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# Investigations on preparation, upper critical field and low temperature thermal expansion of $LiTi_2O_4$ superconductor

H.X. Geng *, A.F. Dong, G.C. Che, W.W. Huang, S.L. Jia, Z.X. Zhao

*National Laboratory for Superconductivity, Institute of Physics, Chinese Academy of Sciences, P.O. Box 603, Beijing 100080, China*

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**Abstract**

A new preparation route for $LiTi_2O_4$ superconductor was presented. The upper critical field and low temperature thermal expansion of $LiTi_2O_4$ were investigated. The upper critical field $H_{C2}(0)$ determined by the measurements of resistance and magnetization versus temperature are $8.9 \pm 0.5$ T and $9.3 \pm 0.3$ T, respectively. The linear coefficient of thermal expansion in the temperature range of 90–298 K determined by low temperature X-ray diffraction is $7.97(\pm 0.13) \times 10^{-5}\ K^{-1}$.


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*Keywords:* $LiTi_2O_4$; Preparation; Low temperature thermal expansion; Upper critical field

## 1. Introduction

$LiTi_2O_4$ superconductor with the spinel structure and $T_C \sim 12$ K was discovered by Johnston in 1973 [1]. This superconductor is still interesting due to some unusual properties in both physical and chemical respects. The preparation and crystal structure of the superconductor have been investigated in Refs. [2–12]. In previous publications, the polycrystalline $LiTi_2O_4$ superconductor was prepared by solid state reaction method using the starting materials of $Li_2Ti_2O_5 + Ti_2O_3 + TiO_2$, or $Li_2CO_3 + TiO_2$ and $Li_2CO_3 + TiO_2 + Ti$, with a more complex procedure. The upper critical field $H_{C2}(0)$ (2–32.8 T) of $LiTi_2O_4$ was reported by several groups [3,5,8]. Recently, the $H_{C2}(0)$ of $LiTi_2O_4$ determined by specific heat measurement is 11.7 T in Ref. [13].

In this paper, a new preparation route for $LiTi_2O_4$ superconductor was presented. The upper critical field and low temperature thermal expansion of $LiTi_2O_4$ superconductor were investigated.

* Corresponding author. Tel.: +86 01082649192; fax: +86 01082649486.
*E-mail address:* ghxia@ssc.iphy.ac.cn (H.X. Geng).

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## 2. Experimental

Ceramic $LiTi_2O_4$ samples were prepared by a new preparation route. The chemical reagent TiO and $Li_2Ti_3O_7$ monocrystal powders with the purities better than 99.99% were used as the starting materials. The preparation method of TiO was reported (see the powder diffraction file (23-1078)). The stoichiometric powders were mixed, ground, pelletized and calcined in an evacuated quartz tube at 805 °C for 50 h, then cooled slowly to room temperature, according to the chemical reaction:

$$2Li_2Ti_3O_7 + 2TiO = 4LiTi_2O_4$$

The phase structure of the prepared samples were characterized by powder X-ray diffraction (XRD) analysis on an MXP18A-HF-type diffractometer with Cu-$K_\alpha$ radiation. Powder X program was used for lattice parameter calculations. DC-magnetization $M$ and electrical resistance $R$ were measured versus temperature $T$ using a DC-SQUID magnetometer and four-probe technique, respectively. Low temperature linear thermal expansion was investigated by powder X-ray diffraction at low temperature on Rigaku D/max-2500 diffractometer with Cu-$K_\alpha$ radiation (40 kV × 200 mA) and a graphite monochromator.

## 3. Results and discussion

### 3.1. Sample characterization

The used $Li_2Ti_3O_7$ monocrystal was grown by Czochralski technique in our institute. $Li_2CO_3$ and $TiO_2$ with purities better than 99.99% were used as the starting materials. This crystal has lightly yellow color and is a transparent body. The XRD pattern of the monocrystal powder is shown in Fig. 1. Its lattice parameter $a = 5.015 \pm 0.004$ Å, $b = 9.539 \pm 0.004$ Å, $c = 2.946 \pm 0.005$ Å, which is agreement with those of $a = 5.0182$ Å, $b = 9.5523$ Å, $c = 2.9455$ Å reported in the powder diffraction file (34-393), indicating that the crystal has good quality.

The composition (Li 40 mol% and Ti 60 mol%) of $Li_2Ti_3O_7$ is the closest to that (Li 33.3 mol% and Ti 66.7 mol%) of $LiTi_2O_4$ among the compounds in $Li_2O$–$TiO_2$ system. This preparation route is beneficial to repress the volatilization of Li component, to control the composition of $LiTi_2O_4$ superconductor and to simplify preparing processes.

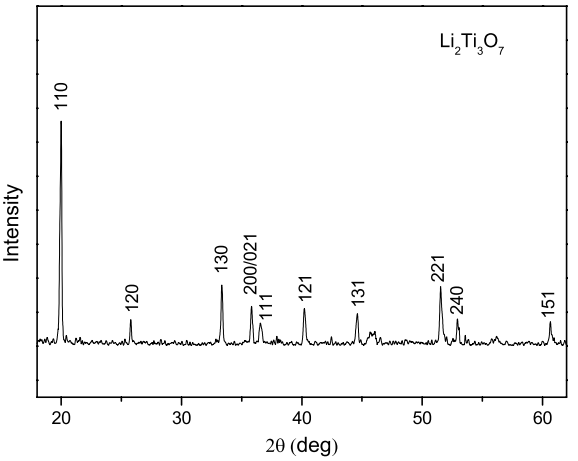


Fig. 1. XRD pattern of $Li_2Ti_3O_7$ monocrystal powder.

Fig. 2 shows the XRD pattern of the prepared $LiTi_2O_4$ superconductor, indicating that it is single phase. Its lattice parameter $a = 8.400 \pm 0.002$ Å, which is good agreement with that (8.405 Å) of $LiTi_2O_4$ with stoichiometric composition [5]. $R$–$T$ and $M$–$T$ curves of the prepared $LiTi_2O_4$ are shown in Figs. 3 and 4, indicating that $T_C$ is 12.1 K, $\Delta T = 0.3$ K and superconducting transition is very sharp.

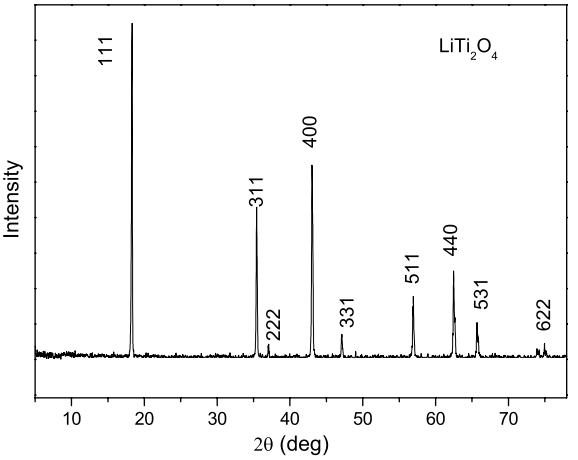


Fig. 2. XRD pattern of $LiTi_2O_4$.