



Study of the Homogeneity of Glass Fiber Reinforced Polymer Composite by Using Neutron Radiography Technique

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Abstract: Direct film neutron radiography (NR) technique has been used to study internal defects and homogeneity of glass fiber reinforced polymer composite. In this study, neutron radiography (NR) technique is used to detect any spot or cracks in any sample because if we find any cracks or defect in the sample by NR, it means that the sample is not homogeneous and the materials are not perfectly distributed. Tangential Neutron Radiography Facility of 3MW TRIGA Mark-II research reactor has been utilized in the study. A series of neutron radiography images were taken to determine the optimum exposure time of the sample. In this experiment, the optimal exposure time is estimated at 40 min and from radiographic images of the sample; we see that there were no spots found in the sample. By measuring the optical density of any sample we detect the homogeneity of this sample. Optical density values of different reference positions of the sample have been found to be unchanged and Optical density values of the central positions of the sample and the reference positions have also been found to be same. These prove that associated solutions of glass fiber-reinforced polymer composite are not diffused and distributed uniformly. From the observation of neutron radiographic images of the sample at optimum exposure time and optical density of the sample at a different position, it revealed that the glass fiber-reinforced polymer composite is uniformly distributed and no voids, defects, and cracks could be found in the composite observed in the radiograph. Thus the elemental distributions of the composite are found to be almost homogeneous. So, the fabrication of the glass fiber-reinforced polymer composite is perfect.

Keywords: Neutron Radiography, Optical Density, Homogeneity, Internal Structure, Defect

1. Introduction

The neutron is a subatomic particle with no net charge. As a result, the Coulomb attraction does not affect it, since it passes through the substance. This is also useful for image processing applications. Neutron radiography is an additional method of X-ray radiography (Figure 1 and Figure 2). The X-ray attenuation increases with higher electron density [1]. Figure 1 and Figure 2 show photograph of X-ray radiography and neutron radiography of a camera respectively. Neutron radiographs show interior details, including a plastic cover, but X-ray radiographs show little without the plastic cover.



Figure 1. X-ray radiograph of a camera [2].

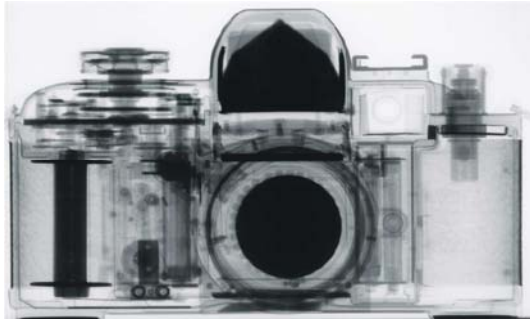


Figure 2. Neutron radiograph of a camera [2].

Neutron radiography (NR) is a visualization method that provides images, such as X-rays and gamma rays. The advantage of neutron radiography is its ability to form very light elements (i.e., having low atomic numbers), such as hydrogen, water, carbon, etc. In addition, neutrons penetrate very heavy elements (i.e. with high atomic numbers) such as lead, titanium etc. as well as distinguish between different

isotopes of the same element. This makes neutron radiography an important tool for the investigation of both radioactive and non-radioactive substances. NR is a photographic image of the internal structure of a substance obtained using neutrons. Neutrons can detect light elements with large neutron absorption cross sections, such as hydrogen and boron. The information provided by the spatial and temporal attenuation of the beam is recorded on the magnetic medium through analog or digital signals. NR is a non-destructive testing (NDT) technique, which is completely complementary to other NDT techniques, like X-ray or gamma-ray radiography.

A comparison of the attenuation coefficients of the mass of x-rays and neutrons is showing Figure 3. Mass neutron attenuation shows a random image since the attenuation coefficient of the X-ray mass regularly increases with the number of the element.

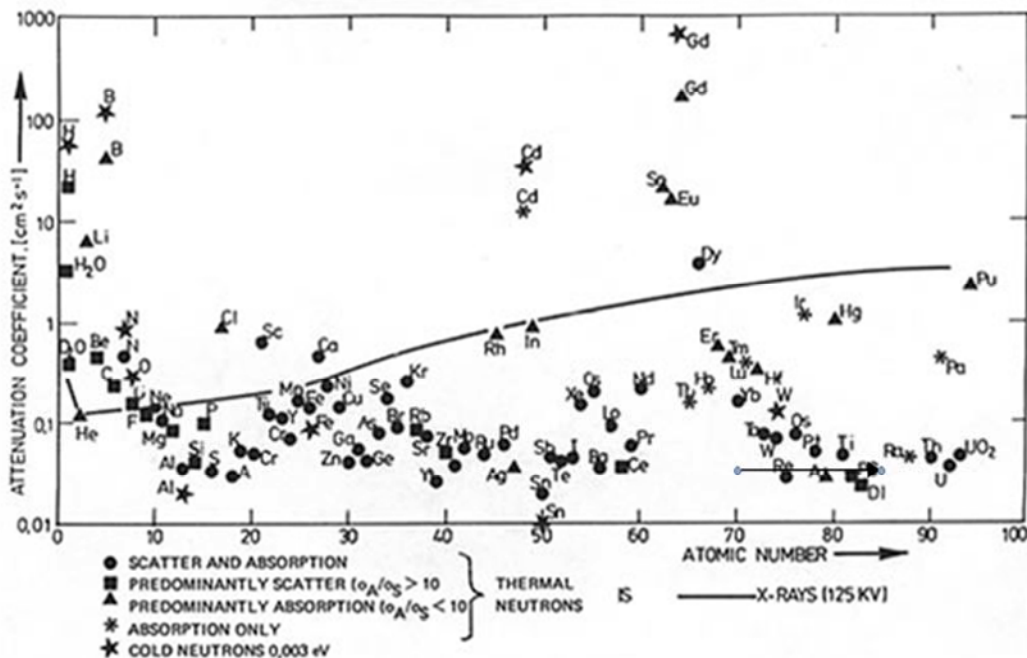


Figure 3. Neutron and X-ray mass attenuation coefficients for the elements [3].

Recently NR method has been applied to detect faults and to study water absorption properties of building materials reported by Islam *et al.* [4]. A neutron radiography standard testing method for the moisture analysis was introduced by Peterka *et al.* [5] to the building industries in order to evaluate the properties, functions and the efficiency of their water protective agents against the penetration of water, water solution etc. In another study, Ahasan *et al.* [6] quality of leather and ceramics have been studied. Study of corrosion in aluminum has been reported by Islam *et al.* [7]. In the present study, the NR set up at the tangential beam port of the 3.0 MW TRIGA Mark-II research reactor of AERE, Savar, Dhaka, Bangladesh has been used. Details of the NR facility of AERE, Savar, Dhaka can be found in reference to Rahman *et al.* [8]. Details of the parameters of the facility have been

given in Ahsan *et al.* [9]. A study of defects and water absorption behavior of jute products was reported by Azad Rahman *et al.* [10]. Study of the Internal Structure of Electronic Components RAM DDR-2 and Motherboard of Nokia-3120 by Using Neutron Radiography Technique was reported by Shahajan Miah *et al.* [11]. Khurshed Alam *et al.* investigate the Quality of Automated Machine Made Environmentally Friendly Brick (KAB) sample using Film Neutron Radiography Technique [12] Mbumbia *et al.* investigated that compressive strength and water absorption are two major physical properties of brick that are good predictors of bricks ability to resist cracking of face [13]. The material aspects of clay and ceramic technology, the physical properties of clay and various firing methods can be investigated using archaeometric techniques [14, 15]. Water

absorption capacity of the brick affects the surface finishing of the brick-laid wall also investigated [16, 17, 18]. Gas Evolution in Operating Lithium-Ion Batteries Studied by Neutron Imaging [19]. A non-destructive testing method of composite materials investigated by S. Gholizadeh [20]. S. Zheng et al investigated of aluminum alloy hemmed joints using neutron radiography [21]. Examining Curing Efficiency using Neutron Radiography by Mehdi Khanzadeh Moradillo et al. [22].

2. Experimental Facility

The experimental neutron radiography facility has been installed at the tangential beam port of 3MW TRIGA Mark-II reactors in the Institute of Nuclear Science and Technology, Atomic Energy Research Establishment, Savar, Dhaka, Bangladesh. The neutron radiography facility consists of the following devices/equipment.

2.1. Bismuth Filter

In the NR facility at TRIGA reactor of BAEC, a 15 cm long Bi filter in the tangential beam port is used to reduce the intensity of gamma-ray significantly from the beam to prevent the unwanted fogginess in the Radiographic image [12, 23].

2.2. Cylindrical Divergent Collimator

A cylindrical divergent collimator made of 120 cm long aluminum hollow cylinder with 5 cm and 10 cm diameter at the inner and outer end, respectively, has been inserted in the tangential beam port to collimate neutron beam of the reactor. The advantage of the divergent collimator is that a uniform beam can be projected easily over a large inspection area. Collimators are required to produce a uniform beam and thereby produce adequate image resolution capability in a neutron radiography facility [12, 23].

2.3. Lead Shutter

The outer end of the tangential beam tube is equipped with a lead-filled safety shutter and door to provide limited gamma shielding. The thickness of the lead in the shutter is 24 cm and the diameter of the shutter is 33 cm [12, 23].

2.4. Beam Stopper

A wooden box with a dimension of 68 cm × 40 cm × 68 cm has been made with the attachment of four ball bearings on the bottom part of it for forward and backward movement in front of the tangential beam port. It looks a wooden box, which contains neutron-shielding materials like paraffin wax and boric acid in 3:1 ratio by weight for neutron shielding [12, 23].

2.5. Sample and Camera Holder Table

There are a sample and camera holder table with both horizontal and vertical movement facility placed in front of

the beam line [12, 23].

2.6. Beam Catcher

To absorb transmitted and scattered neutron and gamma radiations, a beam catcher with dimension 100 cm × 100 cm × 85 cm has been placed behind the sample and camera holding the table. A 30 cm × 30 cm × 30 cm hole has been made in the middle of the front face of the beam catcher which coincides with the central axis of the beam port. A 30 cm × 30 cm × 15 cm lead block weighing 125 Kg has been placed at the back side of the hole for gamma shielding. For neutron shielding a mixture of paraffin wax and boric acid has been used in the catcher. The total weight of the beam catcher is 968 Kg [12, 23].

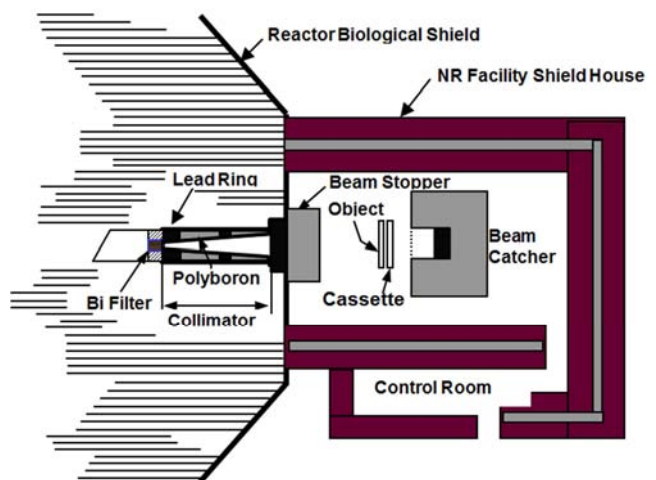


Figure 4. Schematic diagram of the neutron radiography facility [25, 26].

2.7. Biological Shielding House

The emitted neutron and the gamma rays are extremely dangerous for human body. This is why, to prevent these harmful rays to spread over the entire environment, a biological shielding house has been built around the NR facility of the tangential beam port. It is made of special concrete containing cement, heavy sand (magnetite, limonite and ordinary sand) and stone chips in the ratio 1:3:3. Paraffin wax and boric acid in 3:1 ratio by weight were also used inside the biological shielding wall for neutron shielding. The width and height of the biological shielding wall of the facility are ≈ 3.0 ft and 6.5 ft, respectively. Details of the NR facility can be found elsewhere in several studies [24, 25, 26]. The schematic diagram of the neutron radiography facility of 3 MW TRIGA Mark II Reactor, AERE, Savar, Dhaka is shown in Figure 4.

3. Experimental Methodology

It's worth mentioning that, this is the experimental work where we detect the homogeneity of this material. The following experiments have been carried out in the present study using direct film neutron radiography technique. They are as follows:

A. Determination of optimum irradiation time for the

present sample. This process done to take an image of the sample by passing neutron through the sample and from the picture, we see the defect or homogeneity of the sample.

- B. Determination of homogeneity or any defects in the sample through optical density variation measurements.

3.1. Pre-Irradiation Methodology for Imaging of the Sample

Before performing the irradiation of the experimental objects, the following steps were undertaken in the present experiment:

1. Sample collection/preparation.
2. Loading the film and converter foil in the NR cassette.
3. Setting the sample in the neutron beam.

3.1.1. Sample Preparation/Collection

Glass-fiber-reinforced polymer composite has been collected from the Halfen Moment India Pvt. Ltd., Ghatkopar West, Mumbai.

3.1.2. Loading the Film and Foil in the NR Cassette

Gadolinium (Gd) metal foil of 25 μ m thickness was used as a converter in the NR cassette and Agfa structruix D4DW industrial X-ray films were used as a detector in our experiment. The films have emulsions in single side only. The sample and the NR camera were placed on their respective tables across the neutron beam. In this position, the camera was placed just after the sample. The sample holder table was set at the optimum sample position from the reactor biological shielding assembly.

3.1.3. Irradiation of the Sample

To find out the optimum irradiation time of the sample, a series of experiments were performed with different exposure time. To do these experiments, the reactor was operated at 3 MW power level. Finally, we found the optimum irradiation/exposure time for the sample. From the observation of the final radiograph, we found out the internal details such as cracks, voids, homogeneity of their compositions etc. of the sample.

3.2. Post-Irradiation Methodology for Imaging of the Sample

Different steps after pre-irradiation like film developing, washing. Fixing, washing, and drying are described in detail elsewhere in several studies [4, 5, 6].

The neutron radiographic images of the sample show that the region in which the sample was at close contact on neutron radiography cassette were light whereas, the backgrounds were comparatively dark. This is because more neutrons were attenuated by the test sample and allowing more neutrons to pass freely through the rest.

4. Methodology to Determine the Optical Density of the Sample

In this work, the term homogeneity means the uniformity in the distribution of the composite materials. The homogeneity of a material depends on the proper distribution of the composite materials. Measuring the optical density of the radiographic film background (without image), the optical density of the center point of the sample image, and at different reference levels of the radiographic image of the sample, one can comment about its homogeneity. The reference points are selected in such a way that almost whole sample is covered when optical densities are measured at these reference points. The best homogeneity is ensured if constant optical density values at all places/levels [25, 26]. The mathematical expression for the optical density, D , at a point of the film/image is given by:

$$D = \ln \left(\frac{A_0}{A} \right) \quad (1)$$

Here, A_0 = response of densitometer without the image and A = response of densitometer with the image. The fractional change in image density of neutron radiograph can be represented by ΔD and the expression can be written as,

$$\Delta D = \left(\frac{D_c - D_n}{D_c} \right) \quad (2)$$

where D_c = Average optical density of the total radiographic image and D_n = Optical density at different positions of the radiographic image. We have measured the optical density, of the neutron radiographic images of the sample by a digital densitometer (Model – 07 -424, S - 23285 Victorian Inc., USA). Densitometry data of optical density of the radiographic image of the sample is shown in Table below.

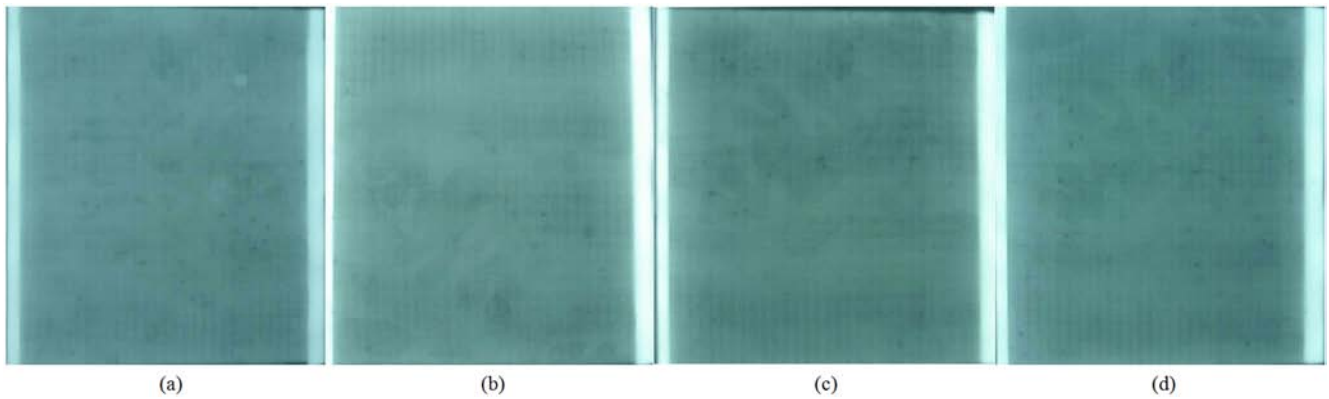
5. Results and Discussion

5.1. Optimization of Irradiation/Exposure Time

The optimum exposure time of the samples was determined by taking a series of experiments with different exposure time. The experiments were done at thermal neutron radiography facility of 3 MW TRIGA MARK-II Research Reactor of AERE, Savar, Dhaka. During the experiment, the reactor was operated at 3 MW power level. 25 micro-meter thick gadolinium metal foil was used as the converter in the experiments. Table: 1 shows that the optimum exposure/irradiation time for the glass fiber-reinforced polymer composite is 40 minutes. In this neutron radiograph, we see that there are no voids or cracks, so the Glass Fiber-Reinforced Polymer Composite is homogeneous.

Table 1. Optimum irradiation/exposure times of the object.

Samples	Irradiation time (minute)	Optimum irradiation time (minute)
Glass-fiber-reinforced polymer composite	45	40
	35	
	50	
	60	
	40	

**Figure 5.** Neutron radiographic images of the sample at (a) 40 min immersion time, (b) 45 min immersion time, (c) 50 min immersion time, (d) 60 min immersion time.**Table 2.** Data of optical density for glass fiber-reinforced polymer composite material.

Samples	Optical density at the center	Average density (D_c)	Optical density at the different positions (D_n)	The fractional change in image density $\Delta D = (D_c - D_n) / D_c$
Glass fiber-reinforced polymer composite	1.88	1.68	1.68	0.000
	1.88		1.68	0.000
	1.86		1.66	0.011
	1.88		1.68	0.000
	1.88		1.68	0.000

5.2. Homogeneity of the Sample

The homogeneity of a material depends on the proper distribution of the composite materials. In the present work, the quality /defects of the samples have been studied by using densitometric measurements from the neutron radiographic images. If the optical density values of the radiographic images are different at different levels, the constituent compounds of the samples are surely not uniformly distributed.

Hence the average value of the optical density difference between film background and the radiographic image of the sample can be introduced as the measuring stick for assessing the defects/quality of a sample. The absence in the variation in image density ensures uniformity of the composites materials of the samples. This proves that in the glass fiber-reinforced polymer composite the associated elemental distributions are uniform or homogeneous. So, the prepared glass fiber-reinforced polymer composite is suitable for use as an alternative to building material.

Table 3. The optical density of the different levels of neutron radiographic images of the samples.

Different level	Optical density					
1	1.88	1.95	1.91	1.84	1.84	1.85
2	1.67	1.61	1.81	1.76	1.61	1.68
3	1.58	1.51	1.63	1.61	1.51	1.53
4	1.53	1.52	1.64	1.64	1.53	1.47
5	1.76	1.89	1.73	1.68	1.73	1.71

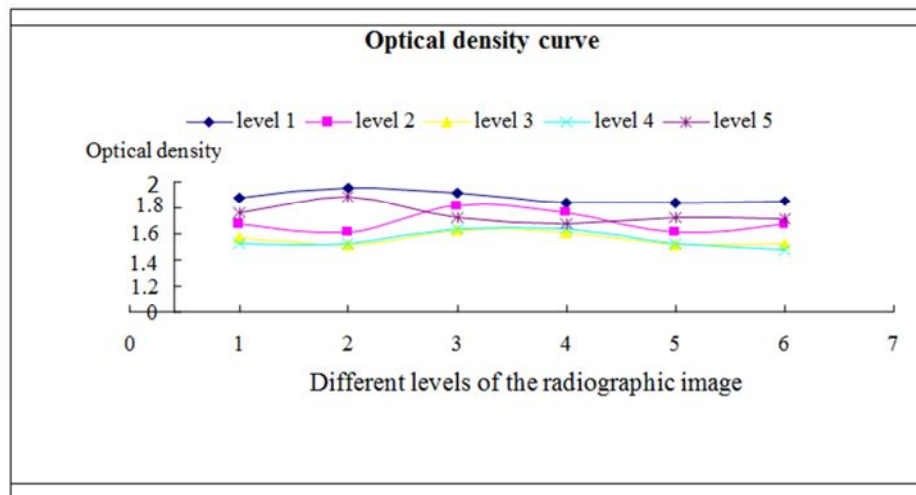


Figure 6. Optical density differences at different levels of the film background and the radiographic image of the glass fiber-reinforced polymer composite

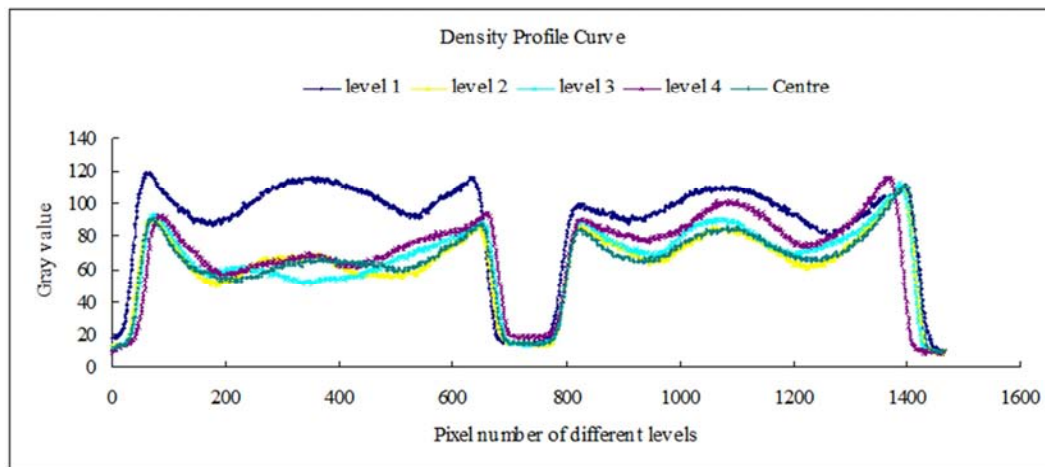


Figure 7. Pixel number of different levels.

6. Conclusion

Glass fibers reinforced polymer composites have been prepared by various manufacturing technology and are widely used for various applications. Initially, ancient Egyptians made containers by glass fibers drawn from heat softened glass. Continuous glass fibers were first manufactured in the 1930s for the high-temperature electrical application. Nowadays, it has been used in electronics, aviation, and automobile application etc. Glass fibers are having excellent properties like high strength, flexibility, stiffness and resistance to chemical harm. The radiograph of glass fiber-reinforced polymer composite is almost clear which shows that the mixture of the constituent elements of the sample is quite uniform. The optical density data also proves the uniformity in mixing the constituent elements in the glass fiber-reinforced polymer composite. From experience in handling the radiographic films and optical density data, we can conclude that the quality of glass fiber-reinforced polymer composite is good and the fabrication of the glass fiber-reinforced polymer composite is homogeneous.

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