NEUTRONS FOR THE STUDY OF ADHESIVE CONNECTIONS

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Abstract: Adhesive bonding or sealing applications are increasingly used in many technical areas: for example in mechanical engineering, in the car industry, the aircraft industry, wood or paper industry, etc. The advantages of this method are manifold if it is used correctly. There is a growing demand for non-destructive testing of new bonding products and applications. Contrary to X-ray, thermal and cold neutrons are very sensitive to thin layers of glue (containing hydrogen) within metallic or other non-plastic materials. This is due to the much higher linear attenuation coefficient of hydrogen for thermal neutrons compared to X-ray's with energies above 10 keV. We used thermal neutron radiography/tomography for the analysis of glue distributions in redesigns of various car components. The glue distribution can be visualized with high spatial resolution in a radiography image or in a 3D tomography volume. Dynamic neutron radiography can be used to study the propagation of adhesives injected into carbon fiber fabrics when forming aircraft or car components by the resin transfer molding technique. One major disadvantage of neutron radiography compared to X-ray is due to the source of radiation, which is available only at research fission reactors or spallation neutron sources. Therefore neutron non-destructive evaluations are mainly performed for prototype sample or process optimization purposes. Together with typical sample investigations, we present the spatial resolution and material sensitivity characteristics of the thermal neutron radiography beam line NEUTRA.

Introduction: Neutron radiography (NR) is a non-destructive evaluation method known since many decades i.e. since the early 1960's. Its application however has been limited to the rather few places, where neutron sources i.e. nuclear research reactors or accelerator driven neutron sources like the SINQ spallation neutron source exist. Neutron radiography is based on the same principles as the conventional X-ray technique, which due to the availability of powerful, small X-ray sources has found wide spread application in the medical and industrial field. Both methods can be used either in 2D mode to take radiographs or with a tomography setup in 3D mode. Neutron radiography and tomography however is limited to non-living matter investigations because the neutrons are strongly attenuated and highly ionizing when traversing tissue. In the following we shortly review the main differences between X-ray and neutron imaging. Contrary to X-ray's which interact mainly with the electrons in the shell of the atoms, neutron matter interaction occurs with the atomic nuclei via very short range forces. That's why X-ray attenuation coefficients increase steadily with atomic number Z - which is equal to the number of electrons in the atomic shell - of the material under investigation [1]. Light elements as found in tissue or plastic material are penetrated easily, but metallic elements cause strong X-ray attenuation or need higher X-ray energy for reasonable transmission.

Thermal or cold neutrons however have quite opposite behavior as shown in Figure 1. Especially hydrogen (due to its high neutron scattering cross-section) stops neutron propagation effectively, whereas many metallic materials (even heavy metals like lead and uranium) are penetrated easily by neutrons. It is this kind of complementary attenuation characteristic, which makes neutron imaging a valuable tool for non-destructive evaluation.



Fig. 1: Comparison of mass attenuation coefficients (left), camera by X-ray (top), neutron radiography (bottom).

Besides the radiation attenuation properties, there are however other important differences between neutron and Xray imaging. X-ray source spots can be made quite small. This allows a magnifying mapping of small objects and spatial resolutions of a few μ m (or even sub μ m) resolution using synchrotron sources). Additionally, the X-ray energy can easily be adapted to the sample size and composition. Neutron source geometry is rather large. Well collimated neutron beams using a pin-hole optic result in low neutron flux intensities. Most neutron sources deliver only polychromatic thermal (mean energy ~ 0.025 eV) or cold neutrons. These are suited best for imaging purposes. A neutron energy selection is possible, but at the expense of even lowers neutron flux intensity. Modern X-ray and neutron area detectors use almost the same technology, the only difference being the type of scintillator material. Due to the above-mentioned neutron source characteristics most neutron radiography inspections are performed on of larger objects. Typical sample dimensions are in the range of some cm up to 20 cm (depending on object penetrability), which leads to spatial resolutions in the order of tens of μ m up to 1 mm (typical 100 – 400 μ m/pixel).

Adhesive joints consist of a thin layer of glue i.e. hydrocarbon compounds like epoxy or polyurethane resins, etc, which are in tight contact with the surface of the structures to be connected. A radiographic inspection of an adhesive bond should therefore have a high sensitivity for thin layers of hydrogenous material. This is exactly the case for thermal neutrons as shown in Figure 2. Glue has a 2-3 order of magnitude higher linear attenuation coefficient for neutrons (2.9 [1/cm]) than for X-ray's. Missing glue even in thin layers therefore shows with high contrast in neutron radiographs. At the same time metallic structural materials have similar or even better neutron transmission characteristics than X-ray's.



Fig. 2: Comparison of X-ray (μ) and thermal neutron (Σ) linear attenuation coefficients for glue and structural materials.

Results: We now show several examples of 2D or 3D neutron radiographic inspections of adhesive connections. We used the thermal neutron radiography facility NEUTRA (<u>http://neutra.web.psi.ch/</u>) at the spallation neutron source SINQ (<u>http://sinq.web.psi.ch/</u>) of the Paul Scherrer Institute (PSI), Villigen, Switzerland. Main parameters of the NEUTRA beam line are given in Table 1. Figure 3 indicates the 3 measuring positions.



Fig. 3: Overview and layout of the PSI neutron radiography facility NEUTRA.

Standard detector is a Peltier cooled slow scan CCD camera DV 434 with a 1024 x 1024 pixel chip from Andor Technology (<u>http://www.andor-tech.com</u>), which registers the light emitted from a 0.2 mm thick ⁶Li doped thermal neutron sensitive screen from Applied Scintillation Technologies (<u>http://www.appscintech.com/</u>). Various camera objectives are used for the active field of view ranging from 270 x 270 mm down to 35 x 35 mm. High

resolution 2D radiographs are taken by Fuji (<u>http://home.fujifilm.com/products/science/basms/</u>) neutron sensitive imaging plates providing a imaging area of 200 x 400 mm with a spatial resolution of 50 µm.

Measuring Position	1	2	3
Distance from aperture [mm]	3820	7292	10547
Beam diameter [mm]	150	290	400
Neutron flux $[cm^{-2} s^{-1} mA^{-1}]$	1.6e+07	5.0e+06	3.0e+06
Collimation ratio L/D	200	350	550
Geometric unsharpness [mm] (s=80mm)	0.4	0.2	0.16

Tab. 1: Main performance parameters of the NEUTRA facility (actual proton current ~1.2 mA).

The first example is a car air intake manifold made from two half shells, which were glued together. The neutron radiography inspection of the adhesive joint shows the glue distribution in very detail (see Figure 4).



Fig. 4: Air intake manifold: NR overview taken by CCD camera (left) and detail by imaging plate (right).

The NR inspection of this test prototype revealed where there were bad spots in the glued joint, possibly inducing leakage. The size of the sample was too big to fit into the field of view of one imaging plate. It therefore had to be scanned by moving it on a remotely controlled sample positioning table. The exposure time for one imaging plate radiograph was 15 seconds.

In order to visualize the 3D distribution of the glue in a critical area of the joint, such regions of interest had to be cut out for a tomography inspection [2]. The cut out was fixed on a rotary table and neutron radiography images were taken while the sample was rotated in angular steps of 0.75 degree over 180°. After normalizing each projection for neutron exposure and eliminating inhomogeneities of the scintillator and illuminating neutron field by flat field correction, plane slices perpendicular to the rotation axis were then reconstructed by "filtered backprojection". The resulting 3D volume gives a mapping of the neutron attenuation coefficients in the sample. Special rendering software VG Studio Max (<u>http://www.volumegraphics.com/</u>) allows to segment the sample into structure material and glue (see Figure 5) and to quantify the thickness of the glue layer or to measure the surface area, where glue is missing. Although the tomography inspection is much more time consuming, it allows a better visualization of the sample and a more quantitative analysis of the data.



Fig. 5: Segmented glued joint of cut out from car air intake manifold.

A second similar example from the car industry too is the radiographic inspection of glued car doors. There is a trend towards using light weight materials for such car structures. Sometimes car safety asks for the combined use of steel and light metal parts. Here adhesives technology has some advantages over conventional welding technology or using blanks. Neutron radiography was used to test prototype samples of glued car doors.



Fig. 6: Neutron radiography of a car door. Enlarged area on the left of interest shows a part of the glued joint.

The car door was fixed on the sample positioning table of measuring position 3 at the NEUTRA radiography facility. Single frames with a 279 x 279 mm field of view were taken using the slow scan CCD and 20 seconds exposure time. This provided an available dynamic range of ~ 12000 grey levels in a neutron radiograph. Performing multiple image alignment an overview radiograph of the whole car door could be provided. The main area of interest is the glue distribution on the border of the door. On the right of Figure 6 a contrast enhanced enlarged area of the border is shown. The darker grey values indicate increasing glue layer thickness. There are many voids shown in the glue layer, which might facilitate corrosion attack. Neutron radiography inspections on selected doors were performed after manufacturing and some time later after a forced corrosion attack simulation test. The aim of the neutron radiography inspections of door prototypes was to find an appropriate gluing procedure, which would guarantee a long enough lifetime of the door.



Fig. 7: Neutron tomography of cut out border of glued car door: outside view (left), glue distribution (blue).

A third example of neutron radiography inspection of a glued connection from car industry was performed on a prototype of a passenger cab for a sports car. This prototype part was manufactured from a light metal honeycomb structure glued to aluminum girders. The solidity of the structure depends critically on the glue distribution around the girders. Neutron radiographs were taken by exposing neutron sensitive imaging plates for 15 seconds. The epoxy glue resin shows in the dark regions around the girders. A neutron radiographic inspection of critical regions of the whole passenger cab however had to be abandoned due to missing space at position 3 of the NEUTRA measuring hatch (see Figure 3).



Fig. 8: High resolution (50 µm/pixel) neutron radiographs of glued honeycomb structure.

A fourth example of adhesive material investigation using neutron radiography deals with the resin injection process in the resin transfer molding technology. It is applied for the manufacturing of carbon fiber reinforced composites structures used in the airplane or car industry.



Fig. 9: Dynamic neutron radiography series of resin injection into carbon fiber textile layers.

In this technology carbon fiber textile layers are put into molds and resin is injected at different locations under high pressure. The flow of the adhesive resins through the carbon fiber fabric can be simulated. The dynamic neutron radiography measurements of resin flow through a plane model mold provided quantitative data about the resin permeabilities in different types and layer combinations of such fabrics [3]. Multiple layers of carbon fiber fabrics (fibers arranged in one direction or bidirectional) are pressed between 2 aluminum plates with the dimension 400 x 400 x 20 mm. The fiber volume content or thickness is controlled by spacers put between the plates. Resin is injected through a central bore at constant flow rate or pressure. Pressure and flow rate are measured simultaneously with taking neutron radiographies of the elliptical expanding resin flow front. A detailed analysis of the time course of the expanding flow front along the main axes of the ellipse provides information about the effective resin permeabilities.

Conclusions: Thermal neutron radiography and tomography is a valuable tool for the non-destructive evaluation of adhesive connections. This is due the high sensitivity of thermal/cold neutrons for small quantities of hydrogenous material. Unfortunately high performance neutron radiography beam lines are available only at few places around the world. Therefore mainly industrial prototypes or new production processes are investigated, where neutron imaging has distinct advantages over conventional NDT procedures like X-ray, ultra-sound, thermography, etc available at the production location or at the place of installation. Neutron imaging can be used as a validation tool for these conventional techniques. The neutron imaging of adhesive connections presented in this article is only a small selection of adhesive applications in the diversified and rapidly expanding technology of adhesive of bonding.

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