EFFECT OF GAMMA RAY IRRADIATION ON INTERLAMINAR SHEAR STRENGTH OF GLASS FIBER REINFORCED PLASTICS AT 77 K

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ABSTRACT

It is known that an organic material is damaged by gamma ray irradiation, and the strength after irradiation has dependence on the gamma ray dose. These issues are important not only to make global understanding of electric insulating performance of glass fiber reinforced plastics (GFRP) under irradiation condition but also to develop new insulation materials. This paper presents the dependence of fracture mode and interlaminar shear strength (ILSS) on the material and the gamma ray irradiation effect on the fracture mode and the ILSS. 6 mm radius loading nose and supports were used to prompt ILS fracture for a short beam test. A 2.5 mm thick small specimen machined out of a 13 mm thick G-10CR GFRP plate (sliced specimen) showed lower ILSS and translaminar shear (TLS) fracture, although the same size specimen prepared from a 2.5 mm G-10CR GFRP plate (non-sliced specimen) showed ILS fracture and the higher ILSS. Both type of specimens showed the degradation of ILSS after gamma ray irradiation. The fracture mode of the non-sliced specimen changed from ILS to TLS fracture and no bending fracture was observed. The resistance to shear deformation of glass cloth/epoxy laminate structure would be damaged by the irradiation.

KEYWORDS: Interlaminar shear strength, Interlaminar shear fracture, GFRP, Gamma ray irradiation, Short beam test.

INTRODUCTION

The construction site of International Thermonuclear Experimental Reactor (ITER) was decided to France and the organization was established [1]. The preparation of the ITER construction started and the design detail has been checked. In such a plasma device, streaming of neutron through large ports, such as neutral beam injection ports [2], and...
penetration of fast neutron through shielding blanket will occur [3] and much neutron will come out of plasma vacuum vessel. A large-scale superconducting magnet system locates just outside of the plasma vacuum vessel, and some neutron hit the thermal insulation and magnets. The neutron activates the materials composed of the magnets and the activated materials emit a gamma ray. So, the materials would be irradiated by neutron and gamma ray at simultaneously during the operation, and their properties would be changed. To understand the mechanism of the properties changes and to simulate the irradiation status, some efforts were undertaken and valuable results were obtained [4,5].

Although many kinds of materials will be used for the superconducting magnet system in the burning plasma devices, one of the important materials is an electric and thermal insulation material. For ITER design, a composite of glass fiber reinforced plastics (GFRP) with a glass cloth/epoxy laminate structure is the first candidate. The composite has been used for a lot of superconducting magnets and the treatment technology has been almost established in the world. Since the interlaminar shear strength (ILSS) is a parameter to reveal the materials resistance against the mechanical and thermal stresses or the deformation, a lot of investigations were performed. As for the cryogenic use, two series of round robin tests were performed to establish the test method, and the standardization draft has been presented [6,7]. Nowadays, the short beam tests are considered to be one of screening tests for material selection. However, the effect of test fixture configuration was not investigated clearly in these round robin tests. The new research on this matter has been carried out, and it was clarified that there would be clear effect of fixture radius on the fracture mode and the ILSS, and the large radius loading nose and supports would produce the higher possibility of the interlaminar shear (ILS) fracture [8]. Since the recommended radius was 6 mm for both the loading nose and the supports, the 6 mm radius fixture was used in this study.

To investigate the material dependence of fracture mode, two kinds of plates were prepared. The specimens were cut from these plates and irradiated by gamma ray using Co60. After the irradiation, the ILSS was evaluated at 77 K by the short beam test method together with non-irradiated specimens.

This paper presents the results of the fracture mode and ILSS of non-irradiated and irradiated specimens, and the effect of the irradiation was discussed.

TEST MATERIALS AND PROCEDURE

A test material used in this study was so-called G-10CR with glass cloth/epoxy laminate structure. Two different thickness plates were prepared. One was 2.5 mm thick and the other was 13 mm thick. Both plates were commercial ones produced by Arisawa Manufacturing Ltd. and the details such as resin type and fiber treatment were not clear. The location of the specimen is shown in FIGURE 1. The configuration of the specimen

(a) 2.5 Unit: mm

(b) 13 Unit: mm

FIGURE 1. Specimen location in the original GFRP Plates. (a) and (b) show non-sliced and sliced specimens, respectively.
for the short beam test was 15 mm$^L$ x 10 mm$^W$ x 2.5 mm$^t$, and this size was the same as that used in previous tests [8]. Therefore, the specimen cut from the 2.5 mm thick plate kept the original plate surface covered with epoxy on the right and back surfaces, which is designated as “Non-sliced specimen.” The other specimen was machined out of the 13 mm thick plate. The plate was sliced at the mid plane and specimens were taken out from the middle part of the plate. So, this specimen is called as “Sliced specimen.” The directions of fibers were almost same as the directions of 15 mm and 10 mm of the specimen.

The short beam test procedure is the same as those described in reference 8. The small mechanical testing machine, of which capacity was 10 kN, was used and tests were carried out in liquid nitrogen. The load was applied in the vertical direction to the laminate structure. The span was 12.5 mm and ratio of the span to thickness was 5.0. The stroke rate was controlled to be 0.75 mm/min and the sampling rate was 50 Hz. The 6 mm radius loading nose and supports were used according the previous test results [8].

The ILSS was evaluated by the following equation.

$$ILSS = \frac{3P_b}{4bh}$$

where $P_b$ is the maximum load, $b$ is specimen width and $h$ is specimen thickness. When the load is not the maximum, the value will be called as $S_{sh}$.

The gamma ray irradiation by Co60 was carried out in air and at room temperature at Institute of Scientific and Industrial Research in Osaka University. After machining, three specimens were wrapped together with an aluminum foil into one sample set. 7 sets were prepared and placed at the position where the calculated dose was obtained, and the sample sets were taken out one set by one set after a certain irradiation time. The targeted dose of gamma ray was 0.1, 0.2, 0.5, 1.0, 2.0, 5.0 and 10.0 MGy. Three specimens were tested for one test condition to confirm the repeatability of the test.

**TEST RESULTS AND DISCUSSION**

**Fracture Mode**

The specimens after the short beam test in liquid nitrogen are shown in FIGURE 2-5. In each figure, the top picture shows the right surface where the loading nose attached. The middle is a side surface of the specimen and the fracture mode is very clear. The bottom presents the back surface where the supports contacted. It is commonly recognized that the trace of the loading nose is observed very clearly on the right surface and no supports trace on the back surface. FIGURE 2 shows the non-sliced specimens, which were not irradiated, and the ILS fracture occurred in all three specimens. However, the sliced specimen fractured in the TLS and the bending fracture mode as shown in FIGURE 3.

The bending fracture would occur because of the relatively lower bending tensile stress and the TLS fracture would be caused by the local bending deformation near around the loading nose. If there is a local bending deformation at the bottom surface just under the loading nose, the bending fracture would be accelerated. In other words, both of the bending and the TLS fracture are related to the local bending deformation and/or the stress concentration. On the other hand, the ILS fracture is simply dependent on the shear stress on the neutral plane of the specimen. Therefore, the bending and the TLS fracture would strongly affected by the local stress around the loading nose, although the ILS fracture would be dependent on only the almost uniform shear stress.
FIGURE 2. Fractured specimens of non-sliced specimens which were not irradiated and tested at 77 K. All specimens were ILS fractured on the neutral plane of the specimen.

FIGURE 3. Fractured specimens of sliced specimens which were not irradiated and tested at 77 K. One specimen showed TLS fracture and others were bending fractured.

FIGURE 4. Fracture specimens of non-sliced specimens which were irradiated up to 0.1 MGy and tested at 77 K. One specimen showed TLS fracture and others were ILS fractured.

FIGURE 5. Fracture specimens of sliced specimens which were irradiated up to 0.1 MGy and tested at 77 K. All specimens showed TLS fracture.
TABLE 1. Summary of specimen code and fracture mode. N66 and S66 are non-irradiated specimens and tested with 6 mm radius loading nose and supports. N77 and S77 are non-irradiated specimens and tested with 7 mm radius loading nose and supports.

<table>
<thead>
<tr>
<th>Specimen code</th>
<th>Gamma ray dose (MGy)</th>
<th>Fracture mode</th>
<th>Specimen code</th>
<th>Gamma ray dose (MGy)</th>
<th>Fracture mode</th>
</tr>
</thead>
<tbody>
<tr>
<td>N77</td>
<td>Non-irr.</td>
<td>B, I, I</td>
<td>S77</td>
<td>Non-irr.</td>
<td>T, T, T</td>
</tr>
<tr>
<td>N01</td>
<td>0.1</td>
<td>I, T, I</td>
<td>S01</td>
<td>0.1</td>
<td>T, T, T(B)</td>
</tr>
<tr>
<td>N02</td>
<td>0.2</td>
<td>I, I, I</td>
<td>S02</td>
<td>0.2</td>
<td>T, T, T</td>
</tr>
<tr>
<td>N05</td>
<td>0.5</td>
<td>I, I, T(I)</td>
<td>S05</td>
<td>0.5</td>
<td>T, T, T</td>
</tr>
<tr>
<td>N10</td>
<td>1.0</td>
<td>T, T(I), T</td>
<td>S10</td>
<td>1.0</td>
<td>T, T, T</td>
</tr>
<tr>
<td>N20</td>
<td>2.0</td>
<td>T, T, T</td>
<td>S20</td>
<td>2.0</td>
<td>T, T, T</td>
</tr>
<tr>
<td>N50</td>
<td>5.0</td>
<td>T, T, T</td>
<td>S50</td>
<td>5.0</td>
<td>T, T, T</td>
</tr>
<tr>
<td>N100</td>
<td>10.0</td>
<td>T, T, T</td>
<td>S100</td>
<td>10.0</td>
<td>T, T, T</td>
</tr>
</tbody>
</table>

Note: I, B and T indicate Interlaminar shear, Bending and Translaminar shear fracture, respectively.

To discuss the change in fracture mode, the summary of fracture mode was prepared as shown in TABLE 1. In the table, I, B and T present the interlaminar shear, bending and translaminar shear fracture, respectively. Also, indication in parentheses means the secondary fracture mode. In case of non-sliced specimen, the fracture mode changed from ILS to TLS fracture, and no bending fracture was observed. The sliced specimens indicated all specimens were TLS fractured. As mentioned above, the TLS fracture would be caused by the stress concentration of shear stress. As discussed in the previous paper [8], a shear deformation perpendicular to the plane (ab-plane) connecting the loading point and supporting point occurs near the contact point of the loading nose on the specimen. This deformation produced the shear stress concentration and this concentration was observed even in the 6 mm radius loading nose [8]. From the results of irradiated non-sliced specimens, it would be considered that the apparent Young’s modulus would be kept almost same value, but the bonding strength on an interface between fiber glasses and matrix resin would be damaged. The large shear deformation would prompt to cause the TLS fracture. When the FIGURE 2 and FIGURE 5 are compared, it will be noted that the width of the shear deformation band in the sliced specimens is larger than that in the non-sliced. It would be come from the lower Young’s modulus and the lower bonding strength. Also, it is considered that the gamma ray irradiation would damage the epoxy resin and the apparent Young’s modulus would be decreased in case of heavy irradiation.

Deflection Curves and Interlaminar Shear Strength

FIGURE 6 and FIGURE 7 show the results of deformation curves of the specimens. Since the stroke rate was fixed to 0.75 mm/min, the horizontal axis of time corresponds to the deflection. It must be noted that the deflection would include the local deformation of the specimen at loading point and supporting points.

Most specimens except for heavily irradiated ones showed linear deformation behavior against time and suddenly the fracture happened and the load dropped. When the slope of both types of specimens is compared, the sliced specimens show the gentle slope. It means the sliced specimen has smaller stiffness than that of the non-sliced specimen. This fact supports the previous consideration on stress and deformation concentration. Regarding the effect of irradiation on the deformation behavior, the specimens irradiated up to 2 MGy still show the linear deformation behavior. From the results, it could be noted...
that the materials elasticity is kept to be a certain value, although the critical strength
becomes down after the irradiation. However, when gamma ray of 5 MGy was irradiated,
the specimen did show the elasticity no longer. The slope became smaller and the sudden
fracture did not occur. After showing the maximum load, the deformation underwent
gradually. The heavy irradiation of over at least 5 MGy would change the material plastic
and the strength become meaningless. The specimens irradiated to 5 MGy and 10 MGy
were swelled and softened. It means that the heavy irradiation might change the organic
structure by degradation, bridging or polymerization and make the material porous. The
details will be investigated more and presented in the future.

The dependence of the ILSS (here, it means the maximum shear strength calculated
with the maximum load.) on the irradiation dose is shown in FIGURE 8. The ILSS has a
scatter band as shown in slashed. 0.1 MGy irradiation did not cause the clear degradation
of ILSS, but it decreased gradually and 1 MGy irradiation dropped the ILSS remarkably.
The degradation at 1 MGy is about 70% for both type of specimens, sliced and non-sliced,
and it is very large, comparing with the presented data shown in reference [8]. The degree
of irradiation effect on the degradation is considered to depend on the material. Since the
details of the composed materials of these commercial G-10CR plates are not clear, the
mechanisms for the heavy drops are not able to be discussed in this paper. The further
study on this issue is needed.
DISCUSSION ON IRRADIATION EFFECT

It would be reasonable to consider that the heavy irradiation would make the matrix porous and the apparent stiffness would become small. Also, S. Nisijima presented that the bonding strength on the interface of glass fiber and matrix resin would be decreased in case of E-glass FRP, showing some SEM photos [4]. Therefore, it would be clear that the gamma ray irradiation degrades the bonding strength on the interface. When the stiffness of the material is small, the deflection becomes large and the interface would be separated more easily because of the lower bonding strength. In the case of lower irradiation dose, the apparent stiffness did not change so much but the maximum shear strength decreased. From these results, it would be considered that the degradation of the bonding stress would occur at lower dose than the matrix change. If the bonding strength should have dominant effect on the fracture, the TLS fracture would happen at the lower load, which means smaller deflection and smaller stress concentration, around the contact point of the loading nose, because the critical stress (the bonding strength) might be reduced by the irradiation. The experimental results observed in FIGURE 8 could be explained by this consideration.

The gamma ray irradiation effect will be summarized as follows: There are four possibilities to degrade the ILSS. The first is Young’s modulus, the second is the bonding strength between fibers and matrix resin, the third is the critical strength of the matrix resin and the fourth is the critical strength of the fiber. The weakest matter will be the bonding strength. When the interface between the fibers and the matrix is separated, the laminate structure dose work no longer and the strengthened function will be lost. The Young’s modulus will come down and the critical strength of the composite will become lower. As discussed before, the TLS fracture would be caused by the stress concentration similarly to the bending fracture. Therefore, the degradation of those factors result in producing the stress concentration or promoting the grade of the concentration, and it tends to cause the TLS fracture and give the lower ILSS.

SUMMARY

In this study, the short beam tests were carried out using the two types of specimens. One was the sliced specimen which was machined out from the middle part of 13 mm thick G-10CR commercial plate. And the other was a non-sliced specimen which was cut from the 2.5 mm thick G-10CR commercial plate. After machining to specimen configuration,

![Figure 8](image_url)

**FIGURE 8.** Change in ILSS against gamma ray dose. Non-sliced specimens showed fracture mode change from ILS to TLS fracture. All sliced specimens broke in TLS fracture mode.
both types of specimens were irradiated by Co60 in air at room temperature. After the irradiation, ILSS tests were carried out in liquid nitrogen and the effect of gamma ray irradiation was studied. The main results are summarized as follows.

1. All sliced specimens showed TLS fracture and the smaller stiffness. The inner laminate structure of 13 mm thick plate seems weaker than that of 2.5 mm thick plate. The stiffness and the fracture mode would depend on the production process and the materials properties.

2. In case of the non-sliced specimen, the gamma ray irradiation decreased ILSS with an increase of the dose and the fracture mode changed from ILS to TLS fracture. When the dose was around 0.5 MGy, the specimens showed ILS fracture and ILSS would not change. By increasing the dose, the fracture mode changed from ILS to TLS and ILSS dropped. The strength of laminate structure is considered to be most damaged by gamma ray.

3. In case of the sliced specimen, ILSS was decreased by the irradiation and the fracture mode of TLS was the same as the non-irradiated specimens. The TLS fracture seems to be caused by the stress concentration of $\tau_{ab}$, and the stiffness also affects the fracture mode. The gamma ray irradiation degrades the bonding strength between glass cloth and epoxy resin, and it makes shear deformation band wider resulting in acceleration of TLS fracture.

ACKNOWLEDGEMENTS

Authors would like to thank Mr. Toshiji Ikeda at Institute of Scientific and Industrial Research in Osaka University for the gamma ray irradiation.

Part of this work was supported by Grant-in-Aid for Scientific Research (#16560725), NIFS collaboration research programs (NIFS04KOBF008, NIFS04KFRF007), and Fusion Engineering Research Centre program at NIFS (NIFS05UCFF004, NIFS06UCFF013).

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