Article

The Use of Recycled Carpet in Low-Cost Composite Tooling Materials

Kunal Mishra ¹ , Sarat Das ¹ and Ranji Vaidyanathan ^{2,*}

¹ School of Aerospace and Mechanical Engineering; Oklahoma State University, Tulsa, OK 74106, USA; kunalm@okstate.edu (K.M.); saratd@ostatemail.okstate.edu (S.D.)

² School of Material Science and Engineering; Oklahoma State University, Tulsa, OK 74106, USA

* Correspondence: vaidyan@okstate.edu; Tel.: +1-918-594-8645

Received: 31 January 2019; Accepted: 7 March 2019; Published: 8 March 2019

Abstract: More than 250,000 metric tons (600 million pounds) of carpet are dumped in landfills every year. That creates a significant concern regarding environmental deterioration and economic liability. It is therefore imperative to develop sustainable post-consumer carpet-based products for high-value engineering applications such as composite tooling. To be considered as an acceptable composite tooling material, the composite needs to meet certain required properties such as a low coefficient of thermal expansion, excellent compressive properties, and high a hardness value after repeated exposure to curing cycles. The tooling composites must also exhibit the ability to endure several curing cycles, without deteriorating the mechanical properties. In the present investigation, post-consumer carpet has been recycled in the form of structural composites for tooling applications. The recycled carpet composites have been reinforced with 0.5 wt.% of graphene nanoplatelets to modify the material properties of the carpet composites. The results from compressive and hardness experiments demonstrate that the recycled carpet preserved its mechanical integrity even after several curing cycles. This indicates that recycled carpet composites have the potential to be a low-cost composite tooling alternative for the industry.

Keywords: VARTM; recycled carpet; tooling; compression; hardness

1. Introduction

Tooling is a quintessential but often undervalued silent partner behind complex designed composite parts. Tooling materials are used to produce a wide variety of plastic, metal, rubber, and glass parts in various applications such as aerospace, automobile, and as structural components.

The use of advanced fiber reinforced composites in commercial aircrafts has become a necessity to achieve higher performance and better fuel efficiency. The aerospace composites are processed inside an autoclave (under optimum temperature and pressure) for the best and reliable final product. The tooling material is used to support and shape composite prepregs during the layup and curing process. It is a critical component in the composite manufacturing process in terms of function, logistics, and cost. Conventional tooling composites such as Invar and carbon, used in manufacturing composite panels, are very expensive, and therefore, development of low-cost innovative tooling material is critical.

Tooling materials can be broadly classified into single-use and multi-use based on curing exposure [1]. Tooling materials and their applications deviate significantly in the composite manufacturing process. Fiber-reinforced polymer matrix composites are attractive resources to make tooling materials. They have low coefficients of thermal expansion, high strength and stiffness to ensure dimensional consistency. Table 1 shows a list of ideal target properties desired in a composite

tooling material. They are compared with the properties of several commonly used tooling materials, including invar [1–3].

High specific strength and stiffness, and design flexibility of the composite allow tools to be significantly lighter than metal tools. They are also easier for handling in manufacturing settings.

Table 1. Properties of existing tooling materials and target materials [1–3].

Property	Invar	Epoxy	Polyurethane	Target
CTE [10^{-6} K^{-1}]	1.3	32	50	1.3
Density [g/cc]	8.1	0.67	0.85	<1
Max. Thickness [cm]	N/A	15	15	>30
Temperature Capability [$^{\circ}\text{C}$]	>260	141	142	160 and 260
Compressive Strength [mPa]	-	63	52	21
Cost [\$/L]	300	25–38	25–35	<20

Many advantages and unique requirements of composite tools including hardness, compressive strength, stiffness, dimensional accuracy, durability, and machinability depend on the matrix/fabric system, production methods, production quantity, and costs. Fillers are often added to increase stiffness and hardness, and to decrease cure shrinkage and the coefficient of thermal expansion.

Low-cost tooling materials are easily castable and machined. In high-performance applications, carbon fiber-based composites are used to manufacture parts, and it is often desirable to have a tool/mandrel with a similar CTE (coefficient of thermal expansion).

Post-Consumer Carpet Composite

250 million metric tons of carpet waste is generated every year in the USA. Only 2% of carpet waste is recycled while the rest is disposed into the landfills [4]. Post-consumer carpet accounted for approximately 1 wt.% and 2 vol% of the municipal solid waste stream in the United States in 2008, and this number has been increasing steadily every year [5]. Nylon is the primary component in 65% of the carpet sold in the US market. It has excellent structural performance as well as being very durable which makes post-consumer carpet a potential structural composite [6]. A novel technique has been reported in the past to recycle post-consumer waste carpet into composite panels based on a modified vacuum assisted resin transfer molding (VARTM) process [7]. The process was further scaled up to manufacture large scale (1 m $\times$ 1 m in size) composite panels with excellent sound absorption capabilities and mechanical properties, as reported by our previous work [8]. The present work is an extension of the works reported in [7,8], in this work we have extended the application of carpet structural composites as tooling material. The structural panels were fabricated with different low-cost room temperature cured polymer resins (epoxy, polyester, and polyurethane) infused through the nylon fibers with excellent surface quality and large sizes. A significant advantage of this process is that it is possible to fabricate the composites without having to remove the parts of the carpet. This option leads to the opportunity of recycling carpet materials to manufacture composite tooling articles with a high-temperature resin system. It is also possible to fabricate carpet composite with complex surface profiles, which is an essential requirement for composite tooling.

It has been reported that the addition of graphene nanofiller into the composite improves the electrical and thermal conductivity, mechanical strength, and reduces the coefficient of thermal expansion [9,10]. In this paper, we illustrate the fabrication process of tooling using a nylon-based carpet, a high-temperature curing resin, graphene filler, and a carbon fiber liner. In the present study, we have implemented the vacuum assisted resin transfer molding (VARTM) technique to recycle post-consumer carpet into structural composites as low-cost tooling materials. The preliminary data including compressive strength, surface hardness, thermal conductivity, and thermal expansion

coefficient are presented. We have also investigated the influence of graphene filler reinforcement in the composite.

2. Experimentation

2.1. Materials

In this study, the nylon cut loop type of carpet with the commercial name “Orange Cool Aid” by Shaw Industries (Atlanta, GA, USA) was selected. The carpet consists of nylon fiber and polypropylene as the backing material. The epoxy resin used for this work is diglycidyl ether of bisphenol A based resin (SC 79, Applied Poleramic Inc, Benicia, CA, USA) cured by an aliphatic amine (SC 79 part B, Applied Poleramic Inc, Benicia, CA, USA). The materials for VARTM such as the peel ply, resin infusion media, breather cloth, release film, breather cloth, spiral tubing, and resin traps were purchased from Fiberglast Development Corporation (Brookville, OH, USA). Graphene nanoplatelets “Grade M” used as filler in the experiment were purchased from XG Sciences (East Lansing, MI, USA). Grade M graphene particles have an average thickness of approximately 6 nm and a typical surface area of 120–150 m²/g, for better dispersion in the epoxy resin. Carbon fiber fabric of 0.009 inch thickness used as a liner on one surface of the composite tooling was purchased from Fiberglast Development Corporation (Brookville, OH, USA).

2.2. Tooling Fabrication

Samples were fabricated using a simple, vacuum assisted resin transfer molding fabrication technique. For this particular study, a Bottom-Top-Top-Bottom (BTTB) carpet configuration was used. In the modified VARTM fabrication process, caul plates and green mesh cloth are included to ensure uniform resin infusion and uniform thickness tooling material. The vacuum mold was prepared on a corrugated glass fiber. It was coated with a thin layer of polyester resin to make the surface smooth. Wax and PVA (polyvinyl alcohol) were applied to make the de-molding easier after the composite was cured. Carbon fiber fabric with the same cut piece size of carpet was initially placed on the mold, and top of it two layers of carpet in BTTB configuration was placed. A schematic and the actual mold with and without the composite precursor are shown in Figure 1. Spiral tubes were placed along the length of the carpet preform as inlet and outlet to ensure uniform resin distribution throughout the carpet. Figure 2 shows a close-up image of cross-section of prepared tooling composite that illustrates the homogeneity of the sample.

For graphene reinforced composites, graphene was added to the epoxy resin in the ratio of 0.5 wt.% graphene to 100 wt.% epoxy. The mixture was stirred for 24 h and sonicated for 1 h for uniform dispersion of graphene in the resin. The hardener was to epoxy-graphene ratio was 40:100. The as-prepared mixture was then infused in the carpet using the VARTM process. The mold was then cured at 60 °C for 4 h at a vacuum pressure of 75 kPa. After de-molding, the composite tooling was post-cured at 121 °C for 4 h as per the resin manufacturer instruction. Flat samples were also prepared under the same conditions for density and mechanical properties characterization.

Figure 1. Vacuum assisted resin transfer molding set up for tooling fabrication (a) Schematic diagram, (b) actual VARTM in the oven, (c) cured composite tooling material.

Figure 2. Close up image of the cross-section of composite tooling materials.

2.3. Characterization of Composite Properties

The fabricated tooling piece was subjected to 15 autoclave cycles of 4 h each at 121 °C, based on the manufacturer's recommended curing cycle for the epoxy resin. Compression test samples of size 25.4 mm × 25.4 mm × 12.7 mm thickness were machined from flat panels, as per ASTM C365 [11]. Compression tests were performed on an INSTRON 8802 (company, Waltham, MA, USA). Shore D durometer hardness was measured using an AD-100-D Durometer (Albuquerque Industrial, Albuquerque, NM). The thermal conductivity of the sample was measured using manually constructed instruments developed based on the Fitch method [12]. The apparatus consisted of a heat source and a heat sink. Two copper rods were used in the instruments. One copper rod was maintained at 100 °C, and the other copper rod used as the heat sink was kept at −10 °C. The copper rods were entirely covered with a thick Teflon insulator. Temperature probes were used to measure the temperature at different heights of the copper rod. The test sample of size 25.4 mm diameter and 14.2 thickness was sandwiched between the copper rods and was kept for 24 h for thermal equilibrium. The density of the composites was characterized using the Archimedes principle [13]. For each experiment, 10 samples were characterized.

3. Results and Discussion

3.1. Physical Properties

The densities of the neat and graphene reinforced carpet composites were found to be 1.16 and 1.24 gm/cc respectively from the Archimedes principle. The increase in the density of the composite is due to the presence of graphene fillers (density 2.26 gm/cc).

3.2. Shore Hardness Test

The fabricated recycled carpet has two surfaces—carbon fiber on one side and polypropylene lining on the other side. The Shore D hardness values of the tooling surface are measured for both the surfaces for each cure cycle of 15 total cure cycles. The results of the hardness data plotted as the function of the number of cure cycles are shown in Figure 3. It was observed that the tooling surface demonstrated no change in the surface hardness with the increase of cure cycles, indicating that the composite maintains its strength and hardness after exposure to each cure cycle. The carbon fiber surface shows higher hardness values compared to the polypropylene surface. The increase in hardness is expected due to more cross-linking of polymers with the subsequent curing cycle. The inclusion of the stiffening graphene filler in the composite increased the surface hardness value.

Figure 3. Shore D hardness as a function of curing cycles.

3.3. Thermal Conductivity Test

The thermal conductivity of the samples was calculated using the Fitch method and was found out to be 0.60 and 0.94 w/mK for neat and graphene reinforced carpet composites. This indicated that the inclusion of graphene filler had increased the thermal conductivity of the tooling material. The increase in the thermal conductivity with the reinforcement of graphene filler is due to the in-plane high thermal conductivity of graphene filler that eventually increases the thermal conductivity of the composites through 'contact' or 'percolation' [13,14].

3.4. Compressive Properties

Compressive modulus results as shown in Figure 4 show that it initially increases with the increase in the curing cycles and eventually gets stabilized at higher curing cycles. The increase in the

compressive modulus is attributed to the increase in the cross-link density with the increase in curing cycles. At higher curing cycles, the epoxy is cross-linked, hence the elastic modulus value is saturated around 1350 MPa. Interestingly, the presence of nanofiller decreases the compressive modulus value compared to neat carpet composite. This decrease in the compressive modulus value may be due to the presence of the graphene nanofiller, which interferes with the chemical crosslinking of the epoxy resin. However, as shown in Figure 5, the yield strength does not exhibit any change with the increase of curing cycles as well as by reinforcement of the graphene nanofiller.

Figure 4. Compressive modulus value of neat and nanofiller reinforced carpet composite.

Figure 5. Yield strength values of neat and nanofiller reinforced carpet composite.

3.5. Preliminary Cost Calculations

With the assumption that recycled carpet would be almost free, the only contributor to the tooling material cost would be the cost of the resin, carbon fiber fabric, graphene filler, the cost of supplies of fabrication, and labor costs. At current volumes, the cost per 1 m × 1 m composite tooling would be approximately \$40.10. The material value is obtained from the supplier and the labor cost is for one person working full time in fabrication. The break-down of the total cost is approximately: resin + curing agent: \$12, carbon fiber fabric: \$9, graphene filler: \$3, cost of supplies: \$8, labor cost: \$8.10. The total value of composite tooling is comparable to the value of polyurethane and epoxy types of tooling materials. The significant advantage of the recycled carpet tooling materials would be in its ability to recycle a substantial waste material into a high-value end product. Further cost reductions with lower cost resins may be required to make this tooling material cost-competitive compared to other existing tooling materials.

4. Conclusions

Low-cost composite tooling with improved physical and mechanical properties with the inclusion of graphene filler compared to the composite tooling containing no fillers was achieved. The composite demonstrated consistent results of hardness and compressive properties as a function of the number of cure cycles. The cost of designing this tool is almost same as that of the tools constructed from epoxy and polyurethane resins obtained from primary cost calculations.

Author Contributions: K.M. works in methodology, conceptualization and writing original draft. S.D. worked on methodology, and writing original draft. R.V. worked on validation, resource and writing-review.

Funding: The authors would like to thank the Oklahoma Center for Advancement of Science and Technology (OCAST) for funding this project under project number OARS AR09.1-055 “Structural Materials from Recycled Carpet”, and the Oklahoma Transportation Center under project number OTCREOS9.1049, “Recycle carpet materials for infrastructure applications.”

Conflicts of Interest: The author declares no conflict of interest

References

1. Private communications with Z. Wing, Advanced Ceramic Manufacturing LLC, Tucson, A.Z. January 2012.
2. Boursier, B.; Callis, R.; Porter, J. A New Composite Tooling Material and Concept for Aerospace Composite Structures. In Proceedings of the Annual Society for Advanced Manufacturing and Processing Engineering, Long Beach, CA, USA, 30 April–4 May 2006. CD ROM—15 pages.
3. Wallen, M.; Rossfeldt, J.; Aune, C.; Wing, Z. Washout Tooling for Hollow Composites: Toward Rapid, Low Cost Production Technology. Paper presented at Society of Manufacturing Engineers Composite Manufacturing Conference and Exhibits, Dayton, OH, USA, 12–14 April 2011.
4. Municipal Solid Waste in The United States. Available online: http://www.wastexchange.org/upload_publications/MSWintheU.S.2010.pdf (accessed on 16 February 2016).
5. Jinghong, D.; Hui-ming, C. The Fabrication, Properties, and Uses of Graphene/Polymer Composites. *Macromol. Chem. Phys.* **2012**, *213*, 1060–1077.
6. McIntyre, D.; Carpenter, G.; Powell, R.R. Process for Recycling Fiber Material and Binder with Novel Injection Mold and Part Made Thereby. U.S. 2007/0212531 A1, 13 September 2007.
7. Jain, A.; Pandey, G.; Singh, A.K.; Rajagopalan, V.; Vaidyanathan, R.; Singh, R.P. Fabrication of Structural Composites from Waste Carpet. *Adv. Polym. Tech.* **2012**, *31*, 380–389. [CrossRef]
8. Mishra, K.; Vaidyanathan, R.K. Application of Recycled Carpet Composite as a potential Noise Barrier in Infrastructure Applications. *Recycling* **2019**, *4*, 9. [CrossRef]
9. Shokrieh, M.M.; Ghoreishi, S.M.; Esmkhani, M.; Zhao, Z. Effects of graphene nanoplatelets and graphene nanosheets on fracture toughness of epoxy nanocomposites. *Fatigue. Fract. Eng. M.* **2014**, *37*, 1116–1123. [CrossRef]
10. Rafiee, M.A.; Rafiee, J.; Srivastava, I.; Wang, Z.; Song, H.; Yu, Z.Z.; Koratkar, N. Fracture and fatigue in graphene nanocomposites. *Small* **2010**, *6*, 179–183. [CrossRef] [PubMed]

11. ASTM International. Standard Test Method for Flatwise Compressive Properties of Sandwich Cores. In *ASTM Standard DC365/C365M*; ASTM International: West Conshohocken, PA, USA, 2011. Available online: www.astm.org (accessed on 5 March 2019).
12. Fritchle, F.P. Fitch's. Apparatus for the Measurement of The Thermal Conductivity. *Am. J. Phys.* **1951**, *19*, 475–951. [[CrossRef](#)]
13. ASTM International. Standard Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement. In *ASTM D792-13*; ASTM International: West Conshohocken, PA, USA, 2013. Available online: www.astm.org (accessed on 5 March 2019).
14. Fugallo, G.; Cepellotti, A.; Lorenzo, P.; Lazzeri, M.; Marzari, N.; Mauri, F. Thermal Conductivity of Graphene and Graphite: Collective Excitations and Mean Free Paths. *Nano Letters* **2014**, *14*, 6109–6611. [[CrossRef](#)] [[PubMed](#)]

© 2019 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (<http://creativecommons.org/licenses/by/4.0/>).