1996 SPIE Symposium on Smart Structures and Materials, Smart Structures and Int ed Systems Conference, 26-29 February 1996 San Diego, California, Pap #2717-31

PASSIVE AND ACTIVE TAGGING OF REINFORCED 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 presents. 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 known 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 out of composite materials is developing at a fast rate. Out of the ten 1995 awards of the Civil Engineering Research Foundation[14], 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 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 wide spread 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[1,2,3] have been performed to assess this technology and to find the most promising methods for in-service and in-field implementation.

In this paper, this 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 was varied and covered 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. 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

Report Documentation Page		*Form Approved* *OMB No. 0704-0188*

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1. REPORT DATE **1996**	2. REPORT TYPE **N/A**	3. DATES COVERED **-**
4. TITLE AND SUBTITLE **Passive And Active Tagging Of Reinforced Composites For In Process And Infield Non-Destructive Evaluation**		5a. CONTRACT NUMBER 5b. GRANT NUMBER 5c. PROGRAM ELEMENT NUMBER
6. AUTHOR(S)		5d. PROJECT NUMBER 5e. TASK NUMBER 5f. WORK UNIT NUMBER
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) **Virginia Polytechnic Institute and State University, Blacksburg, VA 24061-0261, USA**		8. PERFORMING ORGANIZATION REPORT NUMBER
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)		10. SPONSOR/MONITOR'S ACRONYM(S) 11. SPONSOR/MONITOR'S REPORT NUMBER(S)

12. DISTRIBUTION/AVAILABILITY STATEMENT
Approved for public release, distribution unlimited

13. SUPPLEMENTARY NOTES

14. ABSTRACT

15. SUBJECT TERMS

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
a. REPORT **unclassified**	b. ABSTRACT **unclassified**	c. THIS PAGE **unclassified**	**UU**	**12**	

Standard Form 298 (Rev. 8-98)
Prescribed by ANSI Std Z39-18

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 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): manufactured by Steward, Inc., is used in powder form with sizes between 5-micron and 44-micron, average 22-micron.
- Nickel Zinc (NiZn) ferrite: manufactured by Steward, Inc., is used in powder form with average size 2-micron.
- Lignosite FML : manufactured by Georgia-Pacific Corp., is 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 characteristic 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., is used in powder form with average size around 2-micron. The MnZn ferrite $\{MnO_xZnO_y(Fe_2O_2)_{1-x-y}\}$, is a polycrystalline compound of

  Manganese ferrite: $MnO\text{-}Fe_2O_3$ 45-70% by weight
  Zinc ferrite: $ZnO\text{-}Fe_2O_3$ 25-55% by weight
  Iron ferrite: $FeO\text{-}Fe_2O_3$ 0-5% 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 1600^0F; 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, fluroelastomers, silicones, ceramics, and water borne polymers.

Description of Specimens

Details about the sample nomenclature, tagging composition, and test status are given in Table 1. The pultruded products from Reichhold Chemicals consisted of several batches incorporating 2 tagging systems (Magnetite and NiZn ferrite), 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 include in Table 1, can be found in ref. 13. The woven glass fiber fabric composites from Clark-Schwebel consisted of three batches: control, 0.3%, and 3%-lignosite FML aqueous solution weight % 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-cuts to represent cracks, and delaminations created by driving a metal blade between the composite plies.

Table 1 Synopsis of industrial samples used in passive and active tagging experiments.

Company	Sample name	Resin	Fiber	Process	Tagging material	Tagging weight fraction		Testing type
						in composite	in resin	
Reichhold Chemicals	A	Polyester	Fiberglass roving and continuous strand mat 55% vol.	Pultrusion	Control			Passive
	B				Magnetite (Fe_3O_4), <5-micron	4.27%	4.76%	Active & Passive
	C				Magnetite (Fe_3O_4), <44-micron	4.27%	4.76%	Passive
	D				NiZn Ferrite, 2-micron	4.27%	4.76%	Passive
Clark-Schwebel	I-583-2	Epoxy	woven fiberglass 65-70% vol.	N/A	Control			Passive
	I-583-5				3%-Lignosite FML	3.79%	N/A	Active & Passive
	I-583-6				0.3%-Lignosite FML	0.34%	N/A	Passive
PPG Industries	95080901	Phenolic	Fiberglass	Pultrusion	Control at 12 IPM			Passive
	95080903				Control w/Cellophane at 12 IPM			Passive
	95080904				Control at 36 IPM			Passive
	95080905				Lignosite FML at 12 IPM	N/A	5%	Active & Passive
	95080906				Lignosite FML with Cellophane at 12 IP	N/A	5%	Passive
	95080907				Lignosite FML at 36 IPM	N/A	5%	Passive
Interplastic		Vinylester	Fiberglass 25% vol.	RTM	Iron Silicide	N/A	2%	Active & Passive
Owens Corning	A	Polyester	Fiberglass	Pultrusion	MnZn Ferrite, 12-micron	N/A	N/A	Active & Passive
	B				MnZn Ferrite, 2-micron	N/A	N/A	Passive
TPI	N/A	Vinylester	Fiberglass 62% vol.	SCRIMP	NiZn Ferrite	1%	N/A	Active & Passive

Note: N/A means "not available"

PASSIVE TAGGING EXPERIMENTS AND RESULTS

Approach

Eddy current testing 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 conductive, depending on the basic electrical properties of their constituents. Glass-fiber composites received from the participating companies 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. These eddy currents, in turn, produce an additional AC magnetic field in the vicinity of the test object. The induced eddy currents modulate the impedance of the exciting coil situated in the vicinity of the test material. The difference between the original coil impedance and the modulated coil impedance (due to the presence of eddy currents) is monitored to obtain meaningful information regarding the presence of defects or changes in physical, chemical or microstructure properties.

Required Equipment for 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.

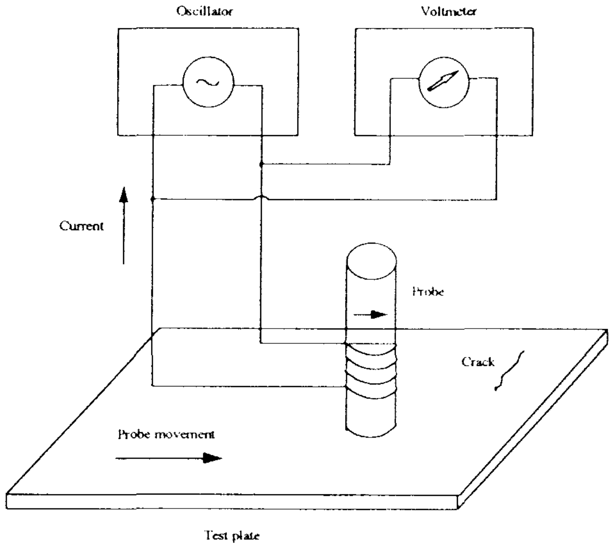


Figure 1 Eddy current equipment used in passive tagging experiments

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 nonconducting 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 materials used, and this aspect can be well noticed in Figure 2 where the curves corresponding to lignosite FML, MnZn ferrite and NiZn ferrite , Fe_3O_4, and iron silicide are all different. However, the presence of a simulated defect (saw cut) does not produce a phase shift in the eddy current response.

For electrically conducting composites, the eddy current technique can be usefully employed for quantification and location of defects the conventional techniques developed for metallic materials. However, for insulating composites tagged with ferromagnetic particles, the eddy current technique is not readily applicable, as illustrated in Figure 2. In this case, an alternative route is that of the flux leakage method. Our experiments have shown that electromagnetic signal of the flaw leakage field can be detected with an eddy current probe for the saw-cut defects, but delaminations are not effectively detected. When a defect or any other kind of discontinuity occurs in the test object, the magnetizing field is diverted in a manner characteristic of the nature of the discontinuity, and changes take place in the impedance of the probe-coil. It has been found that a probe scanning a surface is not able to assess defects oriented in the same direction as the magnetizing field by moving the probe from a position on the test object where there is no defect to another position where a planar defect (say, a delamination) is present.

If the matrix material of the specimens is non-conductive and nonmagnetic, the eddy current response is independent of the matrix and is a direct function of the amount of tagging. In this case, only the amplitude of the eddy current response is sensitive to the cracks (mass loss effect) while the phase is not changed at all. The passive tagging method using eddy current testing is effective in inspecting the presence, the amount, and the distribution of the particles. When the computer-aided eddy current probe scans the surface along the sample, the eddy current response varies due to the presence and density of particles. Without these particles, there is no eddy current response at all. The higher the fraction of particles, the stronger is the eddy current response. A higher drive voltage level yields a stronger eddy current response, too. A saw-cut defect, for example, can be assessed by measuring the changes in the impedance of the coil while moving a probe from a position on the test object where there is no defect to one where a crack is present. For insulating composites, these changes take place in the amplitude of the coil impedance and not in its phase. Since a reduction in the amplitude of the response without a phase shift is more difficult to observe, and since external perturbing factors (this could be a probe wobbling caused by surface condition of a test specimen, uniformly distribution of particles and thermal drift) may also affect the response amplitude, the use of this method for non-conducting composites is much more difficult than for conductive composites or for metals. Eddy current equipment can be calibrated for the size of the defect using saw-cuts of various depths that are cut in blocks fabricated from the same material as the material of the sample. However, difficulties may arise since it is very rare that a real crack bears resemblance to a uniform saw-cut.

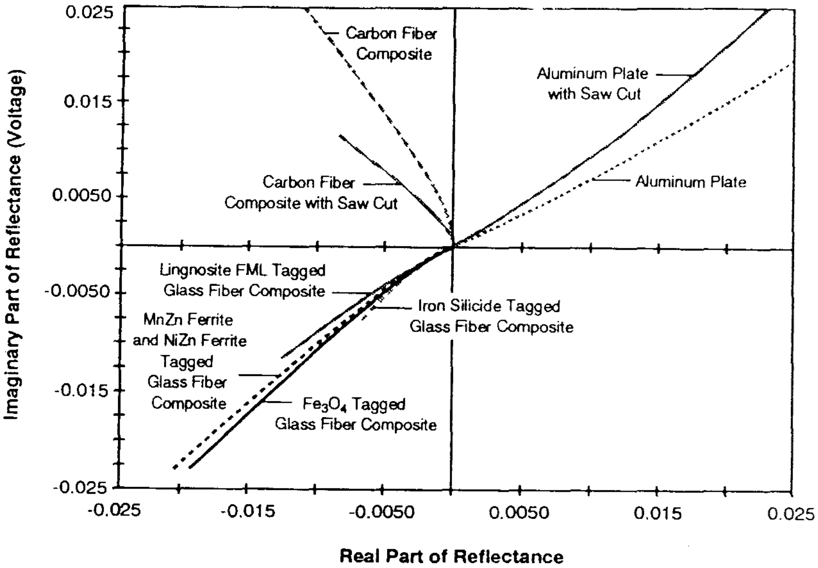


Figure 2 **Eddy-current reflectance response of metallic and composite materials**

Discussion of the Passive Tagging 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 closed 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) magnetite particles and a small difference in the eddy current responses of samples with magnetite 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 an 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 sample without cellophane insert (# 95080905) and sample with cellophane insert (# 95080906). A difference of about 0.001 Volts due to pultrusion 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.

Summary of the Passive Tagging Testing Results for Industrial Specimens

- Lignosite FML concentration: No difference was detected in the eddy-current responses of an 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.
- 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).
- Pultrusion 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 samples 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 samples 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 magnetomotive force (mmf) and apply a distributed force on the specimen. The motion of the specimen is described as mechanical vibration and is equivalent to a single-degree of-freedom (SDOF) vibration of system[1]. 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 magnetomotive 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.*[13] based on the single-degree-of-freedom (SDOF) mass-complex-spring system description of the motion of the tagging particle embedded in the composite under the action of a magnetomotive force (mmf), F_{mi}: Assuming that all the particles move in phase, the overall mmf applied to the composite could be obtained by summation over all the particles

$$F_m(t) = \mu_0 \alpha \; V \Delta w \frac{\rho}{\rho_p}\left[\frac{1}{(L-\bar{Z})^5} - \frac{1}{\bar{Z}^5}\right](C_m^2 + 2C_s^2 + 4C_m C_s \sin\omega t - C_m^2 \cos 2\omega t), \tag{1}$$

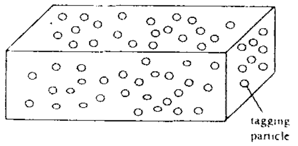
where $\mu_0 = 4\pi \times 10^{-7}$ H / m is the permeability in free space; α is a coefficient that depends on the shape of the particle (e.g., for the sphere $\alpha = 3$); L is the distance between the poles; $\bar{Z}$ is the average distance from the particle to the N-pole of the magnetic yoke; ω is the excitation frequency C_m and C_s are simplified coefficients: V is the volume of the tagged polymer; Δw is the weight ratio of particles; and ρ and ρ_p are the mass density of the polymer and of the particles, respectively. We developed an extension to Rogers[1] 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:

$$-\omega^2 mX + KX = F(\omega), \tag{2}$$

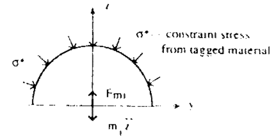
where m is the generalized modal mass of the tagging particles; $K = K'(1 + j\eta)$ is the generalized modal stiffness of the system; and η is the modal damping of the system. The generalized force, $F(\omega)$, is defined from the relation:

$$F(\omega) = F_m(\omega)\int_S \psi_i(\vec{r})ds, \tag{3}$$

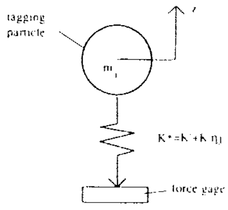
where $\psi_i(\vec{r})$ is the mode shape of the structure and $F_m(\omega)$ is the Fourier transform of $F_m(t)$ of Equation (3). Substituting Equation (1) in Equation (3) yields:



(a) A tagged specimen



(b) Forces applied on a particle



(c) A dynamic model of a particle

Figure 3 Schematics of the active tagging principles.

$$F(\omega) = [F_{m1}\delta(\omega - \omega_n) + F_{m2}\delta(\omega - 2\omega_n)]\int_S \psi_i(\vec{r})ds, \tag{4}$$

where

$$F_{m1} = 4\mu_0 \alpha V \Delta w \frac{\rho}{\rho_p}\left[\frac{1}{(L-\bar{Z})^5} - \frac{1}{\bar{Z}^5}\right]C_m C_s, \qquad F_{m2} = \mu_0 \alpha \; V \Delta w \frac{\rho}{\rho_p}\left[\frac{1}{(L-\bar{Z})^5} - \frac{1}{\bar{Z}^5}\right]C_m^2, \tag{5}$$

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

$$X = \frac{F(\omega)}{K'}\left[(1 - \frac{\omega^2}{\hat{\omega}_i^2}) - j\eta\right]\left[(1 - \frac{\omega^2}{\hat{\omega}_i^2})^2 + \eta^2\right]^{-1}, \tag{6}$$

where $\hat{\omega}_i^2 = \dfrac{K'}{m} = \dfrac{M+m}{m}\omega_i^2$ is the frequency of the equivalent system; and ω_i 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 represented by:

$$Q(\omega) = KX = F(\omega)\left[(1-\frac{\omega^2}{\hat{\omega}_i^2}) - j\eta\right]\left[(1-\frac{\omega^2}{\hat{\omega}_i^2})^2 + \eta^2\right]^{-1} . \tag{7}$$

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

$$Q(\omega) = Q_{01}\delta(\omega - \omega_n) + Q_{02}\delta(\omega - 2\omega_n) , \tag{8}$$

where

$$Q_{01} = F_{m1}\left(1 - \frac{\omega_n^2}{\omega_i^2}\frac{1}{\Delta\omega}\right)\left[(1-\frac{\omega_n^2}{\omega_i^2}\frac{1}{\Delta\omega})^2 + \eta^2\right]^{-1} \int_s \psi_r(\bar{r})ds \tag{9}$$

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

$$Q_{02} = F_{m2}\left[1 - \frac{4\omega_n^2}{\omega_i^2}\frac{1}{\Delta\omega}\right]\left[(1-\frac{4\omega_n^2}{\omega_i^2}\frac{1}{\Delta\omega})^2 + \eta^2\right]^{-1} \int_s \psi_r(\bar{r})ds \tag{10}$$

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 ω, 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 2ω, 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 ref. 13.

Required Equipment for 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 3 shows an illustration of the equipment used in these experiments. Figure 4 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 alternating 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 ref. 13. 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 208M100) whose housing is made of 300 series stainless steel which does not respond to magnetic fields. The force

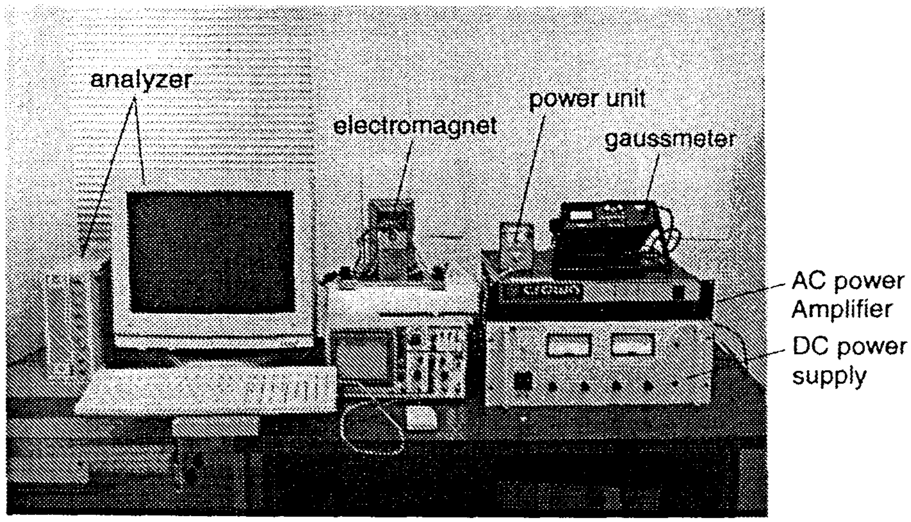


Figure 4 Illustration of the equipment used in the active tagging experiments

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 10,000 Hz. It can be assumed that the base of the specimen is essentially stationary when excited. The Hall probe of a 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).

Table 2. Equipment list for active tagging experiments

Name	Brand	Model	Manufacturer
Analyzer	ZONIC+AND	4000	Zonic A & D Company
Power amplifier	Com-Tech	400	Crown International, Inc.
DC power supply	Hewlett-Packard	HP6268B	Hewlett-Packard Company
Gaussmeter	Walker	MG-5D	Walker Scientific, Inc.
Power unit	PCB	480C06	PCB Piezotronics, Inc.
Force gage	PCB	208M100	PCB Piezotronics, Inc.
Oscilloscope	JDR	2000	Korea
Magnetic exciter	N/A	N/A	CIMSS

Specimen Testing Procedure

Rectangular specimens of approximate size $1\frac{1}{2}\times1\frac{1}{4}$ in^2 were cut from the industrial samples. The rectangular specimens were mounted on top of the miniature force gage using a fixing bolt (Figures 4). 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 the 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 (10), the force gage signal, $Q(\omega)$, is proportional to the magnetomotive 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.

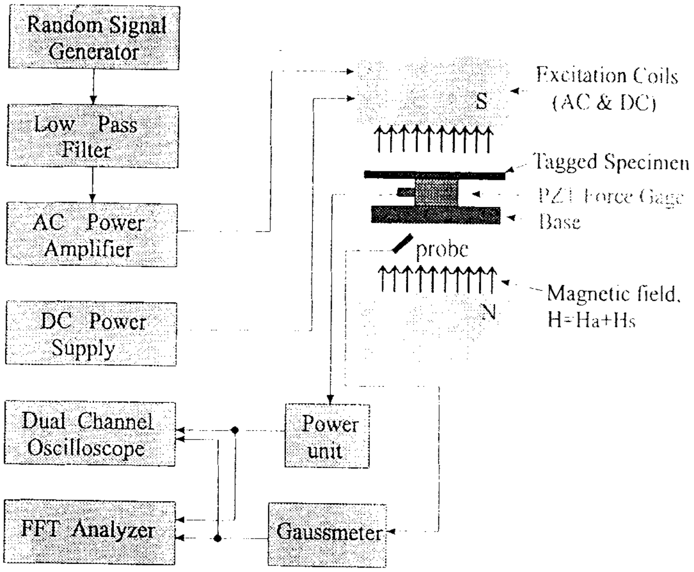


Figure 5 **Schematic diagram of the active tagging experimental configuration.**

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 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[13]. 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 Fe_3O_4 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.

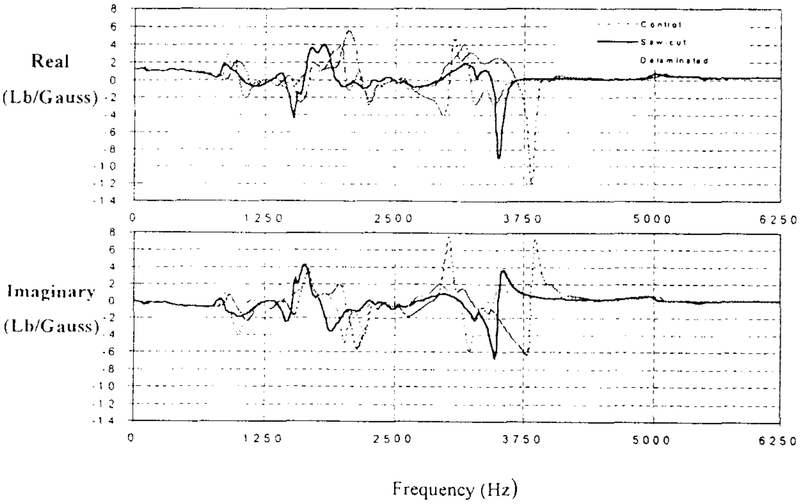


Figure 6 Frequency response functions of Owens-Corning sample A (MnZn ferrite tagging) showing sensitivity to the presence of simulated cracks (saw-cuts) and delaminations.

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

Company	Samples	Condition	Frequency (kHz)	Normalized Frequency	Damping
Reichhold	B-3 Fe_3O_4,<5-micron	control	1.7125	100%	0.077
		saw cut	1.6750	98%	0.071
		delaminated	1.6625	97.1%	0.052
PPG	95080905 Lignosite FML	control	3.7875	100%	N/A
		saw cut	3.5875	94.7%	N/A
		delaminated	3.2500	85.8%	0.058
Clark-Schwebel	I-583-5 3%-Lignosite FML	control	3.5000	100%	N/A
		saw cut	3.3875	96.8%	N/A
		delaminated	3.2125	91.8%	0.060
Interplastic	Iron silicide	control	1.6063	100%	0.065
		saw cut	1.6750	104.3%	0.140
		delaminated	1.6560	103.1%	0.110
Owens-Corning	A MnZn ferrite	control	2.1375	100%	0.099
		saw cut	1.8750	87.71%	0.103
		delaminated	2.0125	94.15%	0.047
TPI	NiZn ferrite	control	0.9688	100%	0.180
		saw cut	0.9063	93.5%	0.200
		delaminated	0.9563	98.7%	0.176

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 happen for sample I-583-5 from Clark-Schwebel (3%-lignosite tagging), shown in the fourth row of Table 3. This sample presented 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 properties of a normally nonconducting material. And adding small amounts of electromagnetic particles to a nonmagnetic 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 regions 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 some 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.

Passive Tagging Testing of Industrial Specimens

1. Sensitivity of particles to eddy currents excitation: All tagged samples presented an eddy current response except the 0.3%-lignosite FML tagged sample I-583-6 from Clark-Schwebel, Inc. due to its low tagging concentration.
2. Delamination detection: The delaminations created by the insertion of cellophane, mylar or nylon tape could not be detected with the Hall effect probe of the eddy current test system.
3. Crack detection: The cracks created by saw-cuts were detectable with the Hall effect probe of the eddy current test system, and the detection was based on comparing the amplitudes of the eddy current responses.

Active Tagging Testing of Industrial Specimens

1. Sensitivity of particles to magnetic excitation: All tested specimens were satisfactorily actuated by the magnetic excitation.

2. Defect detection: The frequency response signature of the specimens was found to change when defects (saw-cuts and delaminations) were introduced. The reduction in natural frequency implied a reduction in modal structural stiffness that can be logically correlated to the presence of delaminations and cracks.
3. Dependence on loading and excitation: The measured parameters (natural frequency and damping ratio) were found to be independent of loading conditions and excitation type.

ACKNOWLEDGMENTS

The authors gratefully acknowledge the financial support of the U. S. Army Corps of Engineers, Construction Engineering Research Laboratories, through contract No. DACA88-94-D-0021-002. The support of the Composites Institute of the Society of Plastic Industry is also gratefully acknowledged.

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