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Multi-Physics Damage Sensing in Nano-Engineered Structural Composites

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Abstract. Non-destructive evaluation techniques can offer viable diagnostic and prognostic routes to mitigating failures in engineered structures such as bridges, buildings and vehicles. However, existing techniques have significant drawbacks, including poor spatial resolution and limited *in situ* capabilities. We report here a novel approach where structural advanced composites containing electrically-conductive aligned carbon nanotubes (CNTs) are ohmically-heated via simple electrical contacts, and damage is visualized via thermographic imaging. Damage, in the form of cracks and other discontinuities, usefully increases resistance to *both* electrical and thermal transport in these materials, which enables tomographic full-field damage assessment in many cases. Characteristics of the technique include the ability for real-time measurement of the damage state during loading, low-power operation (*e.g.*, 15°C rise at 1W), and beyond state-of-the-art spatial resolution for sensing damage in composites. The enhanced thermographic technique is a novel and practical approach for *in situ* monitoring to ascertain structural health and to prevent structural failures in engineered structures such as aerospace and automotive vehicles, wind turbine blades, among others.

Keywords: CARBON NANOTUBES, HIERARCHICAL STRUCTURES, COMPOSITE MATERIALS, SENSORS, STRUCTURE-PROPERTY RELATIONSHIPS

Subject classification numbers:
PACS: 81.07.-b, 81.07.De

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1. Introduction

Catastrophic structural failures cause significant physical and personal losses [1] in applications ranging from vehicles to infrastructure. *In situ* diagnostic and prognostic sensors and systems provide a route to prevent such failures. A variety of non-destructive evaluation (NDE) and structural health monitoring (SHM) techniques have been developed for *in situ* analysis of such structures to detect cracks and other damage at pre-critical levels for remediation [2,3]. These techniques input a controlled perturbation to the structural system, such as ultrasonic waves [4-6], X-ray signatures, heat flux [7,8], electrical current [9,10], etc., and measure the signal changes relative to a baseline. Changes from the baseline are indicative of damage. Existing technologies involve bulky and costly equipment, and require scanning of the complete structure or multiple and complex electrical and/or optical connections, making them impractical for many applications, and furthermore, their resolutions are limited. Thus, *in situ* measurement techniques with higher resolution and efficacy are desired and are the subject of much research and development [11,12].

Among existing damage detection methods, infrared thermography, based on heat transmission within the structure, is a promising technology. The infrared thermography technique works with the widest variety of structural materials, including advanced composites that require more refined inspection and hold interpretation challenges relative to metals [13,14]. A recent study indicates that thermography is advantageous in terms of resolution over all other structural inspection techniques for advanced composites [15]. Thermographic inspection requires an external heating source [16,17] to create a large impulse of thermal energy that is then monitored as it propagates (and dissipates) in the structure of interest over short time periods. The internal state of the structure is assessed with a thermographic camera, usually at high rate to capture temporal effects. However, this technique requires further improvement. Currently, heat is supplied to the structure through external sources such as flash lamps [18], infrared radiators [19] or heat emitting layers [20]. Thus, placement of these large heat generators [20] mitigates *in situ* imaging for nearly all applications. Particularly for composite materials such as those studied here, the thermographic signatures contain significant noise due to material anisotropy and inhomogeneities such as ply interfaces.

Here, we report a new technique for *in situ* sensing that uses local, low-power resistive heating (Joule-effect) [21] to enable high-resolution inspection of damage and damage progression using thermography. The technique is demonstrated for multiple typical and critical configurations. Instead of using large heat generators mentioned above, the (hybrid) nano-engineered composite materials can be locally heated via resistive heating, eliminating the need for external or additional heaters. An exemplary case with a through-crack is demonstrated in figure 1. Long (microns) aligned CNTs are implemented within the microstructure of the advanced filamentary composite, such that damage alters the conductive CNT network, resulting in heat distributions around damage that can be visualize via standard thermography. Effective management of electrical conductivity, particularly low percolation thresholds (0.1-1vol% percolation threshold [22] for polymeric nanocomposites), are key factors for enhanced sensing.

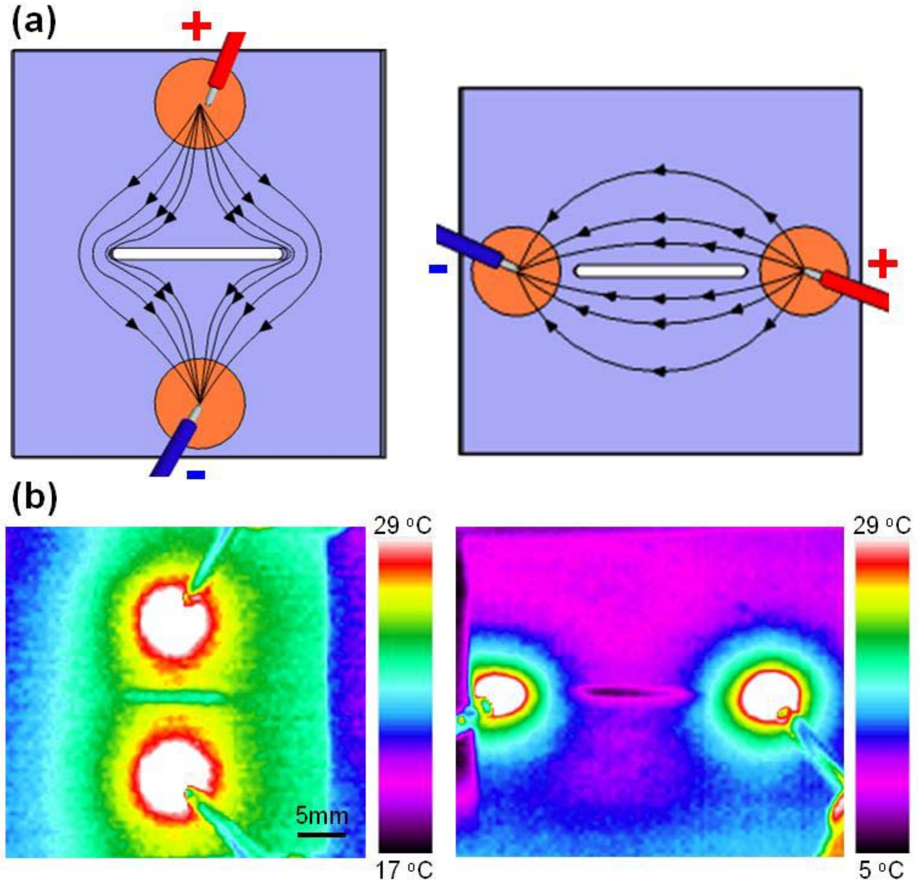

Figure 1. Resistive heating of a through-crack (elongated line defect) in the nano-engineered composite: (a) illustration of electric field line concentrations and local heating, and (b) thermographs with power application of < 1W.

The NDE technique introduced here, termed Nano-Engineered Thermal NDE (NET-NDE), departs significantly from recent work using CNTs and other conductive nanofillers to perform structural sensing. Previously, either the piezoresistive effect correlated with strain, or electrical resistance changes due to damage, are used for structural sensing [1,23-27]. With those techniques, electrode position and density set difficult limits on spatial coverage and resolution. The NET-NDE technique is not similarly limited, but rather is limited only by spatial and temporal resolution of the commercial off–the-shelf thermographic imaging equipment that is advantageous in terms of resolution, especially for sensing damage in advanced composites. Extant NDE configurations utilizing nanomaterials include sensing layers that attach to the surface of the actual structure of interest [1,24]. With such appliqué-type configurations, damage sensing is limited to instances where the structure and the sensing appliqué/film are damaged simultaneously, and can result in false positives (damaged sensor but not damaged structure). By contrast, in the NET-NDE technique, aligned CNT-based conductive networks are integrated within the structure, and thus directly sense internal damage. The nano-engineered composite is truly multi-functional and function as the sensor itself, allowing direct and accurate sensing of the damage state (both surface and internal are demonstrated) with several useful characteristics such as low-power operation, tomographic-type sensing, and beyond state-of-the-art (for composites) resolution.

2. Experimental approach

Nano-engineered hybrid composites are fabricated using a technique previously reported to create laminated composites with enhanced mechanical properties due to the integration of aligned CNTs. Thermal heating is accomplished via simple resistive heating, and damage visualized using a commercial off–the-shelf thermographic camera.

2.1. Nano-engineered composite fabrication

Aligned CNTs were grown on the surface of alumina fibers (11μm diameter) in tow form that are pre-woven into a cloth. The $0^o/90^o$ satin-weave cloth (900g/m^2, ~1mm thick) was dipped in a 50mM solution of iron nitrate in isopropanol. After solvent removal by evaporation, the cloth was placed in an atmospheric pressure quartz tube furnace (Lindberg, 5cm inner diameter) to grow CNTs using chemical vapor deposition (CVD) with ethylene feedstock at 650^oC [28]. Aligned ~30μm-long multi-walled CNTs were grown uniformly on fiber surfaces in a "mohawk" morphology (see figure S1(a)), with an average diameter of 17nm. CNT alignment is important for mechanical enhancement [28,29] and also significantly enhances electrical conductivity over short, randomly-oriented CNTs [30]. Composite specimens were made by hand lay-up using a commercial epoxy system (Resin 105 and Hardener 206, West Systems Epoxy) to create 3-ply laminates. Capillary forces allow the aligned CNTs to be wet completely by the polymer [30,31]. The resulting composites have ~50% alumina fiber volume fraction and CNT volume fraction of ~2%. Fabricated nano-engineered composites were wet-machined using a diamond-grit circular saw and core drill.

2.2. Thermal imaging

Specimens were heated via application of electrical current through the composite (Joule-effect heating). Temperature was recorded using a thermal camera (PCE-TC 3, PCE Group) with 160x120 pixels over a temperature range of -10 to 250°C (a resolution of 0.15^oC). Four damage configurations were tested to demonstrate the NET-NDE concept and assess key characteristics. Baseline specimens (containing no CNTs) were not considered due to their extremely high resistivity (~10^7 more resistive than the CNT-containing composites) that prevents Joule-effect heating at relevant power levels. First, thermoelectrical properties were evaluated by DC power application to a nano-engineered composite with a through-crack via two-probe contact on the sample surface (see figure 1 and S2). Thermal images were taken 30 seconds after voltage application, when the temperature stabilized. Second, required power and resolution for this new technique were evaluated. A pristine (undamaged and unholed) nano-engineered composite sample with copper adhesive tape (Compac Corporation) electrodes was assembled and connected to a 9V alkaline battery via alligator clips, and assessed for power vs. temperature rise. In addition, an aligned CNT forest (40μm long) was also tested, which was grown on a silicon substrate using a similar CVD method (details can be found in [30,31]). The aligned-CNT forest (containing no polymer) was connected to a battery through silver paste contacts and imaged to evaluate resolution limits through detected defect size. Third, after the above evaluation of the NET-NDE technique, a nano-engineered composites sample with a 6.5mm diameter hole was imaged *in situ* during a tensile testing to failure, at a loading rate of 0.2mm/s. This is a demonstration of an *in situ* monitoring of damage state with progressive loading in a relevant (bolt hole) specimen. DC power was applied using silver paste (MG Chemicals) electrodes and steady-state thermal images were taken every 30 seconds with the thermal camera placed ~0.5m from the sample. Fourth, in addition to visible damage observed in the above, nano-engineered composite samples with vanishingly-small surface or internal cracks (delaminations) were imaged to assess sensitivity and full-field capability of this technique. Two types of samples were prepared, the first with an exemplary rivet/bolt hole with a defect, and the second that contains "barely visible impact damage" (BVID) produced by impact. Hand-held test probes were connected to the samples to provide local DC power via contact. All measurements were taken at ambient conditions (25^{o}C, etc.).

3. Results and discussion

Characteristics of the nano-engineered composites and damage visualization via NET-NDE are discussed, followed by several typical and critical engineering configurations.

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3.1. Thermo-electrical behavior of nano-engineered composites

In this work, damage is sensed via thermography due to a combination of effects that damage, such as cracks, has on the electric and thermal transport in the hybrid composite material. Electrical current flows around such damage, and thus electric field lines concentrate in the vicinity of damage (as illustrated in figure 1 for a through-crack flaw), yielding a spatially local temperature increase via Joule heating. In addition to Joule-effect heating due to field-line concentration local to damage, the damage-imaging effect is further enhanced because heat flow (temporally and spatially) is impeded by the increased thermal resistance at the damaged area. Such heating phenomena around cracks due to the concentrated electric field and decreased heat flux are illustrated in figure 1(a) for the simple case of a through-crack perpendicular and parallel to an applied electric field. In the nano-engineered composite, integrated aligned CNTs (~30μm long) form a conductive network throughout the structure, as shown in figure 1(b), enhancing electrical conductivity by ~10^{7}. Percolation is observed in these composites at low concentrations of aligned CNTs; percolation thresholds have been computed for the laminates in both the through-thickness direction (0.03vol%) and in-plane direction (0.7vol%). These low values are below the vol% tested in the samples (2%) indicating that the advanced hybrid composites are well beyond the percolation threshold. With enhanced conductivity (lower resistance), resistive heating is enhanced for a given applied voltage (for 20V, 1.2W and ~16°C are noted in the example in figure 1), resulting in sample temperature rise. At these values, battery-operated power supplies are sufficient to raise temperature to levels that can be easily captured with a standard thermal camera for laboratory-sized specimens.

3.2. Power requirement and resolution of the NET-NDE sensing technique

This new nano-engineered thermographic technique is characterized by low-power operation and the desirable ability to sense small flaws in materials, *i.e.*, damage resolution. As shown in figure 2(a), a large temperature increase (14.5°C) was observed on an open-hole composite plate specimen (6.5mm dia. hole, 80x25x2.2mm^3 in volume) with only 1W applied. Meanwhile, resolution of the technique was investigated by identifying visually unobservable defective (damaged) areas in pure CNT films grown on silicon substrates, as shown in figure 2(b). As for resolution evaluation, when the CNT film is resistively heated (20V and 0.06A), several locations (~0.14mm in diameter) were observed under thermography to have lower temperature than the surroundings, as shown in figure 2(b). Via optical microscopy, these spots were correlated with regions of low CNT density, possibly attributed to catalyst film preparation inhomogeneities or impurities on the substrate prior to CNT synthesis. Regardless of the mode and origin, the sensing technique is able to resolve such defects on the order of 0.1mm.

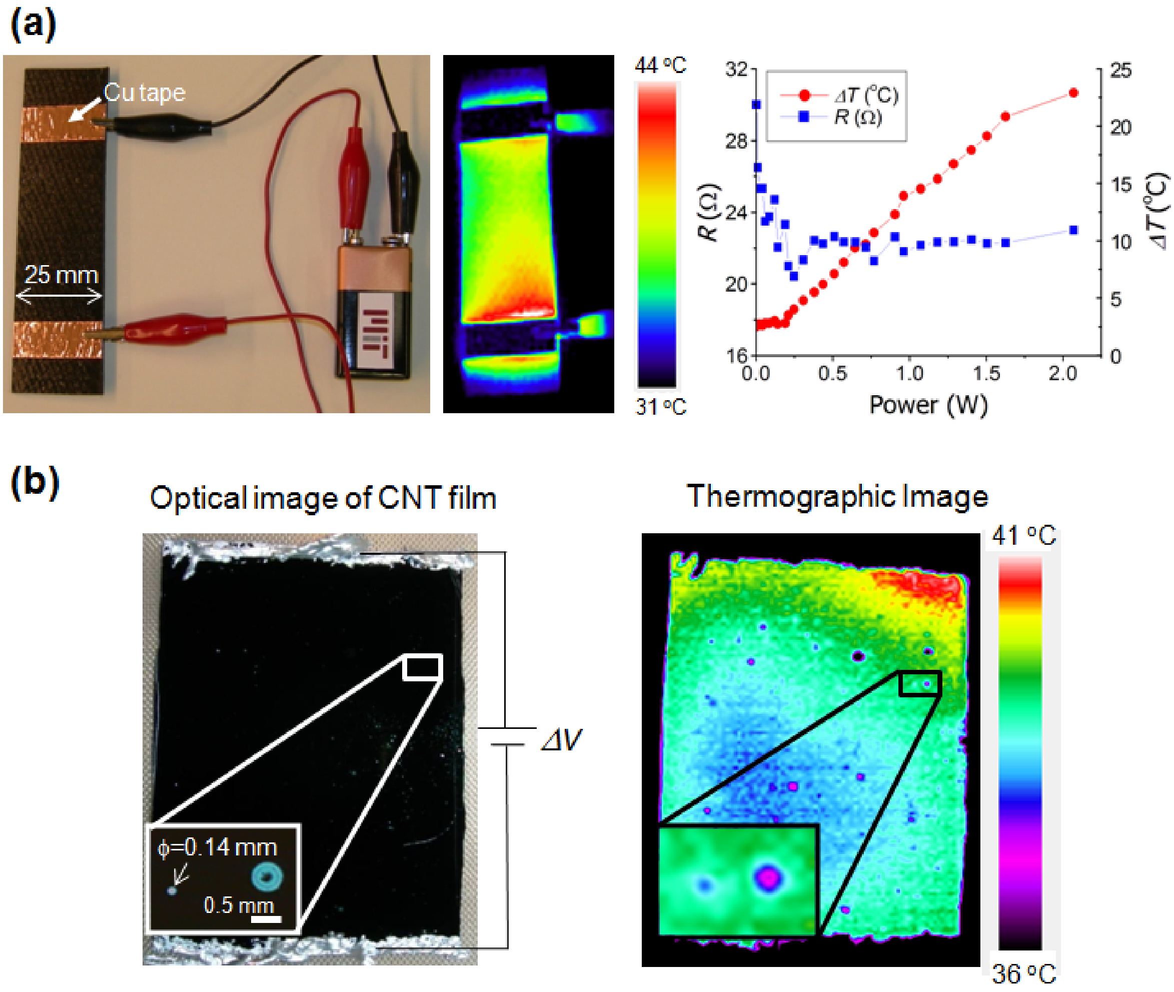

Figure 2. Power and resolution characteristics of the NET-NDE technique: (a) optical image, thermograph, and power-resistance/temperature plot of a composite specimen heated via a 9V battery, yielding ~15°C temperature rise with 1W, and (b) optical image and thermograph of vertically-aligned CNTs with defective regions.

3.3. Progressive damage inspection

A nano-engineered composite coupon with a through-hole was monitored for damage development during a tensile test to failure as shown in figure 3(a) and 3(b), in order to illustrate *in situ* damage detection. A power of 2W (6.8V and 0.3A) increased the temperature of the large sample by 20°C above room temperature. As shown in the collection of thermal images during the loading process (see figure 3(c)), 'hot' regions were observed near the hole where stresses are concentrated, due to electric field concentration and heat concentration (see figure 1). As illustrated in the first three thermographic images of figure 3(c), the average temperature decreases very slightly, which implies that the resistance increases in the applied voltage configuration of the test. This behavior can be correlated with the weak piezoresistive effect of the CNTs in the composite [36]. When the sample began to fail (see figure 3(d)), a principal crack on the right-hand side of the specimen was clearly observed in the last thermograph just prior to full specimen fracture. A higher-frequency thermal camera would allow sub-critical damage (usually matrix cracks and/or delamination) near the hole to be imaged prior to specimen ultimate (catastrophic) failure. In the strength test reported herein, the average temperature on the right side of the hole changed by 3°C whereas on the left side of the hole the change was only 1°C before the specimen failed; the difference was interpreted as a indication of greater sub-critical damage on the right side of the hole prior to net specimen failure, a feature that is rarely observed during such tensile testing using other existing techniques. A thermal gradient was observed in the test sample from top to bottom in the thermographs in figure 3, which is a common feature in the technique (see similar in figure 2).

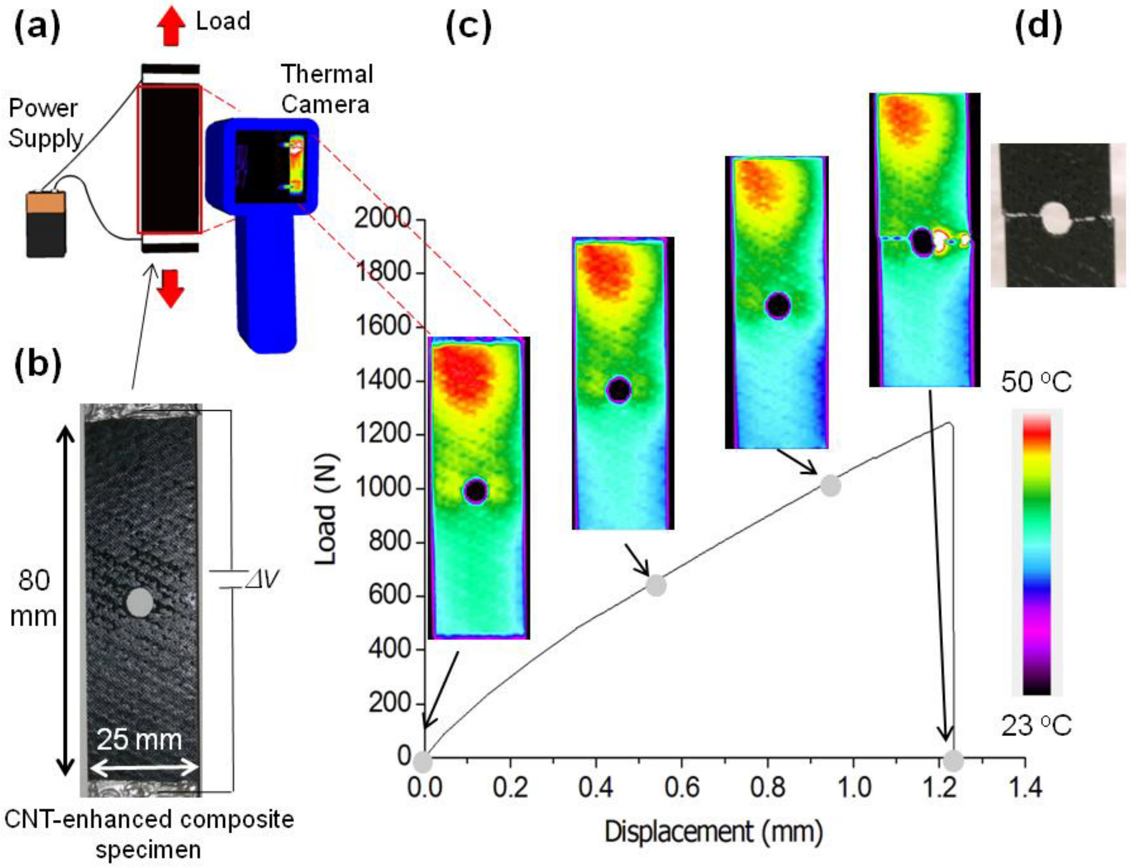

Figure 3. Progressive damage inspection during tensile testing of an open-hole (6.5mm dia.) nano-engineered composite coupon via thermography: (a) setup schematic, (b) optical image of the sample, (c) load-displacement plot with corresponding thermographs (with 2W, taken every 30s at steady state), and (d) optical image of the failed composite after the test.

3.4. Sensing internal damage

Nano-engineered composites were tested for sensitivity to damage states that are much more difficult to detect than simple gross flaws. Attachments such as rivets/bolts (see figure 4(a)) are typical in engineered structures, and are of interest because failures (static or fatigue) often originate in these areas where cracks and other defects may form during the structure’s operational life. Such failure was investigated here via NET-NDE using a nano-engineered composite with a 5mm-diameter hole and a 6.45mm-dia. steel washer and 4.6mm-dia. bolt. Voltage (0.16V and 12.3V) was applied through hand-held probes to induce heating. Cracks introduced at the bolt hole (but under the washer) are easily identified in the thermographs when locally heated via the contact probes, demonstrating a facile method for *in situ* damage detection at critical locations (attachment points are typical sources of failure) in engineered structures. Positioning the electrical heating probes in different orientations local to the bolt hole (as in figure 1) adds another useful dimension for efficient and detailed investigation of damage to create tomographic-like visualizations. Internal damage with little or no visual evidence on the external (visible) surface [32], BVID in composites [33,34], may also be sensed using the NET-NDE technique. A center-impacted composite plate (see figure 4(b)) with BVID was inspected via thermography to reveal the presence of internal damage. Two surface electrodes at the specimen edges were used to heat the specimen resistively ((0.11A at 0.34V) and thermography clearly revealed the presence of BVID at the center of the specimen. This sample nano-engineered composite is equipped with a series of surface electrodes for demonstration of another NDE technique based on electrical resistivity changes induced by damage [35]. The grid patterns observed in the thermograph in figure 4(b) can be attributed to emissivity difference of the non-connected electrode grids. The observed internal impact damage area from this new NDE technique correlate to the location of impact damage and damage revealed using other inspection techniques [35,36].

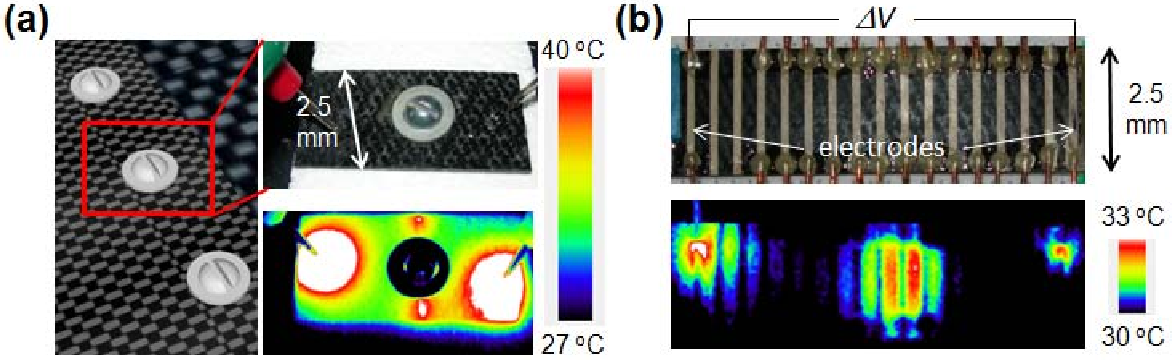

Figure 4. Applications of the nano-engineered thermal imaging technique: (a) damage inspection of a model bolt-hole composite joint, and (b) barely-visible impact damage visualization.

4. Conclusions

We have presented a new technique to evaluate the condition of advanced composite structures using resistive local heating in combination with thermographic imaging. The Joule heating effect of CNT networks embedded within structural composites offers a new and simple way to detect both internal and surface damage *in situ* using thermography when the composite is beyond the electrical percolation threshold. Aligned CNTs provide the conductive network that allows low-power resistive heating, and damage visualization is enabled through change in both electrical and thermal transport around the damage. The local and low-power heating in the NET-NDE technique allow facile and low-power operation, including the opportunity for tomographic-like imaging by varying the spatial positioning of the electrical heating probes in real time. The technique eliminates the need for large external heaters thereby enabling *in situ* sensing for many structural inspection situations. This technique benefits from low-power operation and simple electrical contacts, including hand-applied electrical probes convenient for structural inspection. A series of tests have confirmed that this new technique can sense progressive damage, small cracks (very high resolution damage sensing), and internal damage not visible on the surface. Applications of this new NDE technique include transportation vehicles and infrastructure, including wind turbine blades, concrete-based structures repaired with composite overwrapping [37], etc. The technique is not limited to nano-engineered composites, and can be applied for other building materials such as concrete and foam where CNTs may be incorporated directly. The thermal nano-engineered NDE technique demonstrated here can provide a new and effective inspection route for monitoring future generations of safer vehicles and infrastructure. Additional capabilities related to those demonstrated here include higher resolution thermography, temporal information using high-rate thermographic acquisition, and potentially damage mode detection. In addition to this advantage [38,39] for applications using Joule-heating, CNTs significantly enhance the toughness and strength of the composite materials [28], demonstrating a structural material that is mechanically superior and therefore truly multifunctional when it is used as a sensor (as in NET-NDE).

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Supplementary Information

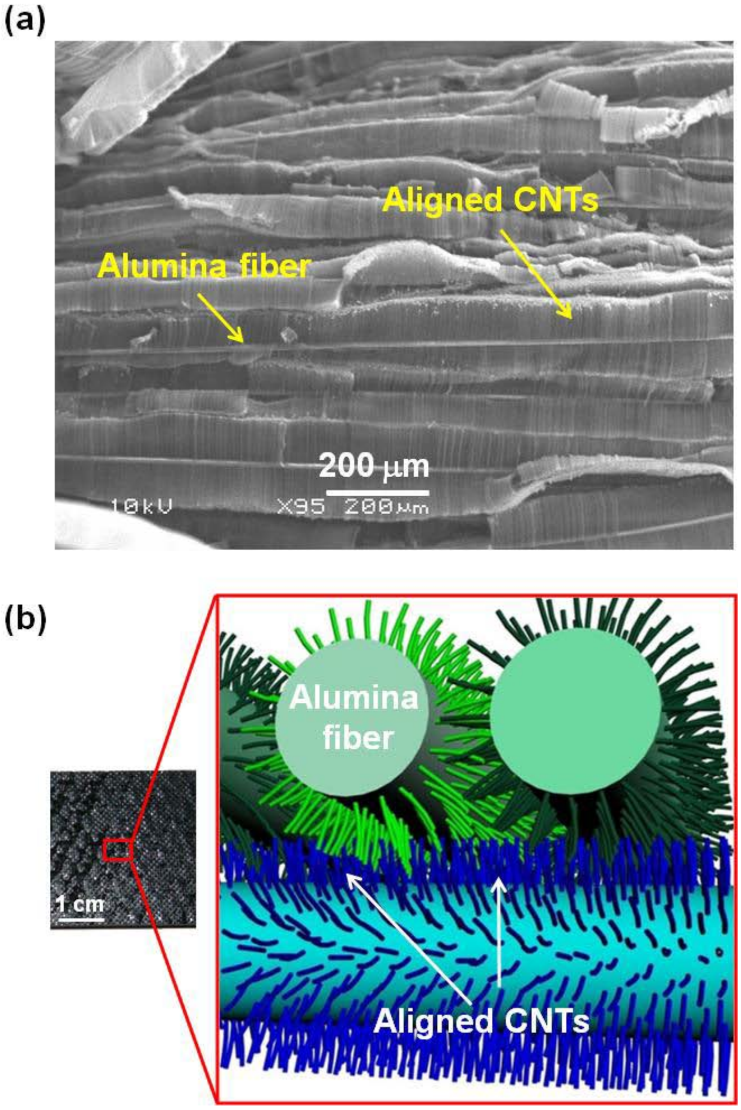

Figure S1. Nano-engineered hybrid composite material and its components: (a) aligned carbon nanotubes (CNTs) grown directly on the surface of fibers comprising a woven fabric, and (b) optical image and illustration of internal nano/microstructure (CNTs and fibers not to scale).

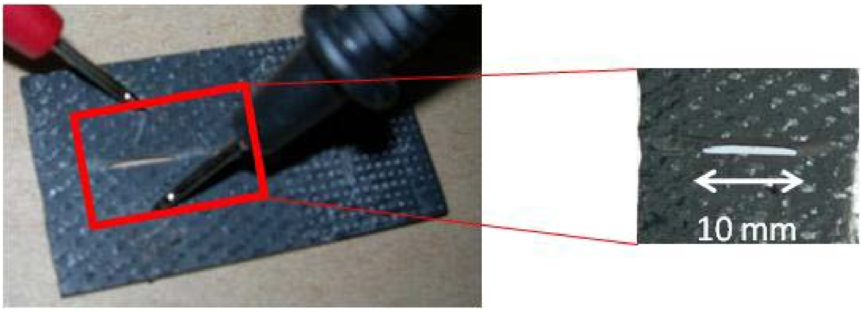

Figure S2. Electrically-induced heating in a nano-engineered composite plate with a 10 mm long through-crack using simple electrical probes; thermographs shown in figure 1(b).