



# Lattice location of O<sup>18</sup> in ion implanted Fe crystals by Rutherford backscattering spectrometry, channeling and nuclear reaction analysis



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## ABSTRACT

There are contradictory theoretical predictions of lattice location of oxygen interstitial atom at tetrahedral and octahedral interstices in bcc Fe. For validating these predictions, 300 keV O<sup>18</sup> ions with fluence of  $5 \times 10^{15}$  ions/cm<sup>2</sup> are implanted into bcc Fe single crystals at room temperature and annealed at 400 °C. The Rutherford backscattering spectrometry (RBS) and nuclear reaction analysis (NRA)/channeling measurements are carried out with 850 keV protons. The lattice location of implanted O<sup>18</sup> is analysed using the  $\alpha$ -particles yield from O<sup>18</sup>(p, $\alpha$ )N<sup>15</sup> nuclear reaction. The tilt angular scans of  $\alpha$ -particle yield along  $\langle 110 \rangle$  and  $\langle 100 \rangle$  axial directions are performed at room temperature. Lattice location of O<sup>18</sup> is found to be at tetrahedral interstitial site by comparing the experimental scan with simulated scans using FLUX7 software.

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## 1. Introduction

The commonly proposed structural materials for advanced nuclear reactors are high-Cr ferritic-martensitic steels, containing oxide particles [1]. This choice is supported by their superior thermal, corrosion resistance properties and radiation resistance against void swelling as compared to austenitic steels. A first approximation to describe such steels in a modelling framework is bcc iron and the Fe-Cr binary model alloys [2]. The impurities present have an influence on irradiation induced hardening and the embrittlement of the materials [3]. Heavy elements like Ti, V, Cr, Mn, Co, Ni, Cu, Ga and Ge are expected to be in substitutional lattice position [4]. Light elements like H, C, N, O, etc are found to be in interstitial lattice position [5–16]. Interstitial sites can be either in octahedral or tetrahedral in bcc Fe. Carbon is reported to be in octahedral interstitial site in bcc Fe by DFT calculations [5] and N is reported to be in octahedral by DFT calculations [5] as well as experiment [6]. The octahedral interstice (0.15r, where r is the radius of Fe atom) in bcc is smaller than the tetrahedral interstice (0.29r) [7]. Since the distance between interstitial site and two atoms of the host octagon is shorter than distance between four other atoms limiting the size of the interstitial site, introducing interstitial atom at octahedral site results in asymmetric distortion. The interstitial atom at tetrahedral site results in symmetric distortion in bcc lattice [8]. Earlier DFT reports pre-

dicted that H occupies tetrahedral site in bcc Fe [9]. Later it has been concluded that H interstitial site will be tetrahedral at lower concentration and it will change to octahedral site at higher concentration [10]. Also for the lattice site of oxygen in bcc Fe pristine crystal contradictory DFT reports claim tetrahedral [5] or octahedral [11–17] interstitial positions with the majority favouring the octahedral interstitial position. There are not much experimental studies to validate these theoretical predictions. Rutherford backscattering spectrometry and channeling is a versatile technique for determination of lattice site location in single crystals with a precision of 0.1 Å [18,19]. In this report we apply this technique to confirm the lattice site position of oxygen in bcc Fe crystal.

## 2. Experimental details

BCC Fe(110) and Fe(100) crystals with a chemical purity of 99.98% are used and all experiments were performed at room temperature. Oxygen being a lighter element, its backscattering signal will not be visible on the large Fe backscattering signal. So, analysing lattice location of oxygen will be difficult by RBS/Channeling. If O<sup>18</sup> isotope is introduced instead of oxygen, the lattice location of O<sup>18</sup> can be analysed by the out coming  $\alpha$ -particle yield from the well-known O<sup>18</sup>(p, $\alpha$ )N<sup>15</sup> nuclear reaction which has a broad resonance at 820 keV and Q-value of 3.981 MeV [20]. The crystal is kept at random orientation and implanted with 300 keV O<sup>18</sup> ions from a 1.7 MV tandemron accelerator [21,22] with a fluence of  $5 \times 10^{15}$  ions/cm<sup>2</sup> at room temperature. The projected range and straggling of O<sup>18</sup> is 280 nm and 90 nm respectively which is

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obtained from SRIM calculations [23]. The sample is annealed in situ in vacuum of  $2 \times 10^{-7}$  mbar at 400 °C for 30 min. Annealing at higher temperatures can improve the crystalline quality further. But oxygen diffuses out of the sample and is lost even at 500 °C in vacuum. So, annealing temperature is not raised above 400 °C. In Gaussian profile of  $O^{18}$ , the concentration is around 0.3% at peak position. Oxygen peak-concentration position is determined experimentally by taking the NRA data at various incident proton energies from 830 to 890 keV and the proton energy of 850 keV has shown the maximum  $\alpha$ -yield and it is chosen for all NRA analysis [20].

A series of Rutherford backscattering spectrometry (RBS) and channelling measurements are performed with 2 MeV  $He^+$  ions along the normal axial directions in virgin,  $O^{18}$  implanted Fe samples to check quality of the crystal. The rest of RBS and NRA/Channelling experiments along  $\langle 100 \rangle$  and  $\langle 110 \rangle$  axial directions are performed with 850 keV protons. The backscattered ions are detected with a surface barrier detector with an energy resolution of 15 keV, kept at a scattering angle of 165°. The same detector was used for NRA by inserting a 12  $\mu m$  thick mylar window before the detector to suppress the backscattered protons, by turning a rotary drive without breaking the vacuum. The tilt angular scans are simulated by FLUX7 program [24].

### 3. Results and discussion

#### 3.1. Crystallinity of Fe crystal

Fig. 1(a) shows the RBS and channelling spectra taken with 2 MeV  $He^+$  ions along  $\langle 110 \rangle$  axis from virgin and  $O^{18}$  implanted Fe(110). The ratio of the backscattering signal from aligned and random spectra taken in the window corresponding to a depth closer to the surface is called minimum yield  $\chi_{min}$  and it is a measure of crystalline quality. It is measured to be 8% for virgin crystal. For an ideal crystal, it has to be 1.3% along  $\langle 100 \rangle$  and 1.83% along  $\langle 110 \rangle$  and the measured value is of reasonable quality. The spectra are also taken in  $O^{18}$  implanted Fe(100) sample with similar crystalline quality which are not shown here. In Fig. 1(a), the aligned spectrum after  $O^{18}$  implantation is having dechannelling step at the implanted ion range due to the defects produced by  $O^{18}$  ion implantation which can be vacancies, self interstitial atom (SIA) defects which are believed to be in dumbbell interstitial configuration [26] or other defect complexes.

The aligned RBS spectrum given in Fig. 1(b) is from the  $O^{18}$  implanted Fe crystal obtained with 850 keV protons. A dechannelling step in the Fe signal is seen in this spectrum also. The  $\chi_{min}$  in the spectrum taken at near surface is found to be 10% and

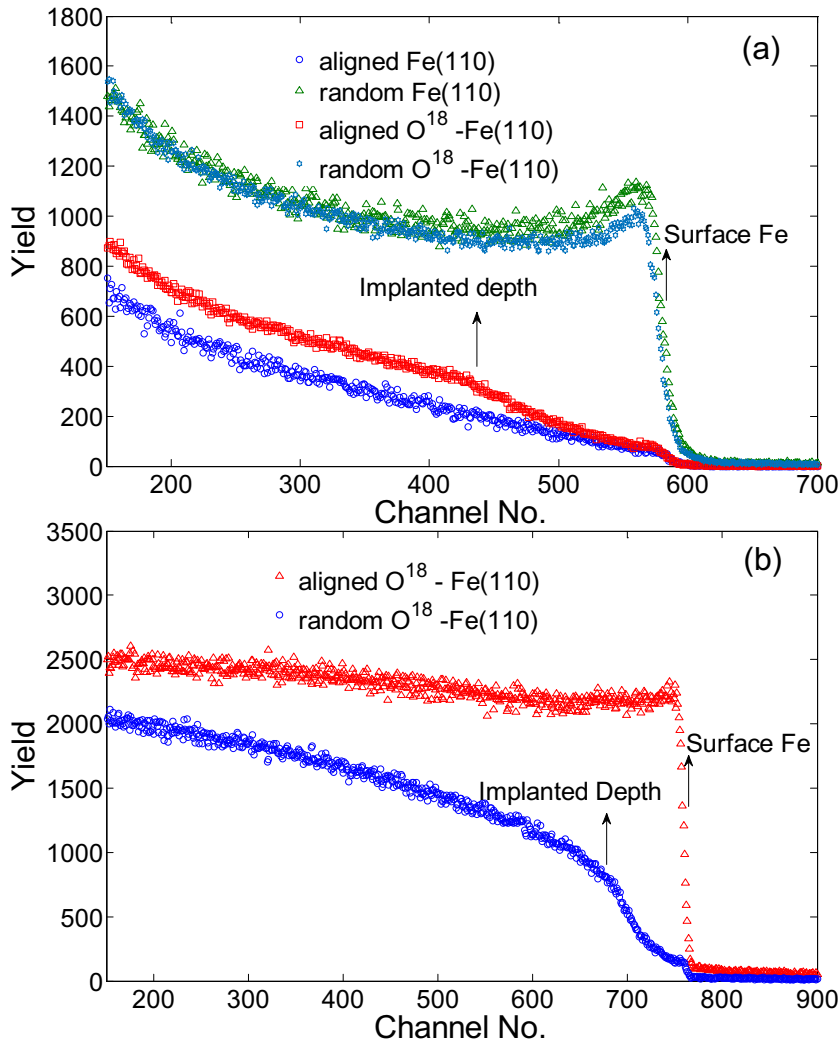


Fig. 1. Rutherford backscattering and channelling spectra from virgin and  $O^{18}$  implanted Fe(110) crystal taken with (a) 2 MeV  $He^+$  and (b) 850 keV protons.

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