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## Functionally graded carbon nanofiber-phenolic nanocomposites for sudden temperature change applications

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### ABSTRACT

Functionally graded carbon nanofiber-phenolic nanocomposites (FGN) with four different gradient patterns and the same total carbon nanofiber (CNF) content and geometry were designed and fabricated. A Non-graded nanocomposite (NGN) was also fabricated for comparison purposes. Microstructural characteristics were examined by optical and scanning electron microscopy. Temperature-dependent thermal conductivity and heat capacity of the FGN components were measured and used in finite element analysis. A finite element analysis of transient thermal behavior of FGNs and NGN subjected to sudden temperature changes was performed to investigate the effects of compositional patterns on temperature gradient field. The finite element model was validated using a custom-made experimental setup. The results showed that the FGN with a gradual decrease in CNF content from the temperature-exposed side to the other had the lowest temperature gradient field and transient time.

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### 1. Introduction

Nanocomposite materials are a relatively new class of materials with a reinforcing constituent in the nano scale [1–5]. They have generated much interest due to their superior thermal [6], mechanical [7], electrical [8,9], and physical [10,11] properties, often for low concentrations of additives. There are many applications in which nanocomposites are subjected to rapid temperature changes. One side of the structure which is exposed to temperature change tends to expand or contract, while the other side resists, inducing thermal stress in the structure and cracking occurs if the stress reaches the yield strength of the material, known as thermal shock. This can result in catastrophic failure under severe temperature changes. Therefore, the investigation of the thermal response of nanocomposites under such rapid temperature changes is vital to optimize the material design and fabrication process.

Considerable efforts have been made to improve this low reliability and durability problem arising from rapid temperature changes. Thermal shock resistance can be improved by enhancing the mechanical properties of the materials as well as reducing the temperature gradient field between two sides of the structure to allow a more uniform expansion or contraction [12]. The latter can be achieved by introducing the concept of graded microstructure and material properties over the thickness of the material, which are called functionally graded materials (FGMs). FGMs are advanced composite materials consisting of two constituents with a controlled compositional gradient in one or two directions of the structure [13]. One of the main applications of FGMs is as thermal barrier coatings, allowing for a smooth transition from metal to ceramic in aerospace industry [14–16]. In the past two decades, a number of computational and experimental investigations have dealt with thermal behavior of FGMs. Zhou et al. [17] used a diffusion model to show that the graded parameter and the heat transfer parameter have a significant effect on the temperature distribution of a continuously graded ceramic/metal composite strip in contact with well stirred fluid. Cho et al. [18] studied the effects of the material variation through the thickness in functionally graded composites on temperature gradient and resulting thermal stress to optimize and control thermal stress using the

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finite element method. It has been reported that in-plane direction (not through the plate thickness direction) properties' variation with a power law considerably affects both in-plane temperature distributions and heat transfer periods in a ceramic/metal composite plate subjected to an in-plane heat flux [19] and edge heat flux [16]. Agarwal et al. [20] obtained time-dependent temperature profiles in laminates having discrete layers of ceramic and metal composites. Effect of layer stacking sequence in laminated composites under dynamic conditions was examined by Tian et al. [21]. Agarwal et al. [20] obtained time-dependent temperature profiles in laminates having discrete layers of ceramic and metal composites. Effect of layer stacking sequence in laminated composites under dynamic conditions was examined by Tian et al. [21]. Lee et al. [22] estimated the temperature distributions and thermal stresses in a functionally graded hollow cylinder simultaneously subjected to inner-and-outer boundary heat fluxes using an inverse algorithm. Hein et al. [23] studied the transient temperature and thermal stresses for a three-layered strip subjected to constant temperature jump on the top surface of FGM using a finite difference method. Zhao [24] analyzed one-dimensional unsteady temperature field and unsteady thermal stress field for a functionally graded ceramic plate using a perturbation method and showed a significant improvement in thermal shock resistance. They also investigated the effects of the radial distributions of thermo-physical properties on the thermal shock resistance of the FGM solid cylinder and compared it with non-graded materials [25].

Wei et al. [26] fabricated functionally graded material based on Ti and TiB using spark plasma sintering (SPS) process and investigated the temperature and stress distributions using a finite element model. Jin et al. [27] fabricated Mullite/Mo functionally graded material using a powder metallurgy process and showed that FGM samples have better thermal shock resistance compared to non-graded materials. Hamatani et al. [28] fabricated FGM using low pressure plasma spraying method and investigated the effects of the composition profile in FGM on the thermal shock resistance by using a modified temperature difference test. Zhao et al. [29] fabricated functionally graded  $Al_2O_3$ -TiC and  $Al_2O_3$ -(W,Ti)C and examined the thermal behavior by water quenching and showed an improvement in thermal shock. Bhattacharyya et al. [30] also showed enhancement in the thermal shock of functionally graded aluminum-silicon carbide (Ni/ $Al_2O_3$ ) and nickel-alumina (Al/SiC) samples. Zhang et al. [31] fabricated SiC/C functionally graded materials and investigated the effect of the number of graded layers on thermal shock resistance.

The concept of FGMs has been recently applied to nanocomposites to optimize the nano-scale reinforcing constituent consumption and provides nanocomposites with different properties for multifunctional applications. Erisken et al. [32] fabricated functionally graded scaffolds from biodegradable polymers to enable the mimicking of native tissue. Ergun et al. [33] developed multiple-layered scaffolds with wide ranges of pore size, porosity, and mechanical properties for tissue engineering applications using the twin screw extrusion and co-extrusion technologies. Chen et al. [34] fabricated gradient multilayer CNTs/ $SiO_2$  composites and reported significant improvement in microwave absorbing properties. Wang et al. [35] presented a novel high flux filtration medium, consisting of a three-tier composite structure for oil/water emulsion separations. We have also previously reported the fabrication of stepwise nanocomposites with a significant improvement in thermo-mechanical and viscoelastic properties [36–38]. Aliofkhaezai et al. [39] fabricated functional gradient nanocomposite coatings by plasma electrolytic oxidation based on the variable duty cycle. However, functionally graded carbon nanofiber/polymer nanocomposites and their transient thermal behavior have not been investigated in the literature. In this paper, The FGN plates

were composed of eight layers including four components of 0, 2, 4, and 16wt% CNF (two layers of each). Four gradient patterns were designed by changing the order of the constituent layers. A transient thermal analysis was performed on FGN plates using the commercially available finite element analysis software, Abaqus. Experimentally measured temperature-dependent thermal properties were used in finite element analysis. Thermal conductivity and heat capacity of nanocomposites were measured using the modified transient plane source (MTPS) technique. An experimental setup was designed to validate the finite element model. The temperature gradient field and transient time in the FGN plates exposed to two extreme temperatures of  $-100$  to  $+100$  °C were analyzed.

## 2. Materials and methods

### 2.1. Materials and sample fabrication

Graphitized carbon nanofibers with diameters of 200–500 nm, 99% purity and density of 1.75g/cm<sup>3</sup> supplied by Nanostructured & Amorphous Materials, Inc., were used as nano-scale reinforcing constituents. A phenolic thermosetting resin with ~9% hexamethylenetetramine obtained from Hexion Specialty Chemicals Pty Ltd was used as the matrix material. Functionally graded nanocomposites (FGNs) with four different gradient patterns with the same thickness and total CNF content (5.5wt%) and a non-graded nanocomposite (NGN) with 5.5wt% CNF were fabricated. The samples were designed to have eight layers consisting of two layers of 0, 2, 4, and 16wt% CNF. Four different gradient patterns were designed by changing the order of the layers. The powder mixtures of CNFs and phenolic resin with 2, 4, 5.5 and 16wt% CNF contents were ball milled for 3 min (8000M, Mixer/Mill, Maker, Spex, USA) to ensure homogeneous dispersion of CNF within the phenolic resin. Premixed composite powders of certain CNF contents were settled in a die of a hot press in a desired sequence using a stacker device. The graded stacks of composite powders were heated up to 130 °C for 10 min and subsequently hot pressed at 180 °C and 15 MPa for 10 min and finally cooled down.

### 2.2. Optical macroscopy

Optical macroscopy was used to investigate the cross section of different configurations of FGNs using Olympus SZX12 digital camera coupled with an Olympus DP70× optical microscope. Prior to imaging, the samples were mounted in casting resin, ground using three different grit silicon carbide papers on a Struers polishing and then polished on an MD-Mol, MD-Nap and MD-Chem surface with the abrasive types of DP-Susp, p 3 μm, DP-Susp, p 1 μm and OP-S, 0.04 μm, respectively.

### 2.3. Scanning electron microscopy (SEM)

The distribution of CNFs within the phenolic matrix was investigated with a scanning electron microscope (SEM Supra 55VP) at an accelerating voltage of 5 kV and a working distance of 7 mm.

### 2.4. Thermal conductivity measurement

The thermal conductivity of the nanocomposites containing 0, 2, 4, 5.5 and 16wt% CNF, as the composing materials of the FGNs and NGN, was measured with the C-Therm Thermal Conductivity Analyzer employing the modified transient plane source (MTPS) technique. Thermal properties were measured at a temperature range from  $-20$  to 100 °C. The hot pressed disk-shaped samples with a diameter of 30 mm and the same thickness were placed on

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