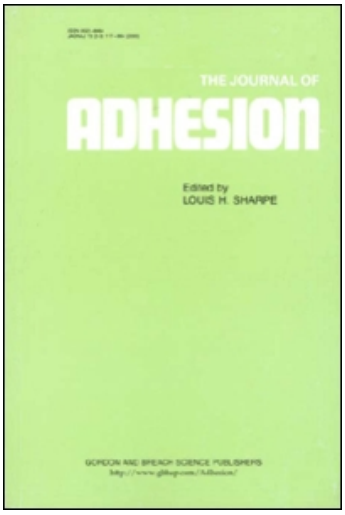

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Analysis of Composite Processing Using Magnetic Resonance Imaging

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NOTE

Analysis of Composite Processing Using Magnetic Resonance Imaging

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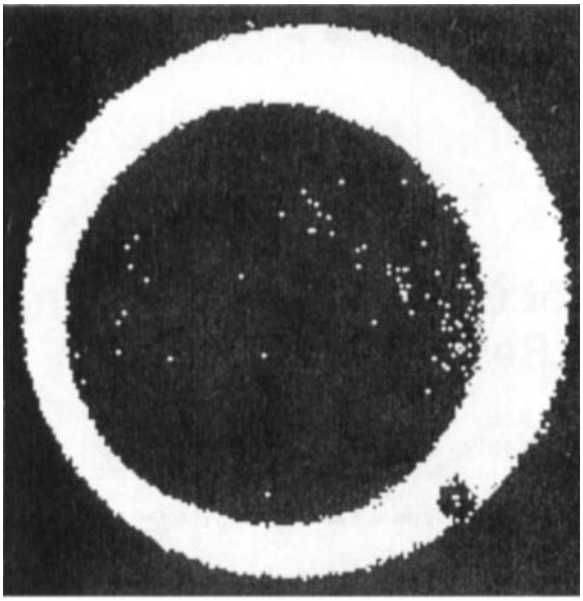
KEY WORDS NMR; imaging; composites; void content; water absorption.

Nuclear magnetic resonance imaging utilizes FT–NMR in conjunction with linearly applied gradient fields to resolve spatially the spins in a protonated solution. Magnetic resonance imaging (MRI) has been used extensively in the medical field. More recently, MRI has been used to image the distribution of liquids in materials.[1–4] Protons are the most extensively used nuclei based on their sensitivity and abundance.

The imaging technique is a means of detecting and mapping previously invisible imperfections in fabricated articles. The internal flaws can be detected directly or indirectly through the introduction of a mobile protonated solution. One of the areas of considerable interest is the absorption of water in composite materials. Rothwell[2] has imaged the absorption of water in fiber-glass reinforced epoxy composites. In this work, MRI is used to study the internal defects in a composite system as a function of processing conditions.

Three pultruded samples were studied using NMR imaging. One sample, a glass fiber-reinforced polyester rod with a fiber content of 51% by volume, was pultruded at a pulling speed of 18 inches per minute. Two glass fiber-reinforced nylon rods with fiber contents of 51% by volume were first mixed with different catalyst contents following the reaction injection molding (RIM) process and then pultruded with a pulling speed of 18 inches per minute. Approximately 4% of the reactive components, sodium hydride and phenyl isocyanate, were used in one nylon rod, while in the other rod 1% of the reactive components were used. The pultrusion die diameter was 9 mm. It was noted that before exposure to water, under the same processing conditions, the nylon rod with the higher catalyst

(1A)



(1B)

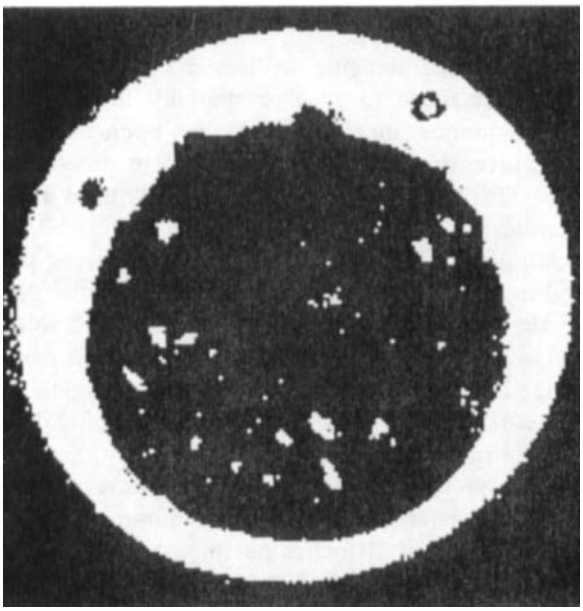


FIGURE 1 NMR image of a 1 mm slice selected from A) a polyester pultruded composite rod (24 scans), B) a high catalyst content nylon RIM-pultruded composite rod (60 scans), and C) a low catalyst content nylon RIM-pultruded composite rod (60 scans). The solid marker is 1 mm in diameter and the hollow marker is 1 mm in diameter with a 0.25 mm wall thickness. The images were enlarged and gray scale expanded.

(1C)

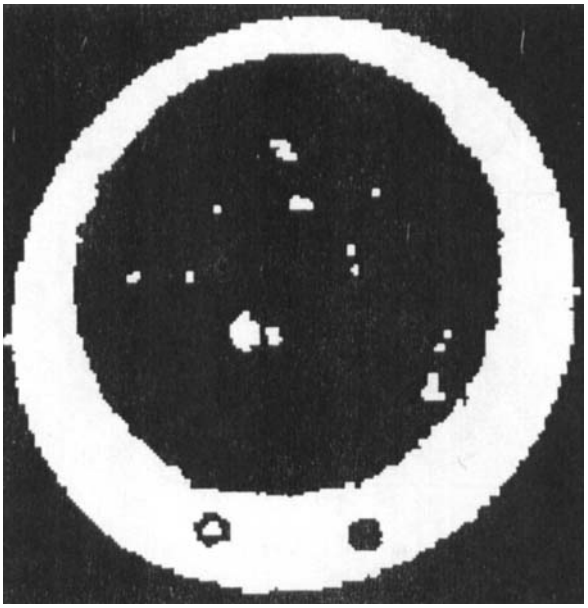


FIGURE 1 (*Continued*)

content exhibited a smoother surface finish with fewer defects than the nylon rod with the lower catalyst contents.

The rods were soaked in an 80°C water bath for 25 weeks before imaging. The uptake of water into the composites was measured by the increase in weight of the composite rods. The images were recorded on a Bruker MSL 300 spectrometer with a ^1H resonance frequency of 300.13 MHz. A spin-echo pulse sequence with a hard 90° and selective 180° pulses was used with a repetition time of 2 s and an echo time of 47 ms. Gradient strengths were typically 2 G/cm. The slice thickness was approximately 1 mm and the slices were taken transverse to the fiber axis.

Figure 1 shows the transverse slices of the composite rods. The display matrix is 256 × 256 pixels. The rods are standing in 1.5 cm diameter vials containing water. The white regions are the regions with the greatest water content. Figure 1A is a slice selected from the polyester composite rod. The rod is centered in the vial of water as shown by the white ring around the rod. The uniform darkness of the rod itself shows that there is very little if any water within the composite. A 1 mm diameter Teflon micro-tubing marker appears in the lower right quadrant of the water ring. Figure 1B is a slice selected from the nylon composite rod with the higher catalyst content. There are two markers present, the aforementioned Teflon tubing and a hollow tube with a wall thickness of 0.25 mm in the upper right. The nylon rod exhibits an inhomogeneous pattern of light and dark regions. The many white regions in the rod indicate the presence of absorbed water in the composite. The size of these white regions is approximately 1 mm. Figure 1C is a

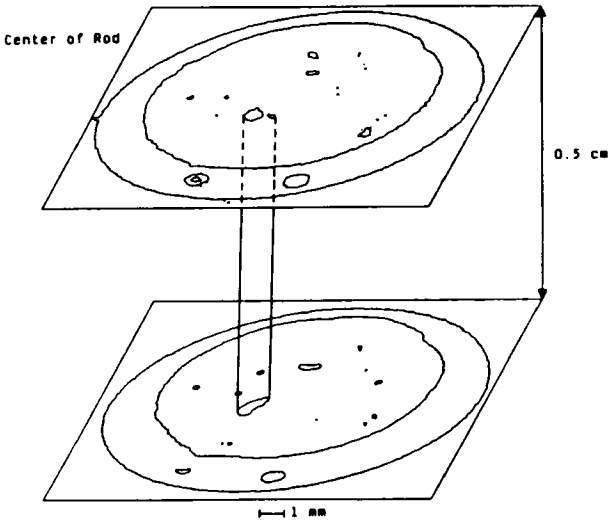


FIGURE 2 A contour plot of two 1 mm thick slices 0.5 cm apart in the low catalyst content nylon composite rod.

slice from the nylon rod with the lower catalyst content. The same markers are present. There is an inhomogeneous pattern of water but only a few large patches of water are present. Figure 2 is a contour plot of two slices 0.5 cm apart in the low catalyst nylon rod. One can clearly observe water in the same regions indicating the formation of tubular voids created by the manner in which the fibers were pultruded.

The amount of water observed in the composite by NMR imaging correlated with the increase in weight of the composite. The polyester rod showed very little diffusion of water and only exhibited a 1.2% gain in weight (Table I). Patches of water appeared in the nylon composite rods which showed an increase in weight of 3.7% and 3.8%. There was a greater distribution of large water regions in the high catalyst nylon rod while the low catalyst nylon rod had only one large region of water. An approximate measure of the relative water content was calculated using the histogram intensities of the background and of the water. The histogram intensity of the water was ratioed against the histogram intensity of the background or the composite rod itself. Two measurements were made for each sample and the average value is shown in Table I. The polyster rod shows a

TABLE I
Increase in weight of composite rods exposed to 80°C water bath for 25 weeks and average ratio of histogram intensity of water to histogram intensity of background.

Sample	Increase in weight (%)	Avg. of water/background
Polyster Rod	1.2	0.005
Nylon Rod (High Catalyst Content)	3.8	0.039
Nylon Rod (Low Catalyst Content)	3.7	0.034

negligible amount of water in the slice. The low catalyst nylon rod showed an average ratio of 0.034 while the high catalyst nylon rod showed an average ratio of 0.039. These values correlate fairly well with the observed increase in weight shown in Table I. It should be noted that histogram intensities can only give an approximate indication of water content; due to the limitations in echo times, only free water can be imaged while tightly bound water cannot be imaged.

It has been shown that a good surface finish does not guarantee a low void content. The ability of MRI to slice at different positions in the rod shows that the water filling the voids can be mapped over the entire rod. It appears that water diffuses by following the fibers in the composite. As seen in previous studies, MRI can be used to study the absorption of water into composite materials. In this study, it has also been demonstrated that MRI can be used to map the voids in composites due to processing conditions.

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References

1. W. P. Rothwell and P. P. Gentempo, *Bruker Reports* **1,** 45 (1985).
2. W. P. Rothwell, D. R. Holecek, and J. A. Kershaw, *J. Polym. Sci.: Polym. Lett. Ed.* **22,** 241 (1984).
3. S. Blackband and P. Mansfield, *J. Phys. C: Solid State Physics* **19,** L49 (1986).
4. L. D. Hall and V. Rajanayagam, *J. Mag. Reson.* **74,** 139 (1987).