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The influences of deuterium irradiation defects on mechanical properties for ceramic breeder material Li_2TiO_3

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HIGHLIGHTS

- The reduction of Vickerness hardness is caused by F-center defects in Li2TiO3 crystalline grains after deuterium irradiation.
- F-center defects in crystalline grains will be aggregated to form defect clusters with the increase of heating temperature, which lead to a hardening tendency within a certain temperature range.
- The aggregated defect clusters in the crystalline grains begin to recover when the annealing temperature increases to a point temperature, which results in the decrease of Vickers hardness.

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ABSTRACT

Tritium breeder materials are significant for blanket design of fusion reactor. However, during blanket operation, the ceramic breeder materials will be subject to neutron irradiation, which could be detrimental to mechanical properties. Because of good chemical stability and available tritium release behavior, Li_2TiO_3 is becoming one of candidate ceramic breeder materials. In this study, Li_2TiO_3 samples are irradiated by 120 keV deuterium ions. For sample characterization, the phase composition is investigated by X-ray diffraction (XRD) before and after irradiation. After deuterium irradiation, the Electron Spin Resonance (ESR) experiments are employed to investigate the irradiation defects. Micro-hardness measurement is applied to study the changes of mechanical properties. XRD results indicate that Li_2TiO_3 crystals are damaged by deuterium irradiation, but no new phases are produced. According to ESR experiment, the main defect type after deuterium irradiation is F-center which are vacancies trapping one electron. According to Vickers hardness measurement, size effect of micro-hardness is observed. The Meyer coefficient obtained in the experiment is 1.65, which is less than 2. Vickers hardness increases as applied loads decrease which are consistent with Meyer theory. And Vickers hardness of Li_2TiO_3 decreases as irradiation doses increase.

1. Introduction

The world-wide study on ceramic breeder materials focuses on Li_2O, Li_2TiO_3, Li_4SiO_4 and Li_2ZrO_3 [1,2]. During future reactor operation, the ceramic tritium breeder materials will subject to neutron irradiation, energetic tritons (2.7 MeV) and helium ions (2.1MeV), which could influence the mechanical properties of breeder materials. This may be result in crush of breeder materials. The destruction of the pore structure may effect on the tritium breeder ratio (TBR) in Test Blanket Module (TBM), and impact on the thermal conductivity which could be detrimental to safe operation. Because of good chemical stability and available tritium release behavior, Li_2TiO_3 is becoming one of candidate ceramic breeder materials. In previous studies, a convenient way to evaluate pebble mechanical strength of ceramic breeder materials is to measure its crush load. Jean-Daniel Lulewicz et al. measure crush load of several size fractions

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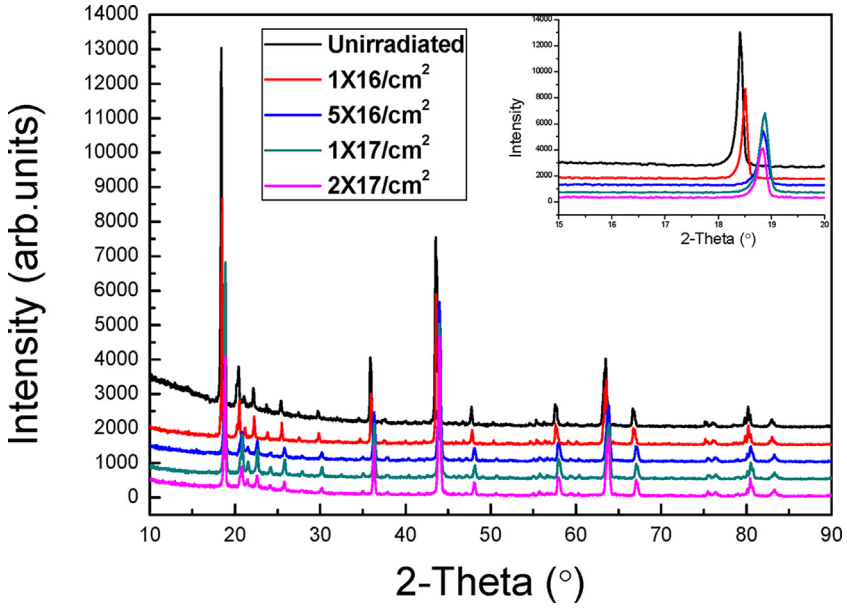

Fig. 1. XRD spectra for Li_2TiO_3 samples: comprehensive spectra including original sample, samples after irradiation, and inserted graph of amplifying spectra for the strongest peaks.

of Li_2TiO_3 [3]. They discovered that crush load is proportional to the square of the pebble diameter. Nicola et al. study mechanical characterization of Li_2TiO_3 and Li_4SiO_4 pebble beds [4]. Their results showed that the confining pressure is very important parameter for the determination of the pebble bed mechanical properties and in particular for the effective shear modulus and the damping coefficient. And in the compression test without radial constraints, the single sphere shows a greater strength than the pebble bed in the oedometric tests. Vickers hardness of original Li_2TiO_3 was also tested in some early literatures [5,6]. To the best of our knowledge, there has not been any reported work on Vickers hardness of Li_2TiO_3 after irradiation. Due to the important role of ceramic breeder materials in Test Blanket Module (TBM) for fusion reactors, it is necessary to investigate Vickers hardness before and after deuterium irradiation. It may be the first time to try to investigate the evolution of Vickers hardness after ion-irradiation. Data from this article can make a contribution to the engineering design of blanket in future fusion reactor.

In the present work, deuterium implantation is applied to simulate the irradiation damage in future fusion reactor. The damage caused by D^+ ion beam cannot be completely compared to the neutron one. But due to the extreme lack of 14 MeV neutron sources, energy ion beam has long been used to simulate radiation effects of neutrons [7,8]. Li_2TiO_3 samples are obtained from University of Science and Technology Beijing. Further details concerning fabrication description could be found elsewhere [9]. These Li_2TiO_3 samples are irradiated by 120 keV deuterium ions. After irradiation, the phase composition is investigated by using X-ray diffraction (XRD). ESR experiments are performed to elucidate the defect type of Li_2TiO_3. The Vickers hardness of Li_2TiO_3 is investigated by using Micro-Hardness analyses before and after irradiation.

2. Experiments

The average grain dimension of the Li_2TiO_3 plates applied in this study is about 3 μm measured by scanning electron microscope. Sample with plate shape and pebble shape have the similar properties due to the same preparation technique, such as grain size and porosity. And Vickers hardness were also studied as a function of porosity [5,6]. The porosity of the samples is about 10% [10]. Li_2TiO_3 samples are introduced into quartz tube and annealed at 1173 K in the atmosphere (same with the preparation environment of Li_2TiO_3) for an hour to remove the impurities. The deuterium irradiation experiments are performed at Institute of Semiconductors, Chinese Academy of Sciences. Experimental conditions of deuterium irradiation for Li_2TiO_3 are shown in Table 1. The energy of deuterium ions is 120 keV and the ion flux is approximately $4.6 \times 10^{13} D/cm^2.s$. The implantation depth is about 1 μm calculated by The Stopping and Range of Ions on Matter (SRIM). Deuterium irradiation is performed for about 220s, 1100s, 2200 s and 4400 s corresponding to fluences $1 \times 10^{16} D/cm^2$, $5 \times 10^{16} D/cm^2$, $1 \times 10^{17} D/cm^2$ and $2 \times 10^{17} D/cm^2$, respectively. The temperature (surface temperature of the plates) of samples during irradiation is estimated to be less than 373 K. Before and after irradiation, XRD analysis is performed to investigate the irradiation damage of Li_2TiO_3. After deuterium irradiation, the ESR experiments are performed for original sample and samples with irradiation fluences of $1 \times 10^{16} D/cm^2$, $5 \times 10^{16} D/cm^2$ and $2 \times 10^{17} D/cm^2$ at RT to investigate the irradiation defect type in Li_2TiO_3.

All of samples are transferred to Micro-hardness tester to evaluate mechanical behavior of Li_2TiO_3. Original samples are performed at 100 g, 200 g, 300 g, 500 g and 1000 g respectively, so as to distinguish the suitable stress. Each stress is tested for 6 times and Vickers hardness value employs the average finally. According to calculation and analysis of experimental results, the stress value of 500 g is applied in this work. To elucidate the influence of defects evolution on micro-hardness, Li_2TiO_3 samples with irradiation dose of $1 \times 10^{17} D/cm^2$ and $2 \times 10^{17} D/cm^2$ are taken to measure Vickers hardness after annealing at different temperatures for 1 h in air atmosphere.

3. Results and discussion

3.1. XRD analysis

Crystalline phases are identified by XRD at room temperature. Fig. 1 shows the XRD patterns for Li_2TiO_3 samples irradiated by deuterium ions with different doses. For comparison, a diffraction pattern of original sample is also shown in Fig. 1. In order to observe the change of peaks, the strongest peak is exhibited. No