Applications of various imaging techniques in neutron radiography at BARC, Trombay

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Abstract
Neutron radiography systems based on Gd, Dy and In metallic foils and X-ray films have been used at this centre since early seventies for various NDT and R&D work in nuclear, defense and aerospace industries. In recent years use of digital detection systems such as neutron scintillator and CCD based imaging systems and photostimulated luminescence based phosphor imaging plate systems have been introduced in our work. This has enabled to achieve higher sensitivities and dynamic ranges of recording radiographs with acceptable spatial resolution. These new systems provide digital image information and are more convenient for quantitative evaluations. At BARC these new techniques have been used in variety of radiography techniques such as conventional neutron radiography (NR), neutron induced beta radiography (NIBR), hydrogen sensitive epithermal neutron radiography (HYSEN) using Apsara, CIRUS and Dhruva reactors as neutron sources. The details of the work done are described in this paper.

Keywords: Neutron Radiography, Neutron Scintillator, CCD based imaging camera, Neutron Image Plates

1. Introduction
The property of thermal neutrons, which makes them valuable for studying industrial components, is their high penetration through widely used industrial materials such as steel, aluminium or zirconium. Neutrons are efficiently attenuated by only a few specific elements such as hydrogen, boron, cadmium, samarium and gadolinium. For example, organic materials or water attenuate neutrons because of their high hydrogen content, while many structural materials such as aluminium or steel are nearly transparent. Neutron radiography is an advance technique for non-destructive testing of materials and is exact analogue of X-ray radiography; a method based on the local variations in absorption encountered by a beam in simple transmission (fig.1). Any inhomogeneity in the object such as internal structure, defects like voids, cracks, inclusions, and porosity will be shown up as a change in the detected intensity recorded behind the object. It is possible to investigate very bulky objects and selectively see those parts with high real or apparent absorption cross-section and also inspects large thicknesses of heavy materials by neutrons in much less time than required by an X-radiograph. Therefore, it has some special advantages in Nuclear, Aerospace, Ordnance and rubber & plastic industries [1].

2. Detection techniques in Neutron Radiography:
The function of the imaging techniques in neutron radiography is two fold: Image detection and image recording. The image detectors convert the transmitted neutrons and intensify the
emerging radiation. The image recorders store and/or display the radiographs. Image detectors are made of either metal foil with high absorption cross section for neutrons, like Gadolinium, Dysprosium, Indium, or Boron ($^{10}$B) or Lithium ($^7$Lii) compounds coated on plastic foils or directly on to the film. The scintillator/phosphor screens loaded with gadolinium, boron and lithium are commonly used as image detectors in image plate and electronic imaging methods [2-4].

3. Neutron Radiography Facility at BARC

At BARC, neutron radiography has been actively pursued by Solid State Physics Division. Neutron radiography systems based on Gd, Dy and In metallic foils and X-ray films have been used since early seventies for various NDT and R&D work in nuclear, defense and aerospace industries [3]. In recent years use of digital detection systems such as neutron scintillator and CCD based imaging systems and photostimulated luminescence based phosphor imaging plate systems have been introduced in our work[4,5]. This has enabled to achieve higher sensitivities and dynamic ranges of recording radiographs with acceptable spatial resolution. These new systems provide digital image information and are more convenient for quantitative evaluations. These new techniques have been used in variety of radiography techniques such as conventional neutron radiography (NR), neutron induced beta radiography (NIBR), hydrogen sensitive epithermal neutron radiography (HYSEN) using Apsara, CIRUS and Dhruva reactors as neutron sources. Prior to the utilization the digital detection systems have been characterized for the performance.

In the Apsara (400 kW, swimming pool type reactor) facility (fig. 2a) thermal neutrons from the reactor were collimated by divergent, cadmium lined aluminum collimator with a length/inner diameter (L/D) ratio of 90. A cadmium-lead shutter facilitated the opening and closing of the beam. The object to be radiographed was mounted about 60 cm from the collimator end followed by a cassette containing neutron converter and X-ray film. The neutron flux at the object was $\sim 10^6$ n/cm$^2$.sec.

CIRUS is 40 MW$_{th}$ natural uranium fuelled, heavy water moderated tank type thermal reactor with maximum neutron flux of $6.5 \times 10^{13}$ n/cm$^2$/sec The NR facility [5] is installed at this reactor and is shown schematically in figure 2(b). The thermal neutrons are collimated using a collimator of length 120 cm, installed in the beam hole such that the end face of the collimator flushes with the biological shield surface. The collimator is made up of MS material and its outer and inner diameters are 132 mm and 100 mm respectively. A cavity of 50 cm square and 75 cm length is provided in front of beam hole for locating the sample and devices of the experiment. The sample to be radiographed is mounted about 60 cm from the end of collimator followed by the recording devices. Neutron flux of $\sim 3.3 \times 10^8$ n/cm$^2$/sec and beam size of diameter 15 cm is available at the sample position. The opening and closing...
of the neutron beam tube is facilitated using the inner and outer gates of the beam tube which are motorized and are operated remotely from the desk top. The important parameters of these two NR facilities are given in table 1.

<table>
<thead>
<tr>
<th>Beam parameters</th>
<th>Apsara Reactor</th>
<th>CIRUS reactor</th>
</tr>
</thead>
<tbody>
<tr>
<td>Useful beam area</td>
<td>15 cm dia.</td>
<td>15 cm dia.</td>
</tr>
<tr>
<td>Collimator</td>
<td>Divergent type</td>
<td>Parallel type,</td>
</tr>
<tr>
<td></td>
<td>Length 225 cm,</td>
<td>10 cm ID, 13 cm OD, 120 cm long</td>
</tr>
<tr>
<td></td>
<td>aperture 2.5 cm</td>
<td></td>
</tr>
<tr>
<td></td>
<td>L/D ratio: 90</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>1 x 10^6 n.cm^-2.sec^-1</td>
<td>3.3 x 10^8 n.cm^-2.s^-1</td>
</tr>
<tr>
<td>Cadmium ratio</td>
<td>6.3</td>
<td>8.7</td>
</tr>
<tr>
<td>N/γ ratio</td>
<td>9 x 10^5 n/cm^2/mR</td>
<td>~ 3.6 x 10^5 cm^-2.mR^-1</td>
</tr>
<tr>
<td>NR methods</td>
<td>Gd 50μm, Dy 100 μm, Kodak CN85-B converter screens, (^6)LiF-ZnS(Ag) scintillator/CCD camera based imaging system, X-ray &amp; neutron image plates.</td>
<td>Dy 100μm, In 200μm converter foils,(^6)LiF-ZnS(Ag)scintillator/ CCD camera based system, Fujifilm neutron and X-ray image plates.</td>
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4. Applications with Apsara NR facility:

The NR facility at Apsara was used for a variety of applications in nuclear, aerospace, defense and metallurgical industries [3]. The facility has been extensively used for recording neutron radiographs of experimental fuel elements, water contamination in marker shell loaded with phosphorous, electric detonators, satellite cable cutters and pyro valves, boron-aluminium composites, hydride blisters in irradiated zircaloy pressure tubes, variety of hydrogenous and non hydrogenous materials and two phase flows in metallic pipes. Some of the radiographs are shown in Fig.3.

![Figure 3. Neutron Radigraphs of a) satellite cable cutters, b) pyrovalve, c) fuel pins, d) flower plant partly covered with lead brick (Apsara facility, Gd+X-ray film).](image)

4.1 Neutron Radiographic Investigations of Hydride Ingress in Zr based Pressure Tube Materials

One of the most important applications of NR in the nuclear field is the post irradiation examination of pressure tube (PT) to check formation of hydride blisters if any. To ascertain the detection of hydride blisters in zircaloy pressure tubes, detectability limit of hydride was first established. For this purpose hydride blisters were created in the laboratory on hydrogen charged zircaloy pressure tubes under thermal gradient are examined using neutron radiography. It was established that a zirconium hydride blister of nearly 0.35% of job thickness could be detected using neutron radiography. The theoretical detectability was also analyzed and found to be in good agreement with the experimental results [6]. This work served as reference to examine an irradiated pressure tube from a power reactor [7]. Neutron radiographs of an irradiated pressure tube [Rajasthan Atomic Power Station (RAPS#2, K-7 position, 8.25 EFPY) sample were recorded using Apsara NR facility after modifying it for...
handling the radioactive zircaloy-2 pressure tube coupons. The PT sample of size 59 mm x 29 mm x 4.2 mm was radiographed using transfer technique with 100 µm thick Dysprosium converter screen. A PT strip containing four laboratory generated hydride blisters were also mounted on the same converter screen cassette to act as a reference.

![Figure 4](image)

Figure 4. (a) Hydride blisters in zircaloy-2 (L) and Zr-Nb(R) PT coupons (Lab. Generated) and (b) Their NR, (c) Hydride blister streak in zircaloy-2 PT coupon (L) from a power reactor. Enlarged view of the blister shown to the right (d) NR of zircaloy-2 PT coupons with neutrons incident parallel (L) and perpendicular (R) to the plane of blisters.

Fig. 4(a) shows pictures of laboratory generated hydride blisters in zircaloy-2 and Zr-2.5% Nb pressure tube coupons where as Fig. 4(b) shows neutron radiographs of these blisters. Fig. 4(c) shows hydride blister streaks in the zircaloy-2 coupon of the pressure tube from power reactor RAPS#2. Enlarged view of one of the blisters is also shown to the right of the figure. Neutron radiography was also used to study size and shape of the zirconium hydride blister in the zircaloy-2 pressure tube. Fig. 4(d) shows neutron radiographs of a pressure tube with three laboratory generated hydride blisters and with neutron beam incident parallel and normal to the plane of the blisters respectively. It shows lenticular shape of the blister with nearly 2/3 of the blister embedded in the wall of the tube. In the present photograph maximum width of the blister corresponds to 1.5 mm in 4 mm thick wall of the pressure tube. However, the smallest blister grown in the laboratory was found to be mainly on the outer surface of the pressure tube with almost no penetration in the wall of pressure tube [6].

4.2 Neutron Radiographic Investigations of Hydride Blisters Grown on Zr-2.5Nb Pressure Tube Spool Piece under Simulated Condition of in-Reactor Pressure and Temperature.

This work was carried out with an aim to have a thorough understanding of the mechanism of blister formation and the resulting degradation in the serviceability of the component under simulated in-reactor condition of pressure and temperature [8]. A 220 MWe Indian PHWR Zr-2.5Nb pressure tube spool piece of 165 mm length was charged with a homogeneous hydrogen concentration using lithium hydroxide solution at 300 °C followed by growth of multiple hydride blisters at 10 different locations on the pressure tube outside surface by maintaining cold spots. This blistered tube was subjected to burst test by pressurizing the tube in steps of 20 kg/cm² for 10 minutes up to 380 kg/cm² at which it burst with sound. The failed tube was examined for characterizing the cracked and un-cracked blisters by neutron radiography using transfer technique with 50 µm Gd neutron converter screen. The hydride blisters were detected at all the ten cold spot locations in various shapes and sizes depending on the size of the cold tips touching the surface of the pressure tube. The unique feature of the pressure tube bursting was that a crack is extending from one end of the tube to the other passed through the middle of two blisters, namely 4A and 4B falling in one line. Figs. 5&6 show the photographs of some of the blisters on the tube and the axial crack passing through two cracked blisters 4A and 4B and their neutron radiographs. The depth of this blister was measured to be ~ 600 µm. The blister 4B appears to be of circular with dia. ~ 2 mm.
It appears that blister of depth 600 µm can crack at internal pressure of 380 kg/cm² which gives rise to hoop stress of 4750 kg/cm². The blisters having depth less than 600 µm will require higher stress of cracking.

4.3 Digital Neutron Imaging:

An electronic imaging system (fig.7) was developed for static and dynamic radiography and tomography to use with neutron radiography facility at APSARA reactor [9]. The beam of neutron after passing through the sample is absorbed in a scintillator screen (NE-426). Photons generated by the scintillating screen are reflected by 90° and focused onto the input fibre optic face of an image intensifier tube. The output image is focused onto a CCD camera using a F1.4 lens. The CCD camera has 756 (H) x 581 (V) pixel array and image intensifier has 30 lp/mm resolution with gain of $10^5$ Cd/m²/lx. The video output is connected to PC with frame grabber and processed using onboard processor. Fig. 8 shows some of the radiographs taken with this system. Tomography of several objects (fig.9) and experiments for visualization of water/air flow inside metallic pipes (fig. 10) were performed using this system [9,10].
5. Applications with CIRUS Neutron Radiography Facility:

The CIRUS NR facility has advantage of higher neutron flux available ($\geq 10^8$ n/cm$^2$.sec, 2 orders more than at Apsara reactor) for recording radiographs and electronic images with faster speed (~ 100 times) and enhanced resolution and using thicker objects compared to that at Apsara. Neutron radiographs of thick walled copper tube filled with wax, INSAT cable cutter, automobile carburetor, spark plug, Zr-Nb2.5% plate with hydride blister have been recorded using transfer technique (Dy foil converter). An exposure of 9 minutes to Dy converter and activity transfer of 4.5 minutes to X-ray film is sufficient to get good quality neutron radiographs [5]. Some of these radiographs are shown in figure 11.

5.1 Electronic Imaging Method (Real Time Neutron Radiography):

An in house built electronic imaging system was developed (figs. 12(a) and (b)) for use at the NR facility at CIRUS reactor [5]. The radiographs shown in 12(c) were recorded with neutron scintillator of size 18 cm x 24 cm and a high resolution monochrome CCD camera (pixel resolution 580H x 350V TVL) without image intensifier. There was no need of image intensifier to record the radiographs; neutron flux of $3.3 \times 10^8$ n.cm$^{-2}$.s$^{-1}$ at sample position was good enough to get clear images. The imaging system was also used for recording real time radiography for study of two phase flow in the metallic pipes. Figure 13 shows example of such a study. We have also procured neutron imaging system from Photonic Science, UK which is also being used for real time radiography. Figure 14 shows some of the radiographs using this system.

6. Applications of Neutron Image plate

Imaging plate (IP) technology enables a direct means of recording the distribution of intensity of radiation and has replaced conventional X-ray film in routine X-ray crystallography,
radiography and studies involving the use of synchrotron radiation sources [11,12]. The advantages of this technology include high spatial resolution, high sensitivity and a linear response to radiation dose over five orders of magnitude. Commercially available IP systems have a read-out spatial resolution ranging from 25 to 200 µm and the plates are available in sizes up to 100 x 240 mm to 350 mm x 1520 mm for X-rays and 200 mm x 250 mm to 200 mm x 400 mm for neutrons. We have employed neutron image plate BAS-ND2025 and an off-line scanner Fujifilm BAS-5000 for NR work [13]. A neutron radiograph of INSAT cable cutter and a copper U-tube filled with wax is recorded using Apsara reactor. The image quality of the radiographs recorded with neutron image plate is nearly same compared to that of Gd/X-ray film technique (fig.15) but the speed of recording the radiograph is 40 to 50 times faster with neutron image plate. The PSL intensity linearly increases with increase in exposure time and the range is much higher than for X-ray film. The image brightness and contrast can be controlled with the software provided to take care of under/over exposure of the image plate. In addition there is no need of dark room and wet chemical processing in image production. Thus radiography with image plates is a fast and convenient process.

Figure 12. (a) In house built CCD based camera mounted at CIRUS NR facility, (b) the sample mounted on the back of the neutron scintillator, (c) Electronic images of INSAT cable cutter and Cu-tube filled with wax.

Figure 13. Sequence of Electronic Images of evaporation and boiling of water in a thin walled 1” diameter Al-tube, recorded using the in house built imaging system.

Figure 14. Electronic images of jet turbine blade and INSAT cable cutter and Cu-tube filled with Wax, recorded using Photonics imaging system. Radiographs shown are background correction.
6.1 Neutron induced beta radiography

The neutron induced beta radiography (NIBR) is a very useful technique for non-destructive evaluation of internal structures of thin samples with thickness up to few hundreds of μm [14]. It has many industrial applications to test thickness, uniformity and defects in the manufacture of paper, metal and plastic films. It can be performed at any standard neutron radiography facility with available infrastructure. NIBR makes use of thermal neutron activated Dy or In foils as source of beta with energies 1.28 MeV and 1.0 MeV respectively. The technique is similar to the transfer technique used in neutron radiography. Radiographs are obtained with an aluminium cassette containing image plate (IP), a sample under inspection and the neutron irradiated Dy or In foil kept in tight contact with each other. The irradiation of the foil was performed at the CIRUS NR facility. An exposure of ~ 9 minutes for the Dy foil and ~ 5 minutes for In foil was enough to get the required activity in the foil for the radiography experiment. The radiographs were recorded for an exposure time of 1 to 1.5 minutes with a delay of ~15 minutes after neutron irradiation of the foil. To demonstrate applications of the technique, variety of samples like Indian currency bill of `1000, postal stamps of Indian paintings and personalities, painting on cotton sheet are radiographed.

Figure 16 shows neutron radiographs of a BNC connector, turbo jet blade and INSAT cable cutter recorded using neutron image plates at CIRUS NR facility. The flux at CIRUS facility was 2 orders more than at Apsara reactor which made recording of electronic images without image intensifier and of thicker objects.
Radiograph of the currency note (figure 17) clearly shows the impresses of water marks, hidden denomination value and writings and other security marks not seen in visual inspections. Contrast due to minute thickness and printing colour variations across the postal stamps (painting) is clearly seen in the radiograph (fig.18). These features are not at all visible with the conventional neutron radiography. The NIBR is also performed using In foil. Figure 19 shows radiograph of banyan tree leaves recorded on image plate with an exposure of 1.5 minutes.

6.2 Hydrogen Sensitive Epithermal Neutron radiography

The technique, Hydrogen Sensitive Epithermal Neutron (HYSEN) radiography was first developed [15] for imaging small amounts of hydrogenous materials encapsulated within high thermal neutron absorbers and found to be useful in study of hydride-induced embrittlement of metals. The HYSEN imaging system [16] consists of a converter screen (In) and a neutron beam filter (In + Cd) with the object placed between the screen and filter. For neutrons, In has a resonance peak at 1.49 eV. Combination of Cd foil with In foil almost completely cuts off neutrons with energy lower than 1.49 eV. Incident neutrons with energy higher than 1.49 eV, which pass through, are scattered elastically by hydrogen atoms present in the object and are slowed down to the vicinity of 1.49 eV. They are absorbed by the second In foil placed behind the sample. The image induced the foil represents signature of hydrogen present in the sample and the grey intensity its concentration. Hydrogen concentrations as low as 50 ppm (0.020 mg H/cm$^2$) in 0.62 mm zircaloy coupons have been reported[15]. The detection limit of standard NR techniques is about 0.66 mg H/cm$^2$. Neutron radiograph of 1-, 2-, 3-, 4- layers of cellophane adhesive tape as hydrogenous object was recorded. The gradation of hydrogen concentration in successive layers of adhesive tapes is clearly seen in the radiograph (Fig.20).

![Figure 20](image)

**Figure 20.** (L) Neutron radiograph of 1-, 2-, 3- 4-layers of cellophane adhesive tape as an object, (R) Gradation of hydrogen in successive layers adhesive tapes as PSL intensity.

7. Conclusions

At BARC various imaging techniques have been used in variety of radiography techniques such as conventional neutron radiography (NR), Digital and Real time radiography, neutron induced beta radiography(NIBR), hydrogen sensitive epithermal neutron radiography (HYSEN) etc using Apsara, CIRUS reactors as neutron sources.

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