Low and high neutron energy for damage detection by chemical composition analysis and residual stress measurements

Alba Zanini[1,a], Oscar Borla[1,2,b], G. Vivaldo[1,c]

[1]National Institute for Nuclear Physics

Via Pietro Giuria 1 – 10125 Torino, Italy

[2]Politecnico di Torino, Department of Structural Engineering & Geotechnics

Corso Duca degli Abruzzi 24 – 10129 Torino, Italy

[a] zanini@to.infn.it, [b] oscar.borla@polito.it, [c] vivaldo@to.infn.it

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Abstract. The use of neutrons as an investigative tool has received considerable attention over the last few years. Neutrons, due to their capability to penetrate thick layers of materials, are particularly suited to investigate inside or beneath the surface of an object without damaging it, determining structure at the microscopic scale, thus providing fundamental information. In particular, neutron measurements such as Neutron Diffraction (ND) or Neutron Tomography (NT), can study structural characteristics like composition, presence of alteration, inclusions, structure of the bulk, manufacturing techniques and presence of those elements which give us an overall fingerprint of the object's characteristics.

A neutron beam impinging onto any heterogeneous object is differently transmitted depending on neutron energy and on thickness, density, chemical composition and total cross section of the material along the line of sight. Recording the transmitted beam is possible to reconstruct the internal feature of the objects. Contrary to the photon case, a neutron beam can transmit through centimetres of metal but it is easily attenuated by small amounts of light elements like hydrogen, boron and lithium. The investigation of moisture and corrosion, the detection of explosives and adhesive connections and the inspection of defects in objects or in thick metallic samples are examples where neutron can be utilized favourably. For this reason neutron analysis is an unique tool for non-destructive testing with multidisciplinary applications.

Furthermore, they can be used for physical problems such as residual stress measurements, study of mechanical behaviour in materials, archaeometry and cultural heritage.

Introduction

The use of neutrons to investigate the fundamental properties of materials began in the 1940s. The pioneering applications [1,2] were limited to studies of the physical properties of matter, and in particular to phase transitions, magnetic structures and especially the hydrogen bond. In the last three decades the use of neutrons has vastly expanded following the development of new technologies for the production of thermal and epithermal neutrons. Neutrons with wavelengths of the order of angstroms are able of probing molecular structures and find applications in a wide array of scientific fields, including biology, cultural heritage materials, environmental sciences, engineering, material sciences, mineralogy and solid state and soft matter physics (figure 1).

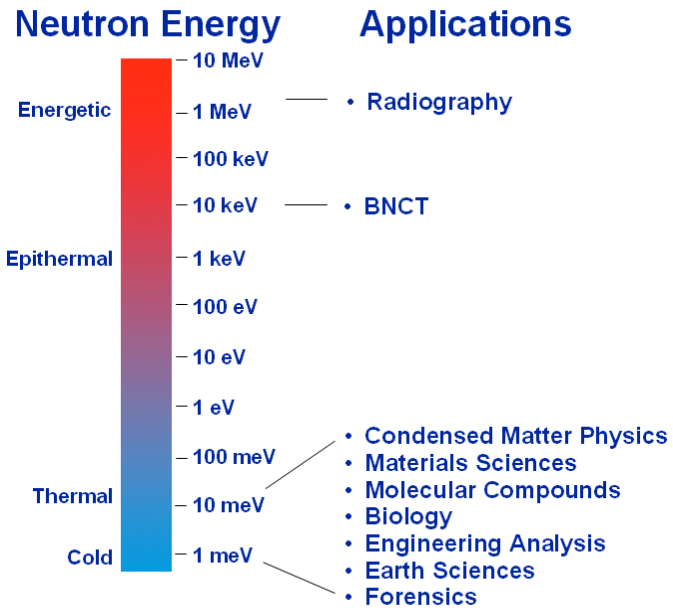

Figure 1: neutrons for pure and applied science.

The special nature of neutron interaction with matter provides important complementary and supplementary data to other techniques.
The large penetration depth and selective absorption of neutrons make them a powerful tool in NDT (Non Destructive Testing) of materials. For example, the residual stress formed in a material during manufacturing, welding, utilization or repairs can be measured by means of neutron diffraction. In fact neutron diffraction is the only NDT method, which make possible a 3D mapping of residual stress in a bulk component. The experimental techniques are described in this paper.

Neutron diffraction
Neutron scattering is the most suitable method for resolving 3D samples and it mainly consists in Neutron Diffraction (ND). ND [3] is based on Bragg law[1] and allows to resolve matter crystallographic structures, determining the atomic and/or magnetic structure of a material. Moreover ND can be applied to study crystalline solids, gasses, liquids or amorphous materials. The method requires irradiating the analyzed sample with a collimated beam of low energy (cold or thermal) neutrons. The revealed intensity pattern gives information about the material structure.
Neutron diffraction uses neutrons generated by fission or spallation. The first is mostly employed in steady-state nuclear reactors while the second usually in pulsed sources. In both cases the neutrons produced are moderated until to the thermal energy range, i.e. $\lambda \geq 0.05$ nm.
When a neutron beam of wavelength λ, comparable with the inter-planer spacing d_{hkl}, impinges a crystalline material, a diffraction pattern is observed and the position of each plane (hkl) is obtained by the Bragg law:

$$2d_{hkl}\sin\theta_{hkl} = \lambda$$

Figure 2 shows $2\theta_{hkl}$ angle related to Bragg peak and is linked to the direction of the incident

[1] Bragg's law gives the angles for coherent and incoherent scattering from a crystal lattice.

neutron beam. Thus, all d_{hkl} are established from the angle θ_{hkl} at which the reflection is detected.

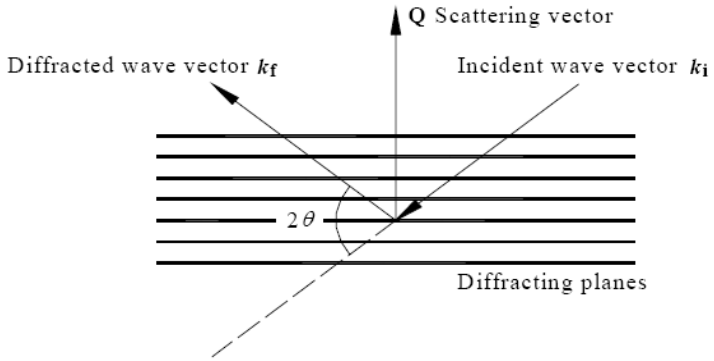

Figure 2: Schematic illustration of Bragg scattering.

Neutron diffractometers

A polycrystalline sample consists of small (few μm) crystallites randomly oriented with respect to each other. When a monochromatic radiations strike a sample, the diffraction from a Bragg plane results in a cone shape, the Debye Scherrer cone, with semi-vortex angle 2θ. The intensity profile is recorded as a circle on a two dimensional detector (Figure 3).

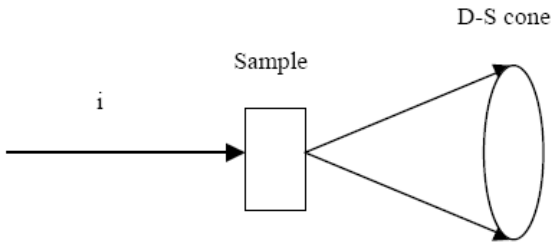

Figure 3: Diffraction from polycrystalline sample in a Debye Scherrer cone

The polychromatic neutron beam is first monochromated to a chosen wavelength by diffraction from a suitable monochromator. The divergence and size of the monochromatic beam is suitably adjusted using appropriate neutron optical devices and is then diffracted from the specimen. In a similar way, the diffracted beam is shaped using suitable optical devices, before being captured by the neutron detector. The gauge volume over which the strain measurement is made is given by the intersection of the incident and diffracted beams (Figure 4).

Strain measurement and determination

The strain is measured towards the scattering vector, Q = kf - ki, which splits the angle between incident and diffracted beams and is perpendicular to the diffracting planes, as shown in Figure 2. Lattice spacing is determined from the measured angular position of the diffraction peak (Bragg reflection) by irradiating the specimen with a monochromatic collimated neutron beam. If the specimen contains no strain, the lattice spacing is the strain free (stress free) values for the material and are denoted by $d_{0,hkl}$. In a stressed specimen, lattice spacing is altered and a shift in each Bragg peak position occurs and the elastic strains then are given by:

$$\varepsilon_{hkl} = \frac{d_{hkl} - d_{0,hkl}}{d_{0,hkl}} = \frac{\Delta d_{hkl}}{d_{0,hkl}} = \frac{\sin\theta_{0,hkl}}{\sin\theta_{hkl}} - 1$$

The angle $\theta_{0,hkl}$ is the angle at which Bragg peak is observed from the strain free reference.

Since ND can measure the elastic strain within a defined volume in a crystalline solid, it is possible to calculate the mean stress in that volume.
Full determination of the strain tensor requires measurements of the elastic strain in at least six independent directions. If the principal strain directions within the specimen are known, measurements along these three directions are sufficient. Measurement along one direction only is needed in the case of uni-axial loading.
Stresses and strains in a specimen are usually direction and position dependent. This leads to measure strains at a number of locations in more than one direction, so an accurate positioning of the specimen with respect to the collimated neutron beam and the detectors is required. Thus, linear translation and/or rotation of the specimen are needed. By successively moving the specimen through the gauge volume the stress spatial variation can be mapped.

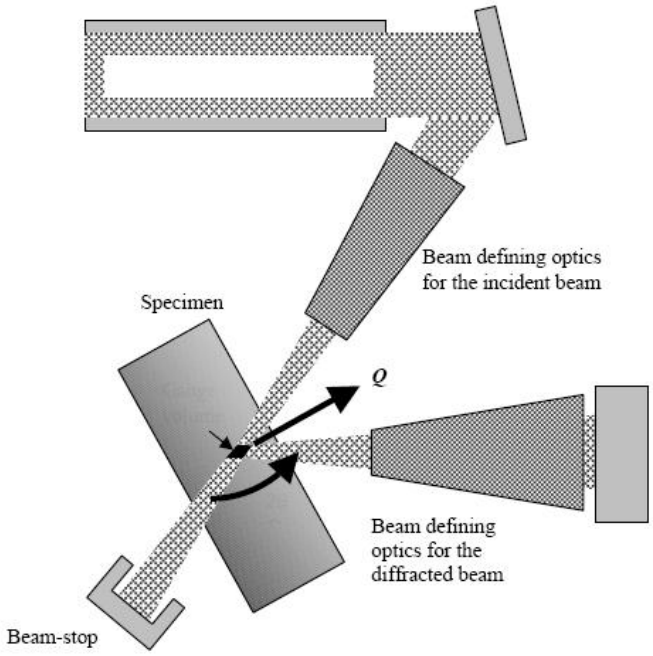

Figure 4: Schematic illustration of a typical continuous source based diffractometer for strain measurement.

Neutron diffraction vs conventional X-ray diffraction. From a physical point of view, neutron diffraction is a kind of *elastic scattering*[2], characterized by output energy comparable to the energy of the incident neutrons. *Thermal neutrons*, mainly employed for neutron diffraction, are free not-bounded particles in a state of thermal equilibrium with the surrounding environment. They are produced within atomic nuclei by slowing down (or *thermalizing*) more energetic neutrons. Usually

[2] In this context the term "elastic" is not linked to the kinetic energy conservation, but rather to the nucleus not-excited state after the reaction.

thermalization is the consequence of the collisions with nuclei (scattering) in a medium (called moderator) at 290 K, after they have been ejected from atomic nuclei during nuclear reactions (e.g. fission). Their low average kinetic energy (E_k = 0.025 eV), and hence their average speed (~ $2 \cdot 10^3$ m/s), are responsible for particular properties, such as large cross sections in fission, that make them suitable in certain chain-reaction applications.

Moreover, thermal neutrons wavelength $\lambda_{th} = 2 \cdot 10^{-10}$ m (~ 2 Å) corresponds to the natural spacing between atoms in crystalline solids. It follows that thermal neutrons beams are suitable for investigating the structure of crystals, and in particular to better locate atoms disposals with respect to the resolution reached by X-ray diffraction techniques. X-rays interact primarily with the electron cloud surrounding each atom, so that the contribution to the diffracted X-ray intensity is therefore larger for high Z atoms. On the contrary, what mainly characterizes neutrons interaction with matter is their capability to reach more easily the nucleus of the atom: these uncharged particles interact directly with nuclei (nuclear reactions), so that *each isotope differently contributes to the diffracted intensity* and the scattering length varies from isotope to isotope, rather than linearly with Z. It follows that light atoms (low Z) contribute strongly to the diffracted intensity, even in the presence of large Z atoms. In other terms, there is no need for an atomic form factor to describe the electron cloud shape of the atom and the atom scattering power does not depend so much on the scattering angle, as it does for X-rays. Besides, contrary to the photon case, a neutron beam can transmit through centimetres of metal, but it is easily attenuated by small amounts of light elements like hydrogen, boron and lithium.

Another main advantage with respect to X-ray diffraction is that neutron diffraction cannot be neglected at high angles: diffractograms show strong well defined diffraction peaks, especially if the experiment is done at low temperatures. This implies higher resolution and the possibility to precisely evaluate the atomic positions in lattice. It provides a method for observing excited states of nuclei after irradiation by thermal neutrons beams.

Finally, a neutron beam impinging onto any heterogeneous object is differently transmitted depending on neutron energy and on thickness, density, chemical composition and total cross section of the material along the line of sight. It is possible to reconstruct the internal feature of the objects by observing resonances in the reaction cross sections or by observing spectra of emitted particles or γ rays.

Neutron radiography

Neutron radiography (NR) [4] exploits the transmission of radiation to obtain visual information on the structure and/or inner processes of a given object. Over the last two decades there has been considerable development of NR techniques founding more and more applications. In the field of NDT, NR has a special role because of the need of high intensity neutron sources generally provided by research reactor or portable sources. NR provides complementary or sometimes completely original information with respect to X-ray or gamma radiography because the interaction of neutrons with material is fundamentally different from X-ray or gamma radiation. The basic principle of NR is very simple. The object under examination is placed in the path of the incident radiation, and the transmitted radiation is detected by a two-dimensional imaging system, as illustrated in Figure 5. The NR arrangement consists of a neutron source, a collimator which forms the beam, and a detecting system which registers the transmitted image of the investigated object. The most important technical parameter of a NR facility is the collimation ratio *L/D*, where *L* is the distance between the incident aperture of the collimator and the imaging plane and *D* is the diameter of the aperture. This important factor describes the beam collimation and limits the available spatial resolution. Moreover, the attenuation coefficient μ is another crucial parameter. The transmitted intensity of the radiation *I*, passing through a sample with an average transmission of μ can be

written as

$$I = I_0 e^{-\mu h}$$

where I_0 is the incident intensity and h is the thickness of the sample. If there is any inclusion in the sample of thickness x and the transmission μ_x then the transmitted intensity, I_x, in given as

$$I_x = I_0 e^{-\mu(h-x)-\mu_x x}$$

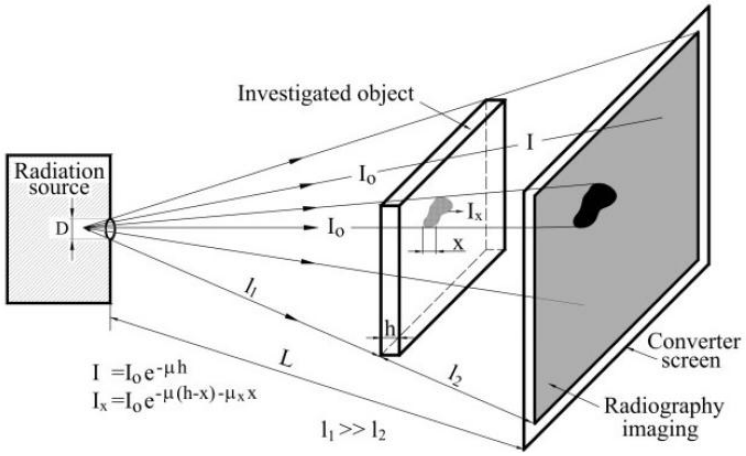

Figure 5: general principle of radiography.

Imaging techniques

Since neutrons are neutral particles, a suitable material is used to convert neutrons to another type of radiation and to enable them to be detected directly. Various detector systems are employed in NR: combinations of film and converter foil, combinations of a light-emitting scintillator screen with a CCD (charge-coupled device) camera and, more recently, neutron imaging plates. Depending on the object to be investigated and the task to be solved two basic types of NR are used: static radiography and dynamic radiography (real-time). Both techniques provide averaged information on the investigated object. To the other hand neutron computer tomography (NCT) is a technique that provides information on the three-dimensional structure of a given object.

NCT offers the unique capability of displaying cross-sectional slices of the samples with high resolution, and produces data which are easily adaptable for 3D representation. Although tomographic techniques have been well known since the beginning of the 1970s in the field of diagnostic medicine, their application in the neutron field was limited by the available neutron detectors. Recently this problem has been overcome by the development of CCD cameras. A scintillator converts the transmitted neutron beam to a visible light pattern, and each pixel of the CCD camera operate as an equivalent neutron detector and it visualizes only a very narrow area of the scintillating screen. In one rotates the sample, the NR images are recorded in several positions and the use of suitable software enables the 3D image of the object to be reconstructed. If the value of μ and μ_x are different from each other then the presence of inclusions will provide a contrast in the radiography image.

In the following some images obtained after neutron tomography are reported (figure 6).

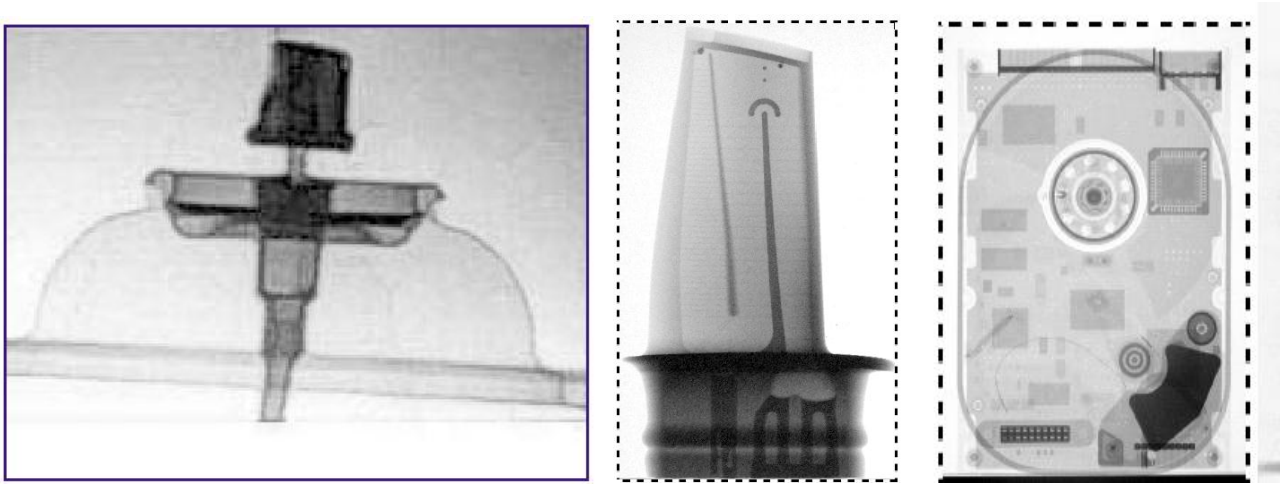

Figure 6: Neutron Radiography of a spray vessel (left), of a turbine blade (middle) and of a hard disk (right).

Conclusion

The use of low and high energy neutrons as an investigative tool has received considerable attention over the last few years. Neutrons, due to their capability to penetrate thick layers of materials, are particularly suited to investigate inside or beneath the surface of an object without damaging it, determining structure at the microscopic scale, thus providing fundamental information. The measurements of deformations and stresses in materials can be achieved relatively easily with neutron scattering techniques. The high penetration of the neutron beam in most materials allows a precise determination of residual stresses (with a spatial resolution of the order of 1 mm) and visual information on the structure of a given object.

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