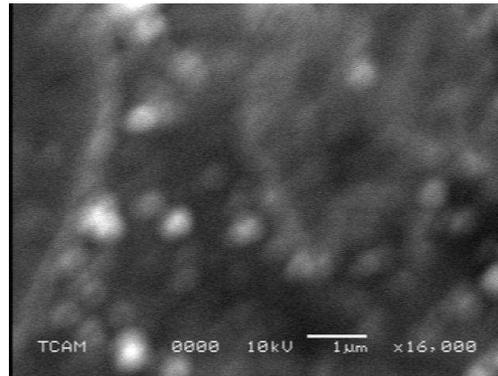
(e)



(f)



(g)



(h)

Figure 2 : SEM Pictures of a) neat expancel foam b) 1% SiC nanophased expancel c) 1% SiO₂ nanophased expancel d) neat expancel foam after compression e) 1% SiC nanophased expancel after compression f) 1% SiO₂ nanophased expancel after compression g) dispersion of SiC on expancel microspheres h) dispersion of SiC on expancel microspheres

Figure 2(d), 2(e) and 2(f) depicts the SEM pictures of the neat 1%SiC and 1%SiO₂ nanophased microspheres after compression test respectively. These micrographs clearly show that most of the microspheres are ruptured after compression. Neat expanded foam showed more collapsed cells and cracks in foam cells in all directions than the nanophased expanded foam after compression. To understand this we have carried out the SEM analysis at higher magnification as shown in figure 2(g) and 2(h). These results show that the SiC and SiO₂ are uniformly coated on the expanded microspheres. This uniform coating with adhering of the SiC on microspheres translated in to the enhanced compression strength and modulus.

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REFERENCES

- [1] DIAB Inc. Technical Information for Divinycell Foam; (2002).
- [2] A.G.Rohm, Technical Information for ROHACELL Foam; (2002).
- [3] Baltek Corp. Technical Information ;(2002).
- [4] J. Marsh, Fire-safe composites for mass transit vehicles. Reinforced Plast 46(9), (2002), 26–30.
- [5] Klas Elfving and Bjorn Soderberg. Reinforced Plastics June 64, (1996).
- [6]. Massimo Tomalino, Giovanni Bianchini, Progress in Organic Coatings 32, (1997), 17-24
- [7] Young-Wook Kim, Shin-han Kim, Hai-Doo Kim Chul B.Park Journal of Material Science 39 (2004) 5647 – 5652
- [8] Lev Vaikhanski, Steven R. Nutt Composites: Part A 34 (2003) 1245–1253
- [9] M.Antonietti, C. Goltner. Superstructures of functional colloids: chemistry on the nanometer scale. Angew Chem Int Ed Engl 36 (1997), 910.
- [10]. G. Schmid. Clusters and colloids. VCH Weinheim; (1994).
- [11]. G.Schmid. Large clusters and colloids metals in the embryonic state. Chem Rev, 92 (1992)1709.
- [12] Hirai H, Wakabayashi H, Komiyama M. Preparation of polymer protected colloidal dispersions of copper. Bull Chem Soc Jpn 59(1986), 367.
- [13] R.N. Katz, Opportunities and prospects for the application of structural ceramic, in: J.B.Wachtman, Jr. (Ed.), Structural ceramics, Treatise on Materials Science and Technology, 29, Academic Press Inc., New York, 1989, pp. 1–26.
- [14] K. Komeya, M. Matsui, High temperature engineering ceramics, in: M.V. Swain (Ed.), Materials Science and Technology. A Comprehensive Treatment, Structure and Properties of ceramics, 11, VCH Publishers Inc., New York, NY, USA, 1994, pp. 517–565.
- [15] I. Moon, G. Cho, Transperant SiC thin films by PEG-SDS-assisted self assembly of SiC nanoparticles, Mat. Sci. Eng. C 24 (2004) 301–305.
- [16] S.P. Lau, J.M. Marshall, T.E. Dyer, Structural and electrical transport properties of excimer (ArF)-laser-crystallized silicon carbide, Phil. Mag. B 72 (3) (1995) 323–333.