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## Advanced carbon fiber composite out-of-autoclave laminate manufacture via nanostructured out-of-oven conductive curing

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#### A R T I C L E I N F O

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#### ABSTRACT

Next-generation composite manufacturing processes are needed to overcome several limitations of conventional manufacturing processes (e.g., high energy consumption). Here we explore, via experiments and modeling, the characteristics of the newly developed out-of-oven (OoO) curing technique that cures a composite laminate via resistive heating of a carbon nanotube film. When compared to oven curing of an aerospace-grade out-of-autoclave (OoA) carbon fiber prepreg advanced composite laminate, the OoO curing reduces energy consumption by over two orders of magnitude (14 vs. 0.1 MJ). Thermophysical and mechanical tests including differential scanning calorimetry (DSC), dynamic mechanical analysis (DMA), short beam shear (SBS), and *ex-situ* and *in-situ* double-edge notch tension (DENT) indicate that the physical and mechanical properties of OoO-cured laminates are equivalent to those of oven-cured (baseline) laminates. In addition to energy savings, the OoO curing process has the potential to reduce part-to-part variations through improved spatiotemporal temperature control.

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#### 1. Introduction

Manufacturing of aerospace structural composites has traditionally focused on using autoclaves to achieve high-quality reproducible parts, including high fiber volume fractions and low porosity [1,2]. Specifically, carbon fibers, which are preimpregnated with a thermoset or thermoplastic resin to form prepreg sheets, are primarily used for autoclave processing techniques because of their ease of use and exceptional mechanical performance. However, manufacturing composites within an autoclave is accompanied by high acquisition and operation costs due to the necessity of a specialized heated pressure vessel to suppress the formation of voids. Furthermore, the capacity of autoclaves limits the size and design of parts, and the production rate is primarily affected by autoclave availability. As a result, there has been an increasing interest in the development of alternative

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https://doi.org/10.1016/j.compscitech.2018.02.031 0266-3538/© 2018 Elsevier Ltd. All rights reserved. techniques. For example, the previous studies reported manufacturing approaches changing the method of heating such as microwave heating, induction heating, laser heating, and resistive heating of carbon fibers or carbon nanotube (CNT) fillers in composites [3–6]. Additionally, specially-formulated and designed prepregs that can be cured in an oven (out-of-autoclave, or OoA prepregs) have been recently developed to remove the need of an autoclave [7,8].

OoA prepregs with oven curing have been introduced commercially as an alternative to autoclave-cured prepreg manufacturing. In contrast to the autoclave prepregs, OoA prepregs do not require the use of pressure vessels to achieve a void-free laminate because of their formulation and unique structure; dry regions between resin-rich regions in OoA prepregs function as built-in void extraction channels [8–15]. Thus, OoA prepregs can be cured with conventional thermal ovens thereby allowing lower cost manufacturing than using autoclave-cured prepregs [16,17]. Nonetheless, even the use of conventional ovens is not completely ideal from a manufacturing perspective. Heat transfer is still based on convection, which leads to inefficiencies and to spatial gradients

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in cure and stress due to convective-to-conductive interactions between the oven gas medium (usually air) and the cure materials [18–20]. This also drives part-to-part variability, and fabrication is still limited because of its fixed geometry despite advantages of an oven vs. an autoclave.

Given the limitations above, the concept of an out-of-oven (OoO) process has been proposed, which uses a CNT film as a heating element directly integrated into the surface of a laminate so that curing does not require any heating vessel or convective medium [21]. However, while the previous study demonstrated the concept of OoO curing along with degree-ofcure (DoC) comparisons, mechanical and physical properties of OoO-cured composites have yet to be comprehensively evaluated, particularly properties dominated by interlaminar failure, a key area to evaluate for laminated composites [22]. To our knowledge, the direct comparison between OoO and traditional curing from an energy consumption analysis has not been reported in the extant literature. In the current study, we compare the OoO curing vs. oven curing using an OoA prepreg system, and find that the OoO curing enables highly efficient manufacturing of composites while preserving the mechanical properties, particularly interlaminar strength, equivalent to the conventional oven method.

Fig. 1a illustrates the overview of the conventional oven vs. the OoO curing process. Conventional oven curing processes as well as autoclaves that use convective heating require the entire vessel volume to be heated, regardless of the geometry of the component. Therefore, as shown in Fig. 1b, the electrical power for resistive heating should pass through several thermal barriers such as heat loss to the environment, the gas medium (here, air), and heating of vacuum bagging and cure materials to heat up a laminate. As a consequence, the energy consumption increases dramatically as the size of the structure increases. In contrast, the OoO curing has no thermal barriers between the heater and laminate, and thus transfers the heat via direct conduction because the CNT heater is installed on a surface of a laminate. Since the heat loss to the environment is connected to the heater in parallel, thermal insulation can suppress the heat loss, enabling most of electrical power to go into the laminate from the heater. Additionally, because the CNT film has extremely low thermal mass due to its low density  $(\sim 25 \text{ gsm})$ , the electrical power can increase the temperature of a laminate immediately.

#### 2. Material and methods

The characteristics of the OoO curing process were explored by tracking thermal responses and electrical power consumption during a cure cycle. To compare the mechanical and physical properties of OoO-cured composites with those of oven-cured composites, degree of cure analysis, short beam shear test, dynamic mechanical analysis, and double-edge notch tensile testing were performed.



**Fig. 1.** Comparison of oven and out-of-oven (OoO) manufacturing process for out-of-autoclave laminate curing: (a) Overview of the physical differences between oven and out-ofoven process. (b) Comparison of thermal equivalent circuit model of oven and out-of-oven process. The circuits are symmetric with respect to the laminate. Note that the OoO process provides direct conductive heat transfer to the laminate, whereas the oven process passes through thermal barriers including the oven medium (air), vacuum bag, and cure materials.

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