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Neutron diffraction measurements at the INES diffractometer using a neutron radiative capture based counting technique

G. Festa [a], A. Pietropaolo [a,*], F. Grazzi [b], E. Barzagli [b], A. Scherillo [b,c], E.M. Schooneveld [c]

[a] Centro NAST, Università degli Studi di Roma Tor Vergata, Roma, Italy
[b] CNR-ISC Firenze, Italy
[c] ISIS facility Rutherford Appleton Laboratory, UK

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ABSTRACT

The global shortage of ^{3}He gas is an issue to be addressed in neutron detection. In the context of the research and development activity related to the replacement of ^{3}He for neutron counting systems, neutron diffraction measurements performed on the INES beam line at the ISIS pulsed spallation neutron source are presented. For these measurements two different neutron counting devices have been used: a 20 bar pressure squashed ^{3}He tube and a Yttrium–Aluminum–Perovskite scintillation detector. The scintillation detector was coupled to a cadmium sheet that registers the prompt radiative capture gamma rays generated by the (n,γ) nuclear reactions occurring in cadmium. The assessment of the scintillator based counting system was done by performing a Rietveld refinement analysis on the diffraction pattern from an ancient Japanese blade and comparing the results with those obtained by a ^{3}He tube placed at the same angular position. The results obtained demonstrate the considerable potential of the proposed counting approach based on the radiative capture gamma rays at spallation neutron sources.

1. Introduction

The global shortage of ^{3}He gas is a widely discussed problem within the neutron detector community [1,2]. Indeed, applications affected by the lack of ^{3}He range from the detection of special nuclear materials [3], oil well logging for hydrogen formation measurements [4] and neutron scattering for the investigation of condensed matter [5,6]. Thus, different neutron detection approaches have to be conceived to face this problem. It is well documented that straightforward ^{10}B lining of a conventional proportional counter will not match the efficiency of a corresponding ^{3}He tube for neutron detection due to the short range of boron capture products in the coating material and to the larger cross-section of the neutron–^{3}He interaction. Various setups were already proposed to enhance detection efficiency with ^{10}B-based detectors, see for example [7]. In the current paper a different approach for thermal neutron detection at pulsed neutron sources is presented, that is based on the use of thermal neutron radiative capture in metallic sheets and gamma ray detectors as neutron counters. The use of a YAP scintillation detector coupled to a neutron converter was already successfully investigated as a neutron counting device in neutron scattering applications [8,9]. This paper describes tests that were done on the INES beam line [10,11] at the ISIS spallation neutron source (UK) to assess the effectiveness of the proposed approach for neutron diffraction measurements.

2. Experiment

Neutron diffraction (ND) is a powerful technique for investigating the crystal structure of materials [12–14]. The diffraction process is described as the reflection of the incident beam by crystal planes $[h\,k\,l]$. The well known Bragg's law

$$\lambda = \frac{2d_{hkl}\sin(\vartheta)}{n} \quad (1)$$

links together d_{hkl} (the spacing relative to a set of lattice planes), the scattering angle 2ϑ and the wavelength λ of the incident radiation. In order to obtain a clear full diffraction pattern from a powder sample one of the two parameters (2ϑ or λ) can be varied leaving the other constant. Diffractometers operating at a spallation source are based on the determination of neutron energy (and hence wavelength) through the time of flight (ToF) technique in which case Bragg's law can be re-written in terms of ToF (t_{hkl}) as

$$d_{hkl} = \frac{ht_{hkl}}{2Lm_n\sin(\vartheta_0)} \quad (2)$$

where $2\vartheta_0$is a (fixed) scattering angle, L is the full flight path of the neutrons from the moderator to the detector through the scattering sample and d_{hkl} is the spacing relative to a set of lattice planes.

* Corresponding author. Tel.: +39 067 2594549; fax: +39062023507.
E-mail address: antonino.pietropaolo@roma2.infn.it (A. Pietropaolo).

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The experimental signal from a ToF diffraction measurement is a pattern of Bragg peaks. Peak positions are directly related to the crystal lattice dimensions and are used to identify phases, structures and/or to infer texture, strain and grain size information through line-shape analysis [15–18].

The experiment described in this paper was carried out at the INES diffractometer, operating at the ISIS pulsed spallation neutron source. The INES beam line is placed on a decoupled-Gd poisoned water moderator at $T=295$ K that provides a neutron spectrum peaked at $E\approx 30$ meV and a $1/E$ tail in the epithermal-fast neutron region. The primary flight path, from the moderator to the sample position is $L_0=22.8$ m and the ^{3}He tubes are at a fixed distance $L_1=1.000$ m from the sample position covering an angular range $11.6^\circ<2\theta<170.6^\circ$ [10]. The ^{3}He tubes of INES have an active volume of $(100\times 12.5\times 2.5)$ mm^3, operating at a pressure of 20 bar, providing a neutron detection efficiency of circa 50% for 25 meV neutrons, decreasing approximately as $1/v$, v being the neutron velocity. The nuclear reaction enabling the neutron detection in this case is $n+{}^3He\rightarrow p+t$ with a Q-value of 0.764 MeV, where p and t are the proton and tritium, respectively.

The experimental setup is schematically shown in Fig. 1. The device under investigation was made of a Cd sheet coupled to a Yttrium–Aluminum–Perovskite (YAP) scintillator. The scintillator in turn was connected to a photomultiplier, using a lower level discrimination threshold (LLD) at a photon energy of 600 keV in order to optimize the Signal to Background ratio (S/B) [19]. The Cd sheet acts as a (n,γ) converter following the nuclear reaction ^{113}Cd$(n,\gamma)^{114}$Cd + 7.6 MeV. The converter sheet was 1 mm thick, 10 cm long and 2 cm wide, the surface area being such to match that of the ^{3}He tubes used on INES. The 1 mm thickness of the Cd sheet provides a neutron absorption efficiency close to 1 up to the Cd cutoff energy at about 400 meV [20]. The YAP scintillation detector with Cd converter is used as a neutron counter: a neutron, elastically scattered by the sample at a given angle, is absorbed by the converter that, in turn, promptly radiates a gamma ray cascade. Detection of the gamma rays by the detector provides the time of arrival of the neutron onto the Cd, with respect to the time of the neutron production in the ISIS target (T0 signal), and thus the neutron energy using the time of flight technique.

The YAP–Cd detector was placed at a scattering angle of 90.565°, the same angular position as one of the ^{3}He tubes in the detector array of the INES beam line. This was done to allow

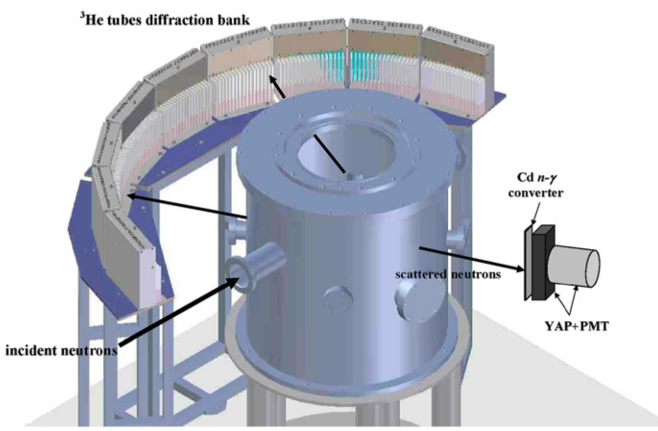

Fig. 1. Schematic layout of the INES powder diffraction diffractometer at ISIS during the experiment. The instrument was equipped with the new YAP–Cd device for non-standard diffraction measurements.

Table 1
Characteristics of the time of flight diffraction peaks recorded by the ^{3}He and YAP detectors.

TOF (μs)	S/B
^{3}He	
9925 ± 3	4.02
12150 ± 3	2.74
17158 ± 3	12.97
YAP–Cd	
9928 ± 3	1.21
12168 ± 3	0.79
17210 ± 3	8.81

First column reports the time of flight position of the main peaks while second column reports the Signal/Background ratio obtained by the ratio of the peak-signal area and the background area under the peak. The two areas are calculated for the same TOF interval.

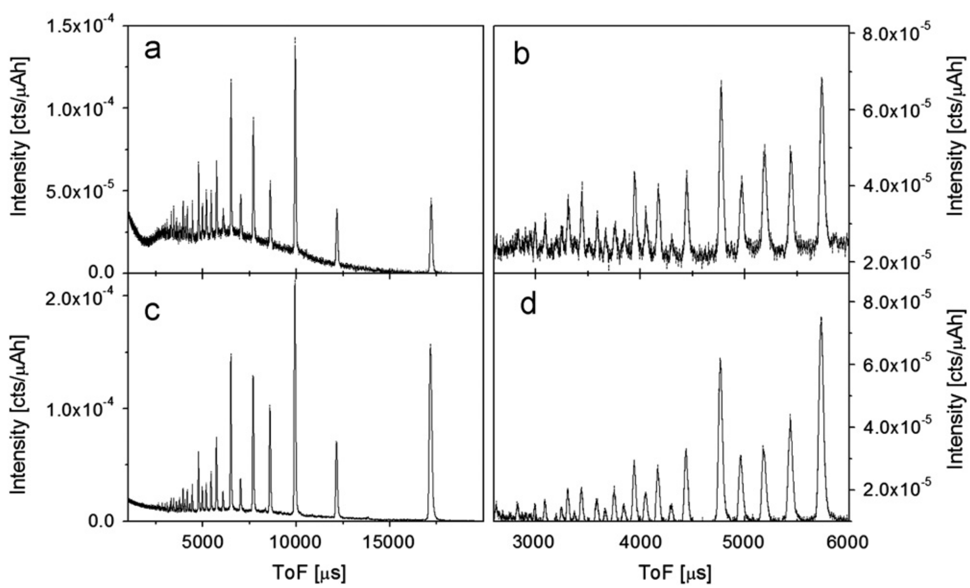

Fig. 2:. (a) Time of flight diffraction pattern recorded by the YAP–Cd counting device in the whole thermal neutron energy region; (b) blow up in the ToF region between 2500 and 6000 μs. Panels (c) and (d) show the same ToF diffraction patterns of panels (a) and (b), respectively, but recorded by the ^{3}He tube at a similar scattering angle of the YAP–Cd (see text for details).