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Neutron (and X-ray) tomography for the study of granular and porous media

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Abstract. During the last few decades, a number of advanced experimental techniques have provided an unprecedented insight into the behaviour of geomaterials. A notable example are the so-called full-field techniques such as X-ray and neutron imaging, which allow the non-destructive characterisation of the 4D (3D+time) response of geomaterials undergoing hydro–chemo–thermo-mechanical loading. While x-ray tomography over the last decade became a pillar in the domain, neutron imaging remains a comparatively less known tool. The unique properties of a neutron beam, such as high sensitivity to hydrogen (*e.g.,* water, hydrocarbons), high penetration into metals (allowing the imposition of extreme boundary conditions), and isotope sensitivity (*e.g.,* D2O/H2O), make neutron imaging an interesting tool for experimental studies of granular and porous media. This contribution provides an overview of recent contributions in this domain. A particular focus is given to the potential of some recent developments, such as the combination with simultaneous x-rays, new contrast options and substantial improvements in spatial and temporal resolution.

1 Introduction

Experimental granular physics and mechanics have advanced significantly in recent decades, driven by technological innovations and increased computational power. A particularly consequential development has been the widespread implementation of full-field, non-contact measurement techniques, which enable spatially resolved data acquisition, in contrast to traditional point-wise or bulk-averaged methodologies. Given the intrinsically heterogeneous nature of granular materials and their propensity for localized deformation, conventional boundary-based measurements often fail to capture the true mechanical behavior of the system—thus rendering full-field approaches indispensable for accurate characterization.

Among these, techniques based on ionizing radiation—X-rays and neutrons—stand out. While X-ray imaging has become a widely adopted tool for studying grain rearrangement, crushing, and deformation, neutron imaging remains comparatively underutilized. X-ray attenuation is a function of atomic number and density. Conversely neutrons interact with the nuclei. Due to different interactions with matter, neutrons attenuation of light elements such as hydrogen (and its compounds such as water and hydrocarbons) and lithium is several orders of magnitude higher than their X-ray attenuation.

Hydrogen sensitivity is crucial for hydro(-mechanical) studies, such as the study of permeability and its evolution (*e.g.,*, for reservoir management), effect of capillary forces (*e.g.,* for slope stability studies), response of hydro-sensitive materials, low and high temperature response in hydrogen-rich materials (*e.g.,* and fire conditions of asphalts and concrete). Furthermore, the superior penetrability of neutrons through metallic structures is advantageous for probing materials under extreme conditions, particularly high-pressure environments. For instance, a 1 cm thickness of aluminum or titanium attenuates approximately 37% and 70% of a 100 keV X-ray beam, respectively, whereas the same thickness attenuates only 1% and 30% of a neutron beam at a wavelength of 1 Å. Conversely, a 1 mm thickness of water attenuates merely 1.8% of X-rays but up to 99.7% of neutrons. Additional contrasts in material-specific attenuation are highlighted in Fig.1.

These examples illustrate only a subset of the key advantages and potential applications of neutron imaging, which this contribution aims to outline, and whose potential in geomechanics has so far been only partially exploited. This may perhaps be explained by the comparative rarity of neutron installations. There are in fact only a few tens of neutron tomographs compared to several thousands X-ray tomographs [1]. It is also noteworthy that, like other large-scale research infrastructures, neutron imaging facilities operate on a proposal-driven, peer-reviewed basis, with restricted scheduling windows, albeit typically free of charge. Another important comparison is of course spatial and temporal resolution. While some commercial X-ray tomographs have reached sub-μm resolution, the current record in "direct" neutron imaging is today around 3μm [20]. Other other hand, most neutron imaging facilities can image with ease large objects (in the tens of centimetres) thanks to the large beams, unlike most synchrotron X-ray facilities. An additional point of attention concerns instead the potential radiological activation

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of the samples that can limit the accessibility of the samples after the test (depending on the incident radiation and the materials, for minutes to days).

Recent developments in neutron imaging speed allows for tomographies down to one second per 3D volume *e.g.,* [7] (albeit at lower resolutions, around a hundred micrometers). At higher resolutions of course the exposure times can increase to even multiple seconds per radiography (e.g., 2s at 7µm at NeXT in “relatively standard ” conditions) meaning multiple hours per tomography. In terms of image analysis virtually all concerns, tools and developments relevant for X-rays are directly applicable to Neutrons.

A number of elements are far more (or less) visible in neutron than in X-ray imaging, making these two techniques highly complementary. It is nevertheless only very recently that it has been made possible to acquire neutron and X-ray Tomography fully simultaneously [2, 13]. This combined use is particularly useful as it allows not only to study different aspects of processes (*e.g.,* the interdependence between the opening of cracks and water penetration) but even aids in the identification of the different phases comprising a sample as shown in Fig. 1.

Another natural comparison is with Magnetic Resonance Imaging (MRI), which is also highly sensitive to hydrogen. It can also provide 3D volumes, albeit its working principle is very different to neutron/X-ray imaging, as it uses strong magnetic fields and radio waves to image the distribution of hydrogen nuclei and their mobility. Currently it can achieve resolutions in the tens/hundreds of microns [28] in minutes/seconds, respectively. In specific conditions (5K) it can reach below 3µm over multiple days [29]. Neutrons are more sensitive to hydrogen (detecting 10^{-3} to 10^{-6} in mass compared to (1–5%) for MRI [31]) but neutrons cannot distinguish the mobility of the atoms, precious for example to distinguish liquids from fats. Relatively few direct comparisons between MRI and Neutron imaging exist in literature [30].

This work builds on the review in [1] and focuses on recent developments, novel options and open challenges.

2 Representative applications

2.1 Fluids in granular and porous media

Neutron imaging is particularly useful for mapping fluid distribution in geomaterials due to the strong neutron attenuation of hydrogen. While X-rays and magnetic resonance are also used, X-rays often require tracers, which is acceptable when studying immiscible fluids but when the response of miscible fluids is of interest tracer diffusion renders their adoption cumbersome. MRI is instead more limited by spatio/temporal resolution. Importantly, neutrons offer isotope sensitivity, allowing clear distinction between hydrogen and deuterium, as well as their compounds like normal and heavy water, making them ideal for fluid studies. This can for example be used to study the migration of water in an already saturated medium (*e.g.,* [8, 21, 22]).

In granular and porous media, neutron imaging has been used for example to study water distribution and retention curves in partially saturated conditions, saturation and hydraulic properties [3, 4]. A peculiarity of water flow into granular media is that it is prone to create fingered structures which can also easily be studied in neutron imaging [5]. Also erosion, capillary barriers and the interaction with plant roots (within the so-called rhizosphere) can severely affect flow and local water content [6]. An interesting domain of application virtually unexplored is also the study of moisture and moisture migration in dynamic conditions such as granular flow.

The converse of fluid injection -drying- can also be studied, for example [10] studies the evaporation patterns occurring at high temperatures in case of metal casting. Its usage for studying the evolution of capillarity and granular collapse also seems under-explored thus far. A similar hydro-mechanical coupling occurs in other porous media, as in cement and concrete. In [24, 25]) neutron imaging is used to study the rapid evaporation, and ensuing moisture clog in concrete. This process is at the root of disastrous failure and spalling in case of fire as well as the slow drying and fissure opening underlying its ageing process [9].

2.2 Extreme conditions

Testing geomaterials under extreme conditions is crucial in numerous engineering applications. While X-ray imaging is a powerful approach, some *operando* test cells pose penetration challenges. Small samples can be studied with powerful X-ray sources such as synchrotrons, but many geomaterials have much larger representative elementary volumes due to inclusions or structural features. These larger samples require thick metal walls to withstand high pressures, making neutron imaging, with its higher metal transparency, an ideal option. The study of extreme pressures typical of dams or [11] or the hydraulic fracturing for geothermal energy applications [12] requires several centimetres of titanium, which are better penetrated by neutrons.

For temperatures below a few hundred degrees and small samples, X-ray-compatible cells are typically sufficient. However, higher temperatures (for example for studying magma or thermal runaway) in situ requires neutron imaging.

2.3 Advanced techniques

Neutrons plus X-rays: While the complementarity of neutron and X-rays was acknowledged since the early days, it is only recently that it has been made possible to acquire neutron and X-ray tomographies fully simultaneously [2, 13]. This complementarity is exemplified for concrete in Fig. 1. Therein we can see how X-rays allows the identification of the pores in the system but fail to distinguish aggregates and cement paste because of the similar density and thus X-ray opacity. Conversely, the hydrogen-rich cement paste is very visible to neutrons while aggregates and pores are both very transparent. Only when we combine these two techniques we can properly

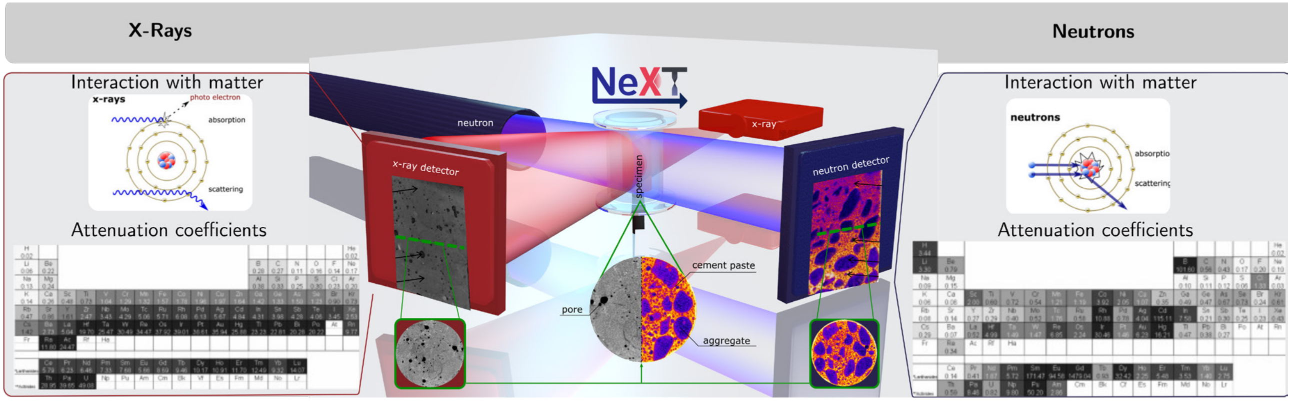

Figure 1. Illustration of the complementarity between neutron and X-ray imaging in a specific case: concrete. Cement is easily distinguished from aggregates and pores by neutrons but not by X-rays, while the reverse is true for pores and the cement-aggregate combination. Their combined use is therefore essential to identify these components. The side plot shows the neutron and X-ray opacities of atoms arranged along the periodic table.

segment this image (*i.e.,* identify its components. A similar consideration applies to several other materials composed of elements with too similar an attenuation in either radiation, which can find an improved contrast in the other. Importantly, the simultaneous acquisition of neutron and X-ray becomes crucial for operando studies of coupled thermo-, hydro-, chemo-, electro- mechanical processes.

This is for example the case of the complex hydro-mechanical couplings such as caking in food and pharmaceutical granular media, where water-sensitive materials, *i.e.,* materials whose mechanical properties change when in contact with water. For example in [23] neutron imaging is used to quantify the water absorbed in a hydro-sensitive granular medium (couscous) to measure the water uptake of this material when exposed to a humid environment. This allows to characterise *in operando* the condition at which caking (the creation of solid "crust" that occurs in some materials under loading in humid conditions) starts. Complementary x-rays provide here insights into the bond creation at the contacts at the root of the increased cohesion.

A similar hydro-mechanical coupling can be found in [14], where a Callovo-Oxfordian clayrock (COx) -crucial for radioactive waste storage- was observed to fragment as it absorbed water. Combining neutron imaging and X-rays (Fig. 3) reveals how water uptake, volume increase, and crack growth are influenced by bedding and confinement. Integrity loss is found to follow suction breakdown and mechanically-driven fracture propagation.

Chemo-mechanical couplings such as the corrosion of metallic components [15] (also reported in the additional material) and chemical sorption of pollutants [16] are also ideally studied with this combination of techniques. A similar consideration can also be made for hydro-thermo-chemo-electro-mechanical couplings such as those in energy materials as in the one reported in the additional materials for a lithium battery.

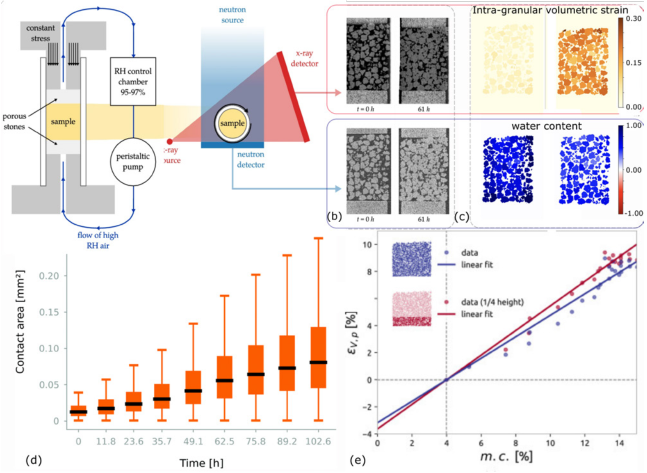

Figure 2. Summary of some results on a hydro-sensitive granular media (couscous). (a) scheme of the experiment on NeXT in [23], (b) representative vertical slices of the neutron and X-rays tomographies along the process, (c) values computed from them, intra-granular volumetric strain and water content.(d) From [26], evolution of the area of the inter-granular contact and (e), from [27] relation between the water content from TD-NMR and volumetric strain from X-rays.

Other techniques A number of new contrast options have also advanced recently: for example monochromatic imaging can be used to study Bragg edges, by varying the energy of the illuminating beam to find at which wavelength the opacity of the material rapidly changes because of Bragg's law. This can be correlated to the strain state of the material for example in powder-bed additive manufacturing [18] as well as to the crystallographic structures in metals [17]. Polarised neutron imaging can also be used to study magnetic fields, for example trapped in superconductors or induced by processes such as Gauss's law [19]. Another technique of particular interest is Neutron Grating Interferometry, which allows the characterization of heterogeneities well below that of the pixel. For

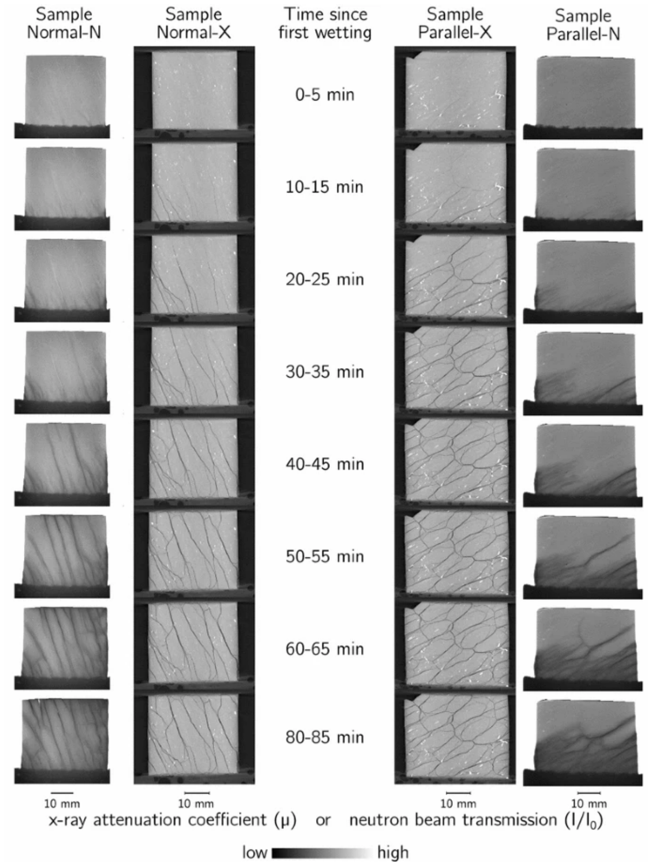

Figure 3. Neutron radiographies and vertical slices of X-ray tomographies highlighting the complementarity of the information in hydro-mechnical processes. The neutron radiographies show the water-rich areas is darker grays, while the X-rays reconstructions highlight the fissure opened by the hydro-mechanical couplings. These are from the experiments detailed in [14], exposing Callovo-Oxfordian Clayrock to water in samples cut at different angles from the depositions direction.

example through differential phase contrast and dark-field imaging it is possible to probe scales 0.1 μm to 10 μm by using (ultra)small-angle scattering-like contrast to detect sub-pixel structures (*e.g.,* pores and defects) that induce variations in the refractive index of materials.

3 Conclusion

This contribution proposes an overview of some key examples of neutron tomography, attempting to highlight some key strengths and *some* possible applications. A core challenge of neutron imaging remains the spatio-temporal resolution which is still lagging behind X-ray (notably because of the lower particle flux) albeit advanced image analysis techniques are being developed to partly compensate for this, such as event-mode reconstruction, algebraic reconstruction techniques, and, more recently, AI-enhanced image de-noising. While this contribution focuses chiefly on hydro-thermo-chemo-mechanical couplings for porous media, several other areas are still under-explored, such as powder-bed additive manufacturing where the high metal penetration of neutron is helpful, or the repose of all-solid-state batteries where the compression can be studied in realistic conditions while also following the lithium response thanks to their high neutron visibility.

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