Evaluation of Glass Fiber Reinforced Plastics by XPS and TOF-SIMS

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(Received October 12 1998; accepted January 18 1999)

Abstract: In this work we have investigated the reason why the tensile strength of glass fiber reinforced plastics decreased during the immersion test in hot water by using XPS and TOF-SIMS. It was found that this decrease of the tensile strength could be attributed to the debonding between the glass fiber and the plastics.

1.Introduction

In recent years, plastics have been widely used as the structural materials for various uses from their light weight, low cost and ease of molding. And, when the strength of the plastics is not enough, it is known that plastics can be reinforced by mixing with glass fibers treated with silane coupling reagents [1,2]. However, it was found that the tensile strength of the glass fiber reinforced plastics dropped by the immersion test in hot water. The tensile strength of the plastics, on the other hand, did not decrease in the same test. Therefore, it was assumed that this decrease of the tensile strength was attributed not to the deterioration of plastics but to the debonding between the glass fiber and the plastics.

Although we tried to confirm this using the fracture surface field emission scanning electron microscopy (FE-SEM) images, there were no difference between glass fiber reinforced plastics before and after immersion test (Figure 1). It was thought that FE-SEM was not suitable for the observation of glass fiber/plastics interface, because the interface layer was much thinner than the information depth of FE-SEM (ca. 1μm). Therefore we have investigated the deterioration of the glass fiber reinforced plastics during the immersion test in hot water by the use of X-ray photoelectron spectroscopy (XPS) and time-of-flight secondary ion mass spectrometry (TOF-SIMS) whose sensitivity to the surface is very high.

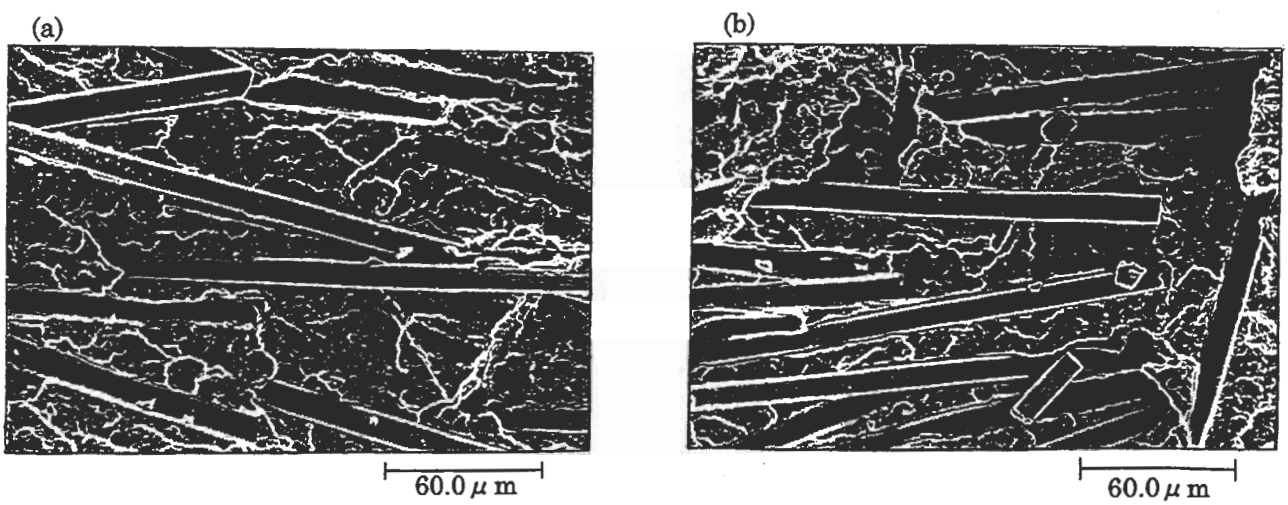

Figure 1. Fracture Surface FE-SEM Images of GF-PPE, (a) before and (b) after immersion test in the hot water.

2.Experimental

We prepared two kinds of glass fiber reinforced plastics; GF-PPE (poly phenylene ether) and GF-PPS (poly phenylene sulfide). They were commercial products of GE plastic Japan Ltd. and Dainippon ink and chemicals inc., respectively. The conditions of the hot water immersion tests were as follows:

Temperature : 120 ℃ for GF-PPE
90 ℃ for GF-PPS
Duration : 500 h. for GF-PPE
600 h. for GF-PPS

The fractured surfaces of the samples before and after immersion tests were evaluated by XPS and TOF-SIMS.

XPS measurements were done by a Phisical Electronics PHI 5700 ESCA system using MgKα X-rays(hν=1253.6eV), and an electron takeoff angle of 45 degrees. The X-ray gun was operated at 14kV and 28.5mA, corresponding to a power of 400W. The analyzed area was 800μm ϕ. XPS data were acquired by both low-resolution survey scans (0-1100eV) and high-resolution multiplex scans for each of the elements detected on the fractured surface region.

TOF-SIMS ion images mesurements were done by a Phisical Electronics TRIFT Ⅱ spectrometer using a ^{69}Ga liquid metal ion source operating at 25kV. Secondary ion extraction was accomplished with ±3kV bias on the sample. Therefore, this extraction field is that the primary ion impact energy is 22kV in positive ion mode and 28kV in negative ion mode.

3.Results and Discussion

3-1.XPS Results

XPS gives information on the kinds and concentration of atoms on the surface.

Table1 summarizes the atomic concentrations of fractured surface of GF-PPEs and GF-PPSs measured by XPS. Both the data show that the relative contents against matrix plastics (C) of glass fiber components (Si, Ca etc.) increased for samples after immersion tests compared to those for samples before tests. These results suggested that the ratio of glass fiber uncovered/covered with matrix plastics on the fractured surface increased by immersion tests in hot water.

Table1. Atomic concentrations of the fractured surface of glass fiver reinforced plastics (atomic%)

Sample		C	O	Si	Ca	Al	Mg
PPE	new one	87.5	9.8	1.1	0.3	0.3	1.0
	tested one	79.7	14.8	2.4	0.6	0.6	2.0

Sample		C	O	S	Si	Ca
PPS	new one	86.3	6.2	6.9	0.5	0.1
	tested one	82.7	9.2	6.2	1.6	0.2

3-2.TOF-SIMS Results

Figure2 shows positive ion images (total ion image and sum of m/z 28(Si) + 40(Ca)) of the fractured surface of GF-PPEs. It shows that the distribution of the components of glass fiber could be identified by the secondary ion images of the fractured surface of the sample immersed in hot water, while the identification was difficult for the sample before the immersion test.

These results suggested as follows:

1. The glass fibers in the sample before the immersion test were found to be covered with matrix plastics on the fractured surface.
2. On the other hand, the glass fibers in the sample after the immersion test were clearly observed on the fractured surface, since they were uncovered with matrix plastics.

That is, the sample before the immersion test was fractured at the bulk of plastics while the sample after the immersion test were fractured at the interface between glass fiber and plastics.

In other words, it was suggested that the bonds between silane coupling reagent and glass fiber, which played the role of adhesives for glass fiber and plastics, were hydrolyzed during the immersion test in hot water.

The results of GF-PPS were almost the same as those of GF-PPE.

4.Conclusions

From the analytical results of XPS and TOF-SIMS, it was suggested that the decrease of the tensile strength during the immersion test in hot water could be attributed to the debonding between glass fiber and the plastics in the glass

fiber reinforced plastics.

It was possible to analyze the very thin surface by using XPS and TOF-SIMS, eventhough impossible to observe by using FE-SEM.

And by using TOF-SIMS, understanding of the local change is possible, while by XPS only understanding of the general tendency of the change is possible.

5.References

[1]E.Mader, Composites Sci.Technol., 57, 1077(1997)

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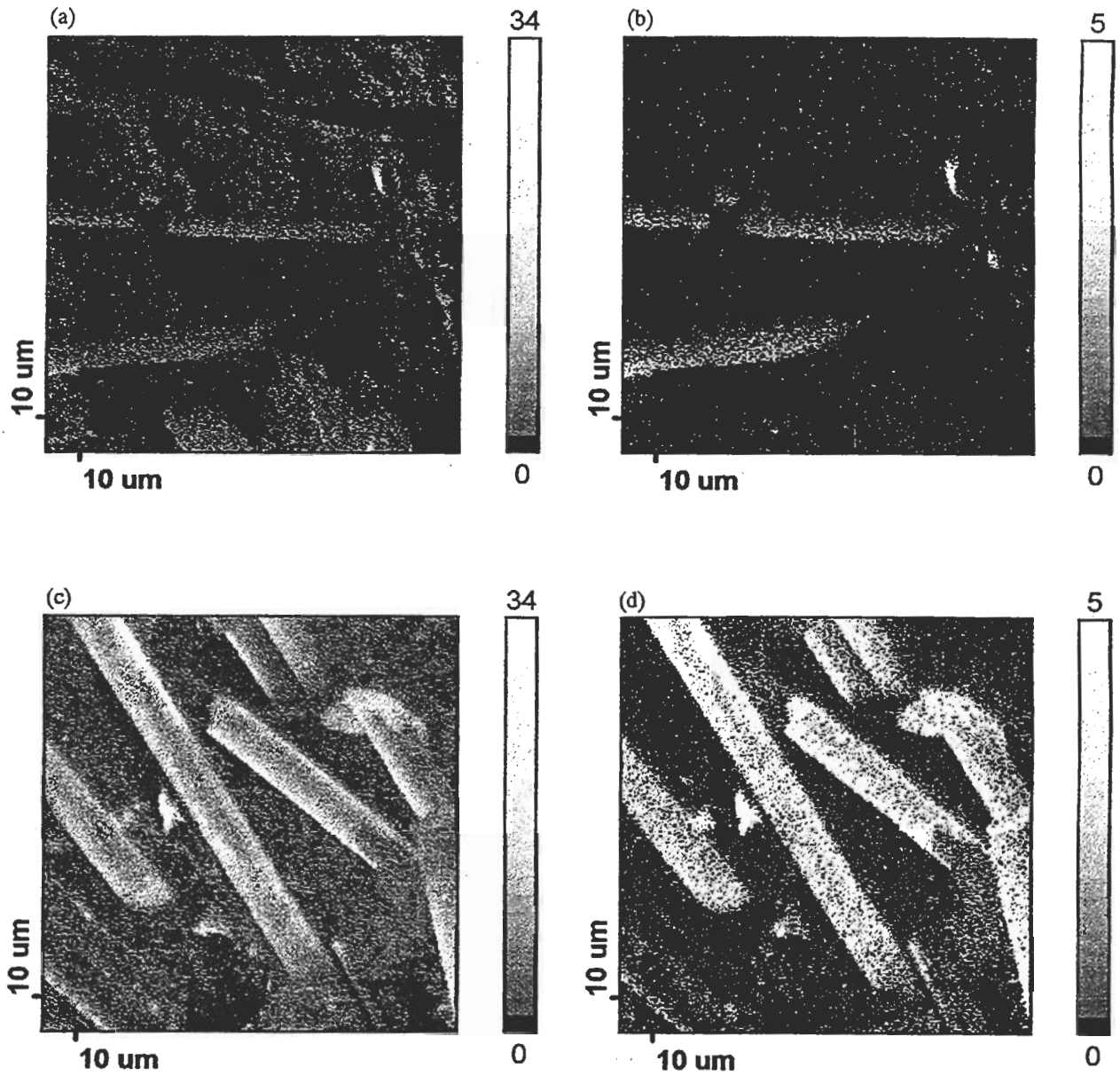

Figure2. TOF-SIMS positive ion images (120μm field of view) of GF-PPEs of before (top) and after (bottom) immersion test. (a) and (c): total ion images, (b) and (d): summed images of Si at m/z 28 and Ca at m/z 40