Passive and Active Tagging of Glass-Fiber Polymeric Composites for In-Process and In-Field Non-Destructive Evaluation

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ABSTRACT: Conventional non-destructive evaluation (NDE) methods are not very effective in monitoring the material conditions of advanced composite and adhesive joints. A technology that has been proposed to enhance the inspectability of advanced composites is the particle tagging technique. Two theoretical models were recently proposed to characterize the dynamic behavior of ferromagnetic and magnetostrictive tagging particles. These theoretical models concerning the development of an active tagging technique with embedded ferromagnetic and magnetostrictive particles and magnetic excitation are now experimentally verified. The experimental results of the active particle tagging shows a variation in the dynamic response of the specimens when defects and/or damage are present. The sensory signature from a tagged polymer is extracted as a result of the interaction between the embedded particles and their host matrix. A study of various types of composites and tagging particles for passive and active tagging was performed. Experimental validation of concepts for tagging of structural materials for on-site inspection prior to installation have also been explored. The on-site particle tagging inspection has been verified on laboratory specimens obtained from industry and was shown to be very efficient.

INTRODUCTION

The use of composite materials in civil engineering constructions has been a tremendous development in recent years. The research into the applications of composite materials to the repair of old concrete structures, and into the design and building of new civil engineering structures, partially or totally of composite materials, is developing at a fast rate. Out of the ten 1995 awards of the Civil Engineering Research Foundation (Anon., 1996), five (50%) are related to composite applications. With such an advent of composites into civil engineering applications, it is understandable that the problem of non-destructive evaluation (NDE) of composite civil engineering structures has become a pressing issue. But conventional electromagnetic NDE methods are not very effective in monitoring the material conditions of advanced composites and adhesive joints, due to the non-conducting, non-ferromagnetic nature of most widespread composites. A technology proposed to enhance the inspectability of advanced composites is the particle tagging technique, in which ferromagnetic particles of micron size are mixed in small percentage quantities with the resin or sized on the fibers prior to composite fabrication. The result of this technology, a "tagged composite" that has the ability to respond to magnetic excitation, offers good opportunities for developing new NDE methods and techniques. Several theoretical studies (Sun, Liang, Rogers, and Vick, 1993; Rogers et al., 1995; Zhou, Chaudhry, Rogers, and Quattrone, 1995a and 1995b) have been performed to assess this technology and to find the most promising methods for in-service and in-field implementation.

In this paper, the technology concerning the development of passive and active tagging techniques with embedded ferromagnetic particles and magnetic excitation is studied experimentally. A number of tagged composite samples were manufactured by industrial partners participating in the program. The tagging materials, composite composition, and architecture were varied to cover many of the possible combinations expected to be met in practical civil engineering applications. These industrial samples were sent to the Center for Intelligent Material Systems and Structures at Virginia Tech and were subjected to passive and active tagging tests. The approach used in the passive tagging experiments utilized eddy currents technology. In the ferromagnetic active tagging experiments, vibration measurements were made on small specimens subjected to magnetic excitations in a specially built excitation yoke. The experimental results show that the passive tagging method using eddy current testing is effective in inspecting the presence, the amount, and the distribution of the particles. Although an effort for defect detection was made with the passive tagging method, the experimental results reveal that eddy current responses are not able to interpret delaminations. The experimental results with ferromagnetic active tagging were much more

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promising for detecting defects, cracks and delaminations. The analysis and information presented in the report indicates that the active tagging technique is a valid new option for quality assurance testing of advanced composites in civil engineering applications.

DESCRIPTION OF THE INDUSTRIAL SAMPLES

Samples of tagged polymeric composites were received from the following participating companies: Reichhold Chemicals, Clark-Schwebel, PPG Industries, Interplastic Corp., Owens-Corning, and TPI, Inc.

Tagging Materials

Five tagging materials are used in the samples supplied by the participating companies:

- **Magnetite (Ferric-ferrous Oxide $Fe_3O_4$):** was used in powder form with sizes of 5-micron (Aldrage Chemicals) and 44-micron (Fisher Scientific).
- **Nickel Zinc (NiZn) ferrite:** manufactured by Steward, Inc., was used in powder form with sizes average 2-micron. The NiZn ferrite is a polycrystalline compound of
  
  \[
  \text{Nickel Oxide (NiO): } 9-13\% \text{ by weight} \\
  \text{Zinc Oxide (ZnO): } 17-21\% \text{ by weight} \\
  \text{Iron Oxide ($Fe_3O_4$): } 68-72\% \text{ by weight}
  \]

- **Lignosite FML:** manufactured by Georgia-Pacific Corp., was used in liquid and powder form. Lignosite FML is an aqueous colloidal solution of a ferromagnetic iron lignosulfonate. The size of the ferromagnetic iron in the lignosite FML is between 50 and 200-Angstroms. The powdered form was ground to less than 5 microns. The magnetic fluid has characteristics of a high molecular weight lignosulfonate with an X-ray diffraction pattern typical of magnetite. It can be dried and re-dissolved without separation of the magnetite from the lignosulfonate and without loss of magnetic properties.

- **Manganese Zinc (MnZn) ferrite:** manufactured by Steward, Inc., was used in powder form with average size around 2-micron. The MnZn ferrite [$\text{MnO}_x\text{ZnO}_y$ ($\text{Fe}_2\text{O}_3$)$_{z+y}$] is a polycrystalline compound of
  
  \[
  \text{Manganese ferrite ($\text{MnO}_x\text{Fe}_2\text{O}_3$): } 45-70\% \text{ by weight} \\
  \text{Zinc ferrite ($\text{ZnO}_x\text{Fe}_2\text{O}_3$): } 25-55\% \text{ by weight} \\
  \text{Iron ferrite ($\text{FeO}_x\text{Fe}_2\text{O}_3$): } 0-5\% \text{ by weight}
  \]

- **Iron Silicide (FeSi):** manufactured by Steward, Inc., is used as 20-micron powder. It has some good characteristics that makes it attractive for composite tagging, such as: oxidation resistant—will not rust even at temperatures over 1600°F; non-corrosive, non-erosive (abrasion resistant); chemical resistant even to strong acids; density less than that of iron; magnetic performance similar to that of iron; fire resistant; compatible with polyurethanes, fluoroelastomers, silicones, ceramics, and waterborne polymers.

Description of Specimens

Details about the sample nomenclature, tagging composition, and test type are given in Table 1. The pultruded products from Reichhold Chemicals consisted of several batches incorporating 2 tagging systems (Magnetite and NiZn ferrite), and several fiber combinations. In some specimens, defects were simulated through the inclusion of small pieces of Mylar and Nylon tape. Details of these specimens that, for brevity, are not included in Table 1, can be found in Rogers, Chen, and Giurgiutiu (1996b). The woven glass fiber fabric composites from Clark-Schwebel consisted of three batches: control, 0.3%, and 3%-lignosite FML aqueous solution weight percentage of composite. The pultruded phenolic composites from PPG Industries were tagged with 5-micron powder Lignosite FML, and had different pulling speeds and cellophane strips to simulate delaminations. The glass-fiber specimens supplied by Interplastic Corp. were tagged with iron silicide. The samples from Owens-Corning were tagged with 12-micron and 2-micron MnZn ferrite powders. The glass-fiber products supplied by TPI were tagged with NiZn ferrite. In some of these specimens, we produced two types of simulated defects: saw-cut to represent cracks, and delaminations created by driving a metal blade between the composite plies.

PASSIVE TAGGING EXPERIMENTS AND RESULTS

Approach

Eddy current testing (Blitz, 1986; Bray and Stanley, 1999) is based on the electromagnetic induction phenomenon and is traditionally applicable to non-destructive evaluation (NDE) of all electrically conducting materials, including electrically conducting fiber-reinforced plastic (FRP) composites. Composite materials may or may not be electrically conducting, depending on the basic electrical properties of their constituents. Glass-fiber composites are non-conductive and nonmagnetic. However, eddy current response of the glass-fiber composites can still be achieved after tagging them with ferromagnetic particles.

In conventional eddy current tests, when a coil carrying an alternating current (AC) is brought near an electrically conducting material, eddy currents are induced in the material by electromagnetic induction. The eddy currents produce an additional magnetic field in the vicinity of the test object. The additional magnetic field modulates the impedance of the exciting coil situated in the vicinity of the test material. The difference between the original impedance and the modulated impedance is monitored to obtain meaningful information regarding the presence of defects or changes in physical, chemical or microstructural properties.
<table>
<thead>
<tr>
<th>Company</th>
<th>Sample Name</th>
<th>Resin</th>
<th>Fiber</th>
<th>Process</th>
<th>Tagging material</th>
<th>Weight Fraction</th>
<th>Testing Type</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reichhold</td>
<td>A</td>
<td>Polyester</td>
<td>Fiberglass roving and continuous strand mat 55% vol.</td>
<td>Pultrusion</td>
<td>Control Magnetite (Fe₃O₄), &lt;5-micron</td>
<td>4.27%</td>
<td>Passive</td>
</tr>
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<td>Chemicals</td>
<td>B</td>
<td>Polyester</td>
<td>N/A</td>
<td>N/A</td>
<td>Control Magnetite (Fe₃O₄), &lt;44-micron</td>
<td>4.27%</td>
<td>Passive</td>
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<tr>
<td></td>
<td>C</td>
<td>Polyester</td>
<td>N/A</td>
<td>N/A</td>
<td>NiZn Ferrite, 2-micron</td>
<td>4.27%</td>
<td>Passive</td>
</tr>
<tr>
<td></td>
<td>D</td>
<td>Polyester</td>
<td>N/A</td>
<td>N/A</td>
<td>Control 3%-Lignosite FML</td>
<td>3.79%</td>
<td>Passive</td>
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<td>Clark-Schwebel</td>
<td>I-583-2</td>
<td>Epoxy</td>
<td>Woven fiberglass 65-70% vol.</td>
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<td>0.3%-Lignosite FML</td>
<td>0.34%</td>
<td>Passive</td>
</tr>
<tr>
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<td>I-583-5</td>
<td>Epoxy</td>
<td>N/A</td>
<td>N/A</td>
<td>Passive &amp; Passive</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>I-583-6</td>
<td>Epoxy</td>
<td>N/A</td>
<td>N/A</td>
<td>Passive &amp; Passive</td>
<td></td>
<td></td>
</tr>
<tr>
<td>PPG Industries</td>
<td>95080901</td>
<td>Phenolic</td>
<td>Fiberglass</td>
<td>Pultrusion</td>
<td>Control at 12 IPM</td>
<td>Passive</td>
<td>Passive</td>
</tr>
<tr>
<td></td>
<td>95080903</td>
<td>Phenolic</td>
<td>N/A</td>
<td>N/A</td>
<td>Control at 12 IPM</td>
<td>Passive</td>
<td>Passive</td>
</tr>
<tr>
<td></td>
<td>95080904</td>
<td>Phenolic</td>
<td>N/A</td>
<td>N/A</td>
<td>Control at 36 IPM</td>
<td>Passive</td>
<td>Passive</td>
</tr>
<tr>
<td></td>
<td>95080905</td>
<td>Phenolic</td>
<td>N/A</td>
<td>N/A</td>
<td>Control at 36 IPM</td>
<td>Passive</td>
<td>Passive</td>
</tr>
<tr>
<td></td>
<td>95080906</td>
<td>Phenolic</td>
<td>N/A</td>
<td>N/A</td>
<td>Control at 36 IPM</td>
<td>Passive</td>
<td>Passive</td>
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<tr>
<td></td>
<td>95080909</td>
<td>Phenolic</td>
<td>N/A</td>
<td>N/A</td>
<td>Control at 36 IPM</td>
<td>Passive</td>
<td>Passive</td>
</tr>
<tr>
<td>Interplastic</td>
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<td>Fiberglass 25% vol.</td>
<td>RTM</td>
<td>Iron Silicide</td>
<td>N/A</td>
<td>2%</td>
<td>Passive &amp; Passive</td>
</tr>
<tr>
<td>Owens-Corning</td>
<td>A</td>
<td>Polyester</td>
<td>Fiberglass</td>
<td>Pultrusion</td>
<td>MnZn Ferrite, 12-micron</td>
<td>N/A</td>
<td>Passive &amp; Passive</td>
</tr>
<tr>
<td></td>
<td>B</td>
<td>Polyester</td>
<td>Fiberglass</td>
<td>Pultrusion</td>
<td>MnZn Ferrite, 2-micron</td>
<td>N/A</td>
<td>Passive &amp; Passive</td>
</tr>
<tr>
<td>TPI</td>
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<td>Vinylester</td>
<td>Fiberglass 62% vol.</td>
<td>SCRMMP</td>
<td>NiZn Ferrite</td>
<td>1%</td>
<td>N/A</td>
</tr>
</tbody>
</table>

Note: N/A means "not available".

Equipment Used in the Passive Tagging Experiments

Generation and detection of eddy currents require an oscillator, a probe-coil (as a means of generating an alternating magnetic field close to the tested material), a sensing coil, and a voltmeter. Figure 1 shows a schematic drawing of the eddy current arrangement. In Figure 1, the exciting probe-coil also serves as the sensing coil, so that the voltmeter detects changes in self-inductance. Hall-type sensors can also be used and configured just as sensing coils are. They, however, require their own drive circuits. Oscillation frequency can vary from 5 Hz to 10 MHz, depending on the instrument module. Voltage measurements consist of amplitude and phase difference measurements from the exciting coil current. We used a SmartEDDY 3.0 test system consisting of:

1. IBM AT compatible host computer
2. SmartEDDY 3000 series instrument module
3. SmartEDDY 3.0 test or measurement software
4. Test and balance coil, e.g., probe, with cable

The oscillator and detection circuitry are combined into one unit plugged in a host computer. The host computer, driven by the SmartEDDY 3.0 software, controls the SmartEDDY 3000 series instrument module. The host computer reads the data gathered by the module, processes it and displays it on its monitor in graphical form. With SmartEDDY, this data may be stored in computer memory, manipulated, redisplayed, or stored on disk.

Testing Procedure for Passive Tagging Experiments

The response of the material to eddy current probing is represented in the complex (real-imaginary) reflectance plane. Figure 2 shows typical reflectance plane responses for a variety of materials. The electromagnetic signal from the eddy current response of a specimen is located in a quadrant of the complex plane depending on the electric and magnetic properties of the material. In the case of metallic materials, like the "Aluminum Plate" curve in Figure 2, the reflectance trace is placed in the first quadrant of the complex plane. Due to the presence of damage, say a saw cut,
the reflectance trace registers a marked phase shift (change in inclination) which can be easily detected with the naked eye. In the case of a conductive composite, as, for example, the "Carbon Fiber Composite" curve of Figure 2, the reflectance trace is placed in the second quadrant of the complex plane. If damage is inflicted, as for example in the form of a saw cut, the reflectance trace registers, again, a marked phase shift which can be easily detected with the naked eye. In the case of non-conducting composites tagged with ferromagnetic particles, the reflectance trace is placed in the third quadrant of the complex plane. The inclination (phase) of the reflectance curve varies with the type of tagging.

Figure 1. Schematic of the eddy current equipment used in passive tagging experiments.

Figure 2. Eddy-current reflectance response of metallic and composite materials.
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Discussion of the Passive Tagging Experiment Results

The industrial samples received from the companies have been measured with SmartEDDY 3.0 test system. All tagging particle types and concentrations were found sensitive to an eddy current probe except the 0.3%-lignosite FML tagging sample I-583-6 from Clark-Schwebel, which did not present a response due to its low tagging concentration.

The eddy current responses of samples with magnetite particles from Reichhold Chemicals presented the following characteristics. The test values of the delaminated samples are very close to those of the control samples, which implies that the delaminations could not be detected. The percentage of fiber and resin did not produce a noticeable difference in the eddy current response. The particle type and particle size made no noticeable difference in the eddy current responses of the samples with the 5-micron (sample B) and the 44-micron (sample C) magnetic particles and a small difference in the eddy current responses of samples with magneto particles (samples B and C) and samples with nickel zinc ferrite particles (sample D).

The eddy current responses of samples with lignosite FML particles from Clark-Schwebel presented the following characteristics. No difference was detected in the eddy-current responses of the untagged control sample I-583-2, and of sample I-583-6 with 0.3%-lignosite FML. However, a noticeable difference was detected for sample I-583-5 with 3%-lignosite FML.

The eddy current responses of the samples with lignosite FML particles from PPG presented the following characteristics. No difference in eddy current responses was noticed between the samples without cellophane insert (#95080905) and sample with cellophane insert (#95080906). A difference of about 0.001 volts due to pull tension speed was detected in eddy current responses of the sample #95080905 produced at 12 IPM (inch per minute) and the sample produced at 36 IPM (#95080907). No difference was detected in eddy current responses due to delamination created by cellophane film (#95080906).

The eddy current testing of samples with MnZn ferrite tagging from Owens-Corning showed that the response of the sample with 2-micron MnZn ferrite tagging is greater than that of 12-micron MnZn ferrite tagging. The eddy current response of sample with iron silicide particles from Interplastic and with NiZn ferrite tagging from TPI was satisfactory, and followed the trend of the other tagging materials described above.

In summary, the passive tagging experiments performed on ferromagnetically tagged glass fiber reinforced plastic (GFRP) specimens showed the following results:

- **Lignosite FML concentration**: No difference was detected
in the eddy-current responses of an untagged control sample 1-583-2, and of sample 1-583-6 with 0.3%-lignosote FML. However, a noticeable difference was detected for sample 1-583-5 with 3%-lignosote FML.

- **Delaminations:** No difference was detected in eddy current responses due to delamination created by cellophane film (sample 95080906).
- **Particle type:** A small difference was detected in the eddy current responses of samples with magnetite particles (samples B and C) and samples with nickel zinc ferrite particles (sample D).
- **Penetration speed:** A small difference was detected in the eddy current responses of the sample produced at 12 IPM (95080905) and the sample produced at 36 IPM (95080907).
- **Particle size:** No difference was noticed in the eddy current responses of the sample with the 5-micron (sample B) and the 22-micron (sample C) magnetite particles. A small difference was noticed in the eddy current responses of the sample with 12-micron manganese zinc ferrite particles (sample A) and the samples with 2-micron manganese zinc ferrite particles (sample B).
- **Defects:** No difference was detected in the eddy current response of control samples and samples with delaminations. However, changes in the amplitude of the eddy current response was noticeable due to saw-cut defects.

**ACTIVE TAGGING EXPERIMENTS AND RESULTS**

**Approach**

When ferromagnetic tagging particles embedded in a polymer specimen are exposed to an alternating magnetic field, the particles are driven by a magnetic excitation force (mef) and apply a distributed force on the specimen. The motion of the specimen can be described as mechanical vibration of an equivalent single-degree-of-freedom (SDOF) vibration system (Ewins, 1984; Meirovitch, 1986; Buzdugan, 1986). The vibration properties (for example, natural frequency, damping, etc.) of the specimen subjected magnetic excitation are expected to indicate and interpret the condition of quality of the specimen. Vibration measurements of a tagged specimen subjected a magnetic excitation were used to validate the previously derived theoretical model. In these experiments, we are concerned with the mechanical properties of the tagged composite and with the interaction between the tagging particles and the polymer matrix. The relationship between the response of the tagging particles and the applied magnetic excitation force is experimentally investigated using vibration measurement analysis techniques. For example, the vibrations of a specimen subjected to harmonic magnetic excitation are measured, and the response characteristics of the system under test, i.e., the frequency response and the phase angle response, are determined.

**Theoretical Model**

A theoretical model for active tagging interrogation of composite materials was developed by Rogers et al. (1995) based on the single-degree-of-freedom (SDOF) mass-spring system description of the motion of the tagging particle embedded in the composite under the action of a magnetic excitation force (mef), \(F_{m}t\). Assuming that all particles move in phase, the overall mef applied to the composite could be obtained by summation over all the particles

\[
F_m(t) = \mu_0 \alpha V \frac{a}{2} \left[ \frac{1}{(L - Z)^2} - \frac{1}{Z^2} \right] 
\times (C_m^2 + 2C_s^2 + 4C_m C_s \sin \omega t - C_m^2 \cos 2\omega t)
\]  

(1)

where \(\mu_0 = 4\pi \times 10^{-7}\) H/m is the permeability of free space; \(\alpha\) is a coefficient that depends on the shape of the particle (e.g., for the sphere \(\alpha = 3\)); \(L\) is the distance between the poles; \(Z\) is the average distance from the particle to the N-pole of the magnetic yolk; \(\omega\) is the excitation frequency; \(C_m\) and \(C_s\) are simplified coefficients; \(V\) is the volume of the tagged polymer; \(\Delta w\) is the weight ratio of particles; and \(q\) and \(q_e\) are the mass density of the matrix and the particles, respectively. We developed an extension of Rogers et al. (1995) model by noticing that, in the case of particles embedded in a massless polymeric matrix, the motion of the particles can be equivalent to that of a single-degree-of-freedom system when the structure vibrates in one of its eigen mode of vibration. Then, the governing equation of the equivalent system can be rewritten in the frequency domain:

\[-\omega^2 m X + K X = F(\omega)\]  

(2)

where \(m\) is the generalized modal mass of the tagging particles; \(K = K' (1 + j\eta)\) is the generalized modal stiffness of the system; and \(\eta\) is the modal damping of the system. The generalized force, \(F(\omega)\), is defined by the relation:

\[F(\omega) = F_m(\omega) \int \psi_i(\vec{r}) ds\]  

(3)

where \(\psi_i(\vec{r})\) is the mode shape of the structure and \(F_m(\omega)\) is the Fourier transform of \(F_m(t)\). Substituting Equation (1) into Equation (3) yields:

\[F(\omega) = [F_m(\delta(\omega - \omega_n) + F_m(\delta(\omega - 2\omega_n))] \int \psi_i(\vec{r}) ds\]  

(4)

where
\[ F_{m1} = 4 \mu_0 \alpha V \Delta w \frac{G}{\varepsilon_0} \left[ \frac{1}{(L - Z)^5} - \frac{1}{Z^5} \right] C_m C_s \]

\[ F_{m2} = \mu_0 \alpha V \Delta w \frac{G}{\varepsilon_0} \left[ \frac{1}{(L - Z)^5} - \frac{1}{Z^5} \right] C_m^2 \]

and \( \delta \) is Dirac's \( \delta \)-function. The solution of Equation (2) is:

\[ X = \frac{F(\omega)}{K'} \left[ \left( 1 - \frac{\omega^2}{\omega_t^2} \right) - j\eta \left[ \left( 1 - \frac{\omega^2}{\omega_t^2} \right)^2 + \eta^2 \right] \right]^{-1} \]

where

\[ \omega_t = \frac{K'}{m} = \frac{M + m}{m} \omega_t \]

is the frequency of the equivalent system, and \( \omega_t \) is the frequency of the original system. When the mass of the polymeric matrix is neglected, the interactive force between the matrix and the particle can be presented by:

\[ Q(\omega) = KX = F(\omega) \left[ \left( 1 - \frac{\omega^2}{\omega_t^2} \right) - j\eta \left[ \left( 1 - \frac{\omega^2}{\omega_t^2} \right)^2 + \eta^2 \right] \right]^{-1} \]

Substituting Equation (4) in Equation (7) yields:

\[ Q(\omega) = Q_{01} \delta(\omega - \omega_s) + Q_{02} \delta(\omega - 2\omega_s) \]

where

\[ Q_{01} = F_{m1} \left( 1 - \frac{\omega_s^2}{\omega_t^2} \frac{1}{\Delta \omega} \right) \left[ \left( 1 - \frac{\omega^2}{\omega_t^2} \frac{1}{\Delta \omega} \right)^2 + \eta^2 \right]^{-1} \times \int_s \psi_s(r) ds \]

is the amplitude of the sin (\( \omega t \)) signal, and

\[ Q_{02} = F_{m2} \left( 1 - \frac{4 \omega_s^2}{\omega_t^2} \frac{1}{\Delta \omega} \right) \left[ \left( 1 - \frac{4 \omega_s^2}{\omega_t^2} \frac{1}{\Delta \omega} \right)^2 + \eta^2 \right]^{-1} \times \int_s \psi_s(r) ds \]

is the amplitude of the cos (2\( \omega t \)) signal. It should be noted that, in Equation (8), the interactive force has two components of different frequencies. The first component, of frequency \( \omega_s \), is the major component in the particle excitation, in which the static bias field has the same contribution as the alternating field shown in Equation (4). The second component, of the double frequency 2\( \omega_s \), is proportional to the square of the alternating excitation field and could be the cause of serious distortion of the excitation signature. Further details about this theoretical model are given in Rogers, Chen, and Giurgiutiu (1996b).

**Equipment Used in the Active Tagging Experiments**

The equipment required to perform the active tagging experiments consists of three main components: exciting equipment, measuring equipment, and signal processing equipment. Figure 4 shows an illustration of the equipment used in these experiments. Figure 5 shows a schematic drawing of the experimental set-up. Table 2 gives a list of the main equipment components for the active tagging experiments. The exciting equipment includes a magnetic exciter, a generator, filters and power amplifiers. The magnetic exciter was constructed at CIMSS and consists of a high-permeability silicon steel yoke and two energizing coils. One coil is driven by an AC power amplifier (Crown CT-400) to generate an alternative magnetic field, while the other coil is powered by a DC current source (HP 6268B) to establish a constant magnetic field (Figure 3). Details about the construction of the magnetic exciter can be found in Rogers, Chen, and Giurgiutiu (1996b). The ferromagnetic yoke is used as a concentrator of magnetic flux lines to create a relatively uniform excitation field. It is assumed that the yoke material is linear and isotropic. The specimen is bolted to a piezoelectric force gage (PCB 28R850) whose housing is made of 300 series stainless steel which does not respond to magnetic fields. The force gage signal was captured through a charge-amplifier signal conditioner. The force gage assembly sits on a stiff plastic plate, where the first natural frequency lies beyond 10000 Hz. It can be assumed that the base of the specimen is essentially stationary when excited. The Hall probe of an MG-5D Walker Scientific Gaussmeter, placed in the magnetic yoke near the specimen, measures the magnetic flux density of the excitation field. A frequency analyzer (Zonic WCA) is employed to process the test data and to obtain the frequency response function (FRF).

**Testing Procedure for Active Tagging Experiments**

Rectangular specimens of approximate size 1.5 \( \times \) 1.25 in\(^2\) (38 \( \times \) 32 mm\(^2\)) were cut from the industrial samples. The rectangular specimens were mounted on top of the miniature force gage using a fixing bolt [Figure 4(b)]. Two excitation types are routinely used to determine the natural frequencies of a system within a specified frequency range: harmonic excitation sweep, and broad-band excitation. Both types of excitation were used in our test. The excitation magnetic field is generated by an alternating current (AC) and direct current (DC) in the different solenoidal coils of the electromagnet. The DC coils create a static bias flux density which pre-magnetizes the particles and maximizes.
the excitation response, thus improving the signal-to-noise ratio. It also suppresses the nonlinear second-order frequency component. The AC coils create an alternating magnetic flux density to produce vibrations of the particles. The alternating component is frequency-dependent as it is proportional to the energizing current. The effective impedance of the coil increases with frequency. Our experiments were conducted under constant voltage excitation, and hence the excitation current decreases with frequency. The input data of the experiment was the electromagnetic excitation field. This was measured with the Hall probe of the Gaussmeter which was placed in the proximity of the specimen. The output data of the experiment was the force response measured with the force gage. According to Equation (7), the force gage signal, \( Q(\omega) \), is proportional to the magnetic excitation force, \( F(\omega) \), applied on the tagging particles.

After conversion and conditioning of the signal, the data could be either displayed, using oscilloscopes, or recorded, i.e., stored on computer disk for signal post processing. Both input and output signals was passed to the frequency analyzer. Processing of these signals yielded frequency response curves. From the frequency response curves, the inherent characteristics of the specimen, the natural frequencies and damping ratios, were obtained. The equipment used in our experiment contained the signal generator, the filter and the Fast-Fourier-Transform (FFT) analyzer combined as a unit in the specialized computer.

**Discussion of the Active Tagging Results**

Figure 6 presents the frequency response function (FRF) for specimen I-583-5 showing that the presence of simulated

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**Table 2. Equipment list for active tagging experiments.**

<table>
<thead>
<tr>
<th>Name</th>
<th>Brand</th>
<th>Model</th>
<th>Manufacturer</th>
</tr>
</thead>
<tbody>
<tr>
<td>Analyzer</td>
<td>ZONIC + AND</td>
<td>4000</td>
<td>Zonic A &amp; D Company</td>
</tr>
<tr>
<td>DC power supply</td>
<td>Hewlett-Packard</td>
<td>HP6268B</td>
<td>Hewlett-Packard Company</td>
</tr>
<tr>
<td>Gaussmeter</td>
<td>Walker</td>
<td>MG-5D</td>
<td>Walker Scientific, Inc.</td>
</tr>
<tr>
<td>Power unit</td>
<td>PCB</td>
<td>480C08</td>
<td>PCB Piezotronics, Inc.</td>
</tr>
<tr>
<td>Force gage</td>
<td>PCB</td>
<td>200M100</td>
<td>PCB Piezotronics, Inc.</td>
</tr>
<tr>
<td>Oscilloscope</td>
<td>JDR</td>
<td>2000</td>
<td>Korea</td>
</tr>
<tr>
<td>Magnetic exciter</td>
<td>N/A</td>
<td>N/A</td>
<td>CIMSS</td>
</tr>
</tbody>
</table>
Figure 4. Illustration of the equipment used in the active tagging experiments: (a) overall set-up and (b) detail of the specimen mounted on the force gage.

Figure 5. Schematic diagram of the active tagging experimental configuration.
defects can create significant changes in the shape of the FRF and in the location of its peaks. Similar behavior was observed for the other specimens considered in the study (Rogers et al., 1996a and 1996b). From the analysis of the FRF curves, the natural frequencies and damping ratio was extracted. A synopsis of the natural frequencies and the damping ratios obtained from the analysis of all the specimens is given in Table 3. Note that, in general, the natural frequency may depend on the specimen thickness, shape and dimensions, and on the material stiffness. For specimens with the same geometry made from the same material, differences in frequency and damping may occur due to the presence of defects. For example, consider the specimen obtained from Owens-Corning sample A (MnZn ferrite tagging) shown in the sixth row of Table 3. The natural frequency of the original sample was 2.1375 kHz, and the damping ratio was 0.099. A similar specimen with a saw cut had a natural frequency of 1.875 kHz, and a damping ratio of 0.100, whereas a specimen with a delamination had 2.0125 kHz, and 0.47, respectively. Another example is that of the specimen obtained from sample B-3 from Reichhold Chemicals (magnetite Fe₃O₄ tagging) shown in the first row of Table 3. The natural frequency of the original sample was 1.71 kHz, and the damping ratio was 0.077. The specimen with a saw cut had 1.67 kHz and 0.071, whereas the specimen with a delamination had 1.66 kHz and 0.052, respectively.

In some cases, the damping ratio could not be determined because of the close proximity of the specimen resonance peaks that did not allow proper determination of the half-power points. This happens for sample I-583-5 from Clark-Schwebel (3%-lignosite tagging), shown in the fourth row of Table 3. This sample presents a natural frequency of 3.5 kHz, for the control specimen, 3.3875 kHz, for the specimen with a saw-cut, and 3.2125 kHz for the specimen with a delamination. However, the damping ratio could only be determined for the specimen with a delamination. Similar difficulties in determining the damping ratio due to the proximity of the resonance peaks were encountered for the specimens cut from sample #95080905 (PPG, lignosite FML tagging).

The active tagging method was found to be sensitive enough to detect simulated defects, such as saw-cuts and delaminations. To differentiate between saw-cuts and delaminations in a real-life random experiment, further refinement of the technique and of the experimental equipment is required. Carefully conducted calibration and training experiments will also be necessary to achieve confidence in the experimental method.

All ferromagnetic particles that have been tested can be candidates for the active tagging method. Perhaps the most important aspect of selection of the tagging particles that needs attention is the impact of ferromagnetic particles on the long-term properties of the matrix materials. Although it is intended that small amounts of metal oxide particles be used in order to minimize potential interactions with the matrix, long-term performance testing is required to confirm this assumption. In addition, an important aspect of ferromagnetic particle tagging is the ability to impart slight electromagnetic properties to a normally nonmagnetic, nonconducting material. Tests to date have shown that this modification does not have a detrimental impact on the material. For example, the addition of ferromagnetic particles does not appear to affect the electrical insulating prop-
erities of a normally non-conducting material, and adding small amounts of electromagnetic particles to a non-magnetic material does not produce significant changes in its magnetic properties. In fact, a specific advantage of using very small ferromagnetic particles for tagging relates to the fact that the particles are smaller than a magnetic domain (a small region in the material in which atomic dipole moments are all aligned in one direction) and cannot become permanently magnetized as a result of electromagnetic testing. It is generally accepted that a higher weight fraction of particles would yield a stronger frequency response. However, this could be detrimental to the structural strength of the composite. We found that specimens with low levels of tagging, say, 5% by weight of composite, could be actuated satisfactorily by the magnetic field in our laboratory tests. In practice, whether or not a tagged specimen could be effectively actuated depends not only on the tagging level in the material but also on the specimen dimensions and local geometry.

CONCLUSIONS

This study has shown useful experimental conclusions regarding the use of passive and active interrogation method for in-field and in-service inspection and monitoring of civil engineering composite materials tagged with ferromagnetic microscopic particles. Under the sponsorship of U.S. Army Construction Engineering Research Laboratory, a number of glass-fiber reinforced plastic (GFRP) composite specimens, tagged with various ferromagnetic particles, were manufactured by some member firms of the Composite Institute of the Society of Plastics Industries and tested in the Center for Intelligent Material Systems and Structures at Virginia Polytechnic Institute and State University. The main conclusions resulting from these tests are presented next.

It was found that all the tagging materials used in the fabrication of the industrial specimens (magnetite, Nickel Zinc ferrite, lignosite FML, Manganese Zinc ferrite, and Iron Disilicide), even when used in relatively small concentrations (3–5% w/w), imparted detectable ferromagnetic properties to the tagged composite. However, when the concentration of tagging material was too small, detection was no longer possible, as in the case of 0.3% w/w lignosite tagging of specimen 1-583-6.

The passive tagging experiments showed that a clear eddy current response is detectable in the ferromagnetically tagged GFRP composites, but the phase relationship of the complex reflectance curve was clearly different from that observed in the conventional eddy-current investigation of metallic materials. For metallic materials, the complex reflectance curve lies in the first quadrant of the complex plane. In the case of ferromagnetically tagged GFRP composites, the complex reflectance curve was found to lie in the third quadrant of the complex plane.

The capability of eddy current method to detect cracks and delaminations in tagged GFRP composites was also investigated. In the case of metallic materials, the presence of cracks and internal defects leads to a clear phase shift in the reflectance curve. In our experiments, such a phase shift in the reflectance curve due to the presence of simulated cracks

Table 3. Test results of the active tagging experiments of industrial composite specimens.

<table>
<thead>
<tr>
<th>Company</th>
<th>Samples</th>
<th>Condition</th>
<th>Frequency (kHz)</th>
<th>Normalized Frequency</th>
<th>Damping</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reichhold</td>
<td>B-3</td>
<td>Control</td>
<td>1.7125</td>
<td>100%</td>
<td>0.077</td>
</tr>
<tr>
<td></td>
<td>Fe₂O₃, &lt;5-micron</td>
<td>Saw cut</td>
<td>1.6750</td>
<td>98%</td>
<td>0.071</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Delaminated</td>
<td>1.6625</td>
<td>97.1%</td>
<td>0.052</td>
</tr>
<tr>
<td>PPG</td>
<td>95080905</td>
<td>Control</td>
<td>3.7875</td>
<td>100%</td>
<td>N/A</td>
</tr>
<tr>
<td></td>
<td>Lignosite FML</td>
<td>Saw cut</td>
<td>3.5875</td>
<td>94.7%</td>
<td>N/A</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Delaminated</td>
<td>3.2500</td>
<td>85.8%</td>
<td>0.058</td>
</tr>
<tr>
<td>Clark-Schwebel</td>
<td>1-583-5</td>
<td>Control</td>
<td>3.5000</td>
<td>100%</td>
<td>N/A</td>
</tr>
<tr>
<td></td>
<td>9%-Lignosite FML</td>
<td>Saw cut</td>
<td>3.3875</td>
<td>96.8%</td>
<td>N/A</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Delaminated</td>
<td>3.2125</td>
<td>91.8%</td>
<td>0.060</td>
</tr>
<tr>
<td>Interplastic</td>
<td>Iron silicide</td>
<td>Control</td>
<td>1.6063</td>
<td>100%</td>
<td>0.065</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Saw cut</td>
<td>1.6750</td>
<td>104.3%</td>
<td>0.140</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Delaminated</td>
<td>1.6560</td>
<td>103.1%</td>
<td>0.110</td>
</tr>
<tr>
<td>Owens-Corning</td>
<td>A</td>
<td>Control</td>
<td>2.1375</td>
<td>100%</td>
<td>0.999</td>
</tr>
<tr>
<td></td>
<td>MnZn ferrite</td>
<td>Saw cut</td>
<td>1.8750</td>
<td>87.71%</td>
<td>0.103</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Delaminated</td>
<td>2.0125</td>
<td>94.19%</td>
<td>0.047</td>
</tr>
<tr>
<td>TPI</td>
<td>NiZn ferrite</td>
<td>Control</td>
<td>0.9688</td>
<td>100%</td>
<td>0.180</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Saw cut</td>
<td>0.9063</td>
<td>93.5%</td>
<td>0.200</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Delaminated</td>
<td>0.9563</td>
<td>98.7%</td>
<td>0.176</td>
</tr>
</tbody>
</table>
(saw cuts) could not be detected. The reason for this difference in behavior lies in the fact that metals are good electric conductors, while ferromagnetically tagged GFRP composites, though responsive to magnetic fields, do not conduct electric currents. However, it was found that the presence of simulated cracks (saw cuts) yields a reduction in the amplitude of eddy current response. This response-amplitude reduction could be utilized to detect cracks, but it would require a different approach to eddy-current interrogation and interpretation methods than those currently used in the conventional eddy-current test system. The same token, delaminations in the ferromagnetically tagged GFRP composite specimens were performed using both harmonic frequency sweep and broadband frequency excitations via the magnetic field created in the air gap of a laboratory electromagnet. Detection of the structural vibration response, done with a miniature force gage fixed to the specimen, and frequency analysis of the data permitted the display of the frequency response curves for the specimens. It was found that the frequency response signature of the specimens changed significantly when simulated defects (saw cuts and delaminations) were introduced in the structure of the specimen. Modifications in natural frequencies and in the amplitude and shape of the frequency-response curves were observed due to the changes in the local stiffness and damping characteristics induced in the specimen by the presence of simulated defects. The measured frequency response parameters (natural frequency and damping ratio) were found to be generally independent of the loading conditions and the excitation type. In conclusion, the active tagging experiments performed on the ferromagnetically tagged GFRP composite specimens were significantly more conclusive regarding the possibility for detection of structural and material defects than the passive tagging experiments. The active tagging method showed good prospects for full-scale implementation, and has potential for development into a reliable non-destructive evaluation and inspection method for in-process and in-field non-destructive evaluation of tagged glass-fiber polymeric composites.

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