Spallation Neutron Sources: A Review of Major Facilities and their Multidisciplinary Applications

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Neutron scattering has been demonstrated to be a ubiquitous tool to study the structure of matter and the evolution over a wide spectrum of spatial and temporal scales. Due to the limitations from research reactors, an alternative approach is the spallation method in which the accelerated protons bombard tungsten targets and thus produce the needed high-flux neutron beams. This work presents an overview of several major spallation neutron sources around the worlds, conducts a bibliometric study, and offers example results from the Spallation Neutron Source (SNS) in the US and the Chinese Spallation Neutron Source (CSNS) in China. Over 59,000 articles with the keyword neutron scattering have been published worldwide since the inception of such studies after World War II. The bibliometric study shows that frontier research activities have been devoted in utilizing such beams to investigate advanced structural and functional materials, identify the kinetics in critical chemical and biological processes, and to help develop novel quantum devices for the next-generation computers, to name a few.

Introduction

The first half of the twentieth century has witnessed a plethora of fundamental discoveries and understanding of the matter. A brief history on the study of structure and dynamics of matter can be compiled from the Nobel Prize in Physics[1]. Bragg and Bragg received their prize in 1915 for their services in the analysis of crystal structure by means of X-rays, which establishes the X-ray diffraction (XRD) technique as a way to probe the atomic structures. Decades after Chadwick winning the prize in 1935 for his discovery of the neutron in 1932, Wollan and Shull at Oak Ridge National Laboratory (then Clinton Laboratory) started the neutron studies after World War II, and Brockhouse started the kinetic studies in Chalk River Nuclear Laboratory in 1950s. The 1994 Nobel Prize in Physics was awarded to Shull and Brockhouse for their pioneering contributions to the development of neutron scattering techniques for studies of condensed matter. These great efforts have made neutron scattering as an indispensable probe for the investigations of the structure and dynamics of matter[2].

Neutrons are charge-less, possessing a noticeable magnetic moment, capable of deep penetration into many condensed matter and living materials, sensitive to isotopes, and therefore can be used to detect structures and their evolution over a wide range of length, time, and energy levels[2–4]. The world has entered into decades of facility constructions and nowadays more competitions over science and technology, as illustrated by the timeline in Figure 1. In early days, neutrons were mostly generated from fission-based research reactors. For example, one of the such facilities, High Flux Isotope Reactor (HFIR) at Oak Ridge National Laboratory (ORNL), generated neutron flux on the order of 10^{15} neutrons per cm^2 and per second in 1970s as shown in Figure 1. Although it is significantly higher than $\sim 10^{12}$ $1/cm^2$s in the era of Shull and Brockhouse, this approach has reached a physical limit. In contrast, the neutron flux generated from the spallation method can be two or three orders of magnitude higher. In the spallation neutron source, protons are first generated and accelerated, and then these high energy particles bombard tungsten targets and thus generate very high-flux neutrons for further usage, as illustrated in Figure 2(a). These large facilities are extremely sophisticated, extensive (e.g., $1.4 billion dollar construction cost in 2006 for Spallation Neutron Source (SNS) at ORNL, USA), and time consuming (e.g., >10 year planning, construction and commissioning of the China Spallation Neutron Source (CSNS) at Guangdong Province, China). Such powerful large facilities have enabled crucial scientific studies over multiple disciplines. Particularly, it should be noticed that in situ, non-destructive evaluation can be made possible, as shown by the neutron imaging example in Figure 2(b). In this example, a deposition-based additive manufacturing technique has been recently developed to manufacture a near-net-shaped body part, but the undetected inter-layer defects remain as the bottleneck. Here neutron imaging plays the same role as the medical X-ray imaging.

It should be recognized that there are other characterization tools for the structure and dynamics of the matter, with a wide range of pros and cons. For example, high-resolution transmis-

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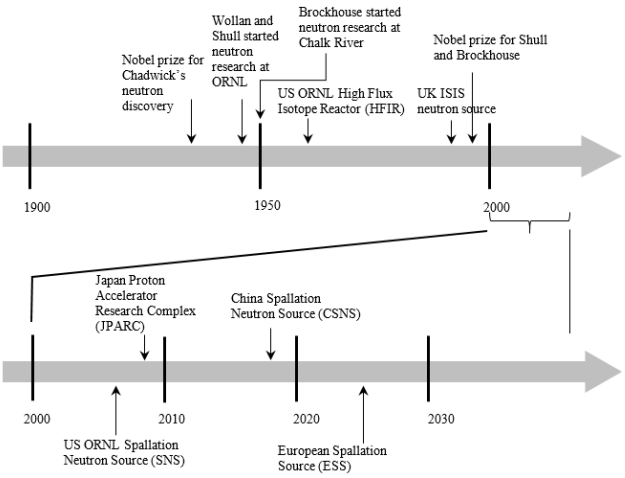

Fig. 1 Timeline of the inaugural neutron and neutron scattering studies and the commissioning of several major spallation neutron sources around the world.

sion electron microscopy (HRTEM) could give better imaging of the inter-layer defects in Fig. 2(b), but HRTEM requires very laborious efforts in sample preparation and is only for post-mortem analysis. Synchrotron radiation cannot penetrate as deep as neutron beam, while the spatial resolution of high energy X-ray beam is much better than neutron beam size. Scanning electron microscopy, even when equipped with electron back scattering diffraction capability, only gives surface information, in contrast to the bulk nature of neutron scattering. Lastly, it should be noted that neutron beams can penetrate through centimeter sized samples and thus provide unique capabilities than other tools.

Fig. 2 (a) Schematic illustration of the Spallation Neutron Source (SNS) at Oak Ridge National Laboratory. This image is a work of a United States Department of Energy (or predecessor organization) employee, taken or made as part of that person's official duties. As a work of the U.S. federal government, the image is in the public domain. (b) A representative research outcome from SNS[5]. Neutron interferometry transmission image showing the inter-layer defects in the layer-by-layer additive manufacturing of an aluminum alloy. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license.

Besides the historical accounts in the above, this reviewer article will further conduct a bibliometric study on the multidisciplinary nature of neutron scattering research, with a focus on the present and future of major competitors. It is then followed with two illustrative examples on Li-ion battery and additive manufacturing, respectively, and concluding remarks.

Bibliometric Results for Neutron Scattering Studies

The Clarivate Web of Science[6] is a world leading database that contains archivable data of scientific publications and their citation information, some of which can date back to the nineteenth century. The selection process of journals is based on the evaluation of publishing standards, editorial and content assessment, and citation analysis. No further refining or filtering of journals is employed in our search inquiries. When analyzing the author affiliations, disciplines, and others, an individual article can certainly fall into multiple countries and multiple disciplines, so that the sum of numbers of publications in Figs. 3(a) and 3(b) could exceed the total number. As shown in Figure 3(a), a keyword of neutron scattering results into the following top-ten disciplines, including: Materials Science Multidisciplinary, Physics Condensed Matter, Chemistry Physical, Physics Applied, Chemistry Multidisciplinary, Metallurgy Metallurgical Engineering, Crystallography, Chemistry Inorganic Nuclear, Physics Multidisciplinary, and Physics Atomic Molecular Chemical. As the Nobel Prize narrative for Shull and Brockhouse, neutron scattering is the critical tool for the structure and dynamics of matter, so that the top areas of study clearly lie on the solid-state materials (non-living or living), chemical reactions or thermodynamic evolution, and nuclear sciences. It should be noted that the publication numbers are accumulated ones from Chadwick's time, resulting into $\sim$ 59,000 total over the past $\sim$ 90 years.

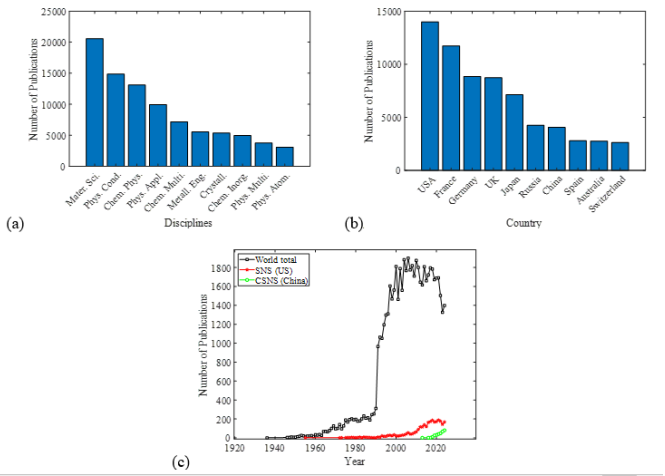

Fig. 3 A bibliometric study with keyword neutron scattering results into the numbers of publications in (a) top 10 disciplines (spelled out in the main text), (b) top 10 countries, and (c) annual outcomes of the world and two spallation neutron sources.

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Next, the results from top-ten countries are listed in Figure 3(b). It will be insightful to compare this result to the timeline in Figure 1, since the research reactor neutron sources are widely available in western countries since 1950s, and the five major spallation neutron sources are chronologically the ISIS Neutron and Muon Source, Rutherford Appleton Laboratory (UK) in 1985, the SNS at ORNL (USA) in 2006, the Japan Proton Accelerator Research Complex (JPARC) in 2008, the CSNS at China in 2018, and the European Spallation Source (ESS) under construction (scheduled in 2027). The majority of the analyzed literature do not specify the detailed neutron facility within the affiliations or acknowledgements. For example, some papers could only list affiliations outside of ORNL, although using the SNS facility in ORNL. Therefore, it is not possible to differentiate the contributions of spallation neutron sources and conventional research reactors. The total scientific publications from Clarivate Web of Science is about two million per year, but it is not advisable to judge the importance of neutron scattering from the miniscule fraction when compared to the total number of papers. One meaningful conclusion could be correlation between the large stepwise jump around 1990 in Fig. 3(c) and the commissioning of ISIS source and many other research reactors prior to that time. This is also observed, though not with bibliometric data, in literature[2].

Also plotted in Figure 3(c) are the annual productivity from the SNS of US and the CSNS. It should be noted that the keyword here does not fully describe the capabilities of such facilities, and many research and user projects do not necessarily result into scientific publications or list these facilities on these publications. Nevertheless, the bibliometric results in Figure 3 are indicative of a strong correlation between neutron studies and major facilities, and it is anticipated that the plateaus in Figure 3(c) will remain for the foreseeable future.

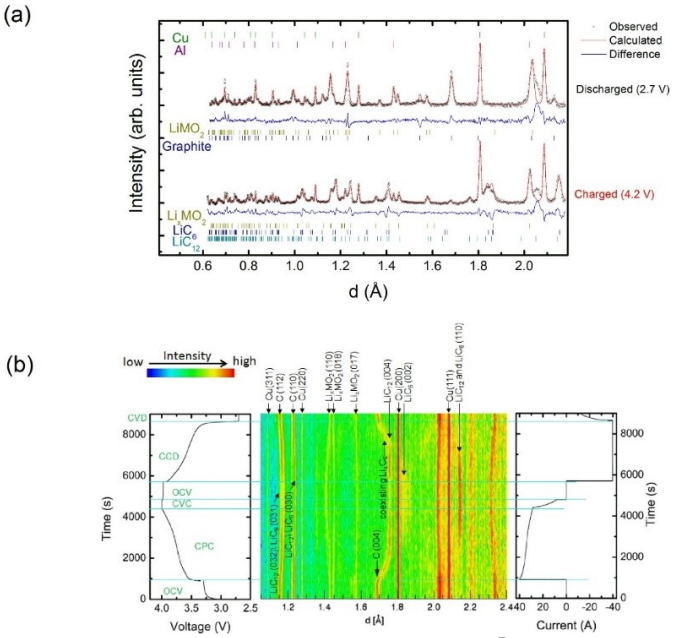

Fig. 4 An example study at the SNS (ORNL, USA)[7]. (a) The cathode material, LixNiyMnzCo(1-y-z)O2, and the graphite anode will be seen as the Bragg diffraction peaks with respect to the so-called d-spacing of the lattice structure. (b) The simultaneous monitoring of such peaks will reveal fundamental insights on how thermomechanical and electrochemical processes are convoluted. This work is licensed under a Creative Commons Attribution-NonCommercial-NoDerivative Works 3.0 Unported License.

Examples from Spallation Neutron Sources

Results in Figure 3 have motivated the authors to find representative research activities from these two major SNSs in the US and China, as shown below in Figure 4 and Figure 5, respectively.

The first example presents an in-situ, non-destructive measurement of a Li-ion battery. A commercial large-format pouch battery cell is used where a sandwich of a metal backing layer with $LiMO_2$ particles as the cathode, electrolyte, and another metal backing layer with granular graphite as the anode is further processed into jelly rolls. During charging and decharging processes, Li ions will pass back and forth between the two electrodes, and the cathode will experience large deformations that usually serves as the precursor for material failure like debonding or cracks. This is a fully coupled problem between crystalline lattice distortions and diffusional kinetics under complex electrochemical conditions. As shown in Figure 4(a), neutron diffraction signals will result into patterns similar to XRD, i.e., these are Bragg peaks from which distortions, defect densities, and crystallographic information can be obtained. Furthermore, the peak shifting during in-situ charging and decharging in Figure 4(b) will simultaneously tell how the distortion and the electrochemical processes are convoluted. Results of this nature, as shown by this illustrative example and technical reviews[8,9], demonstrate the critical role played by numerous spallation neutron facilities in understanding the eventual failure mechanisms in Li-ion batteries.

In the past two decades, one frontier research area is the additive manufacturing (AM) which plays a pivotal role in many industries and has been considered as a strategic direction for research and development. A bottleneck problem is the qualification of such AM-ed parts. For example, the selective laser melting (SLM) utilizes a laser beam as the heating source, melts metallic powers as it traverses over a bed of such powers, and leads to a controlled solidification behind the laser beam. The various ways of moving such heat sources will result into various types of material structures and potentially flaws, e.g., the inter-layer cracks in Figure 2(b). An in-situ, non-destructive evaluation of 316L stainless steel bars as made by SLM was conducted at the CSNS, and the resulting Bragg peaks evolve with the simultaneous loading, as shown in Figure 5. These

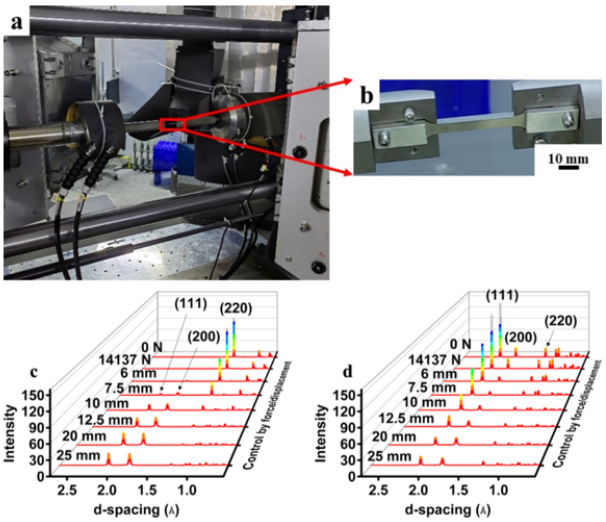

Fig. 5 An example study at the CSNS (China)[10]. (a) A rectangular-shaped sample is placed under two grips for the tensile test under the in-situ neutron beam. The material is 316L stainless steel, made from metallic powers under the selective laser melting (SLM). (b) Again, the Bragg peaks obtained from the neutron diffraction evolve under in-situ loading, thus providing insights for the structural features of such materials. This work is licensed under a Creative Commons Attribution-NonCommercial-NoDerivative Works 3.0 Unported License.

neutron diffraction results can be analyzed and the so-called residual stress distributions can be therefore obtained. As illustrated by this work and numerous recent publications using the facilities in the US[11,12], it can be concluded that the residual stress results, together with the neutron imaging, to investigate how various processing conditions (e.g., laser size and power density, printing direction and pattern, etc.) affect the material structures. Furthermore, it is explained in this work that defect densities and distributions can also be extracted, which is far more advantageous than the neutron imaging example in Figure 2(b). This is because of the limited sampling in Figure 2(b), as opposed to the statistical average nature in Figure 5.

Conclusion

From a bibliometric study, it is found that the advent of neutron scattering facilities worldwide, and especially that of the five major spallation neutron sources, are correlated strongly with active research interests in using neutron beams as a probe of the structure and dynamics of matter. Two examples are introduced to illustrate the importance and uniqueness of this technique. First, in modern energy material studies, it is imperative to understand the complex convolution between thermomechanical and electrochemical processes in the charging and decharging processes of Li-ion batteries. Second, one bottleneck in advanced manufacturing, being the qualification of AM-ed products, requires a tool that can both qualitatively image and quantitative analyze the processing flaws. In both examples, the high-flux neutron beam generated from spallation neutron source enables an in-situ, non-destructive evaluation and provides the critical insights to understand the dominant mechanisms that are responsible for the integrity of the resulting products, be they next-generation energy materials or additive manufacturing methodologies. It is thus believed that these current and under-construction facilities will continue to provide the needed multidisciplinary research outcomes for aerospace, medical, automotive, and many other industries, and thus be actively pursued by the scientific community.

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