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Compatibility with silver electrode and microwave dielectric properties of low firing $CaWO_4$-$2Li_2WO_4$ ceramics

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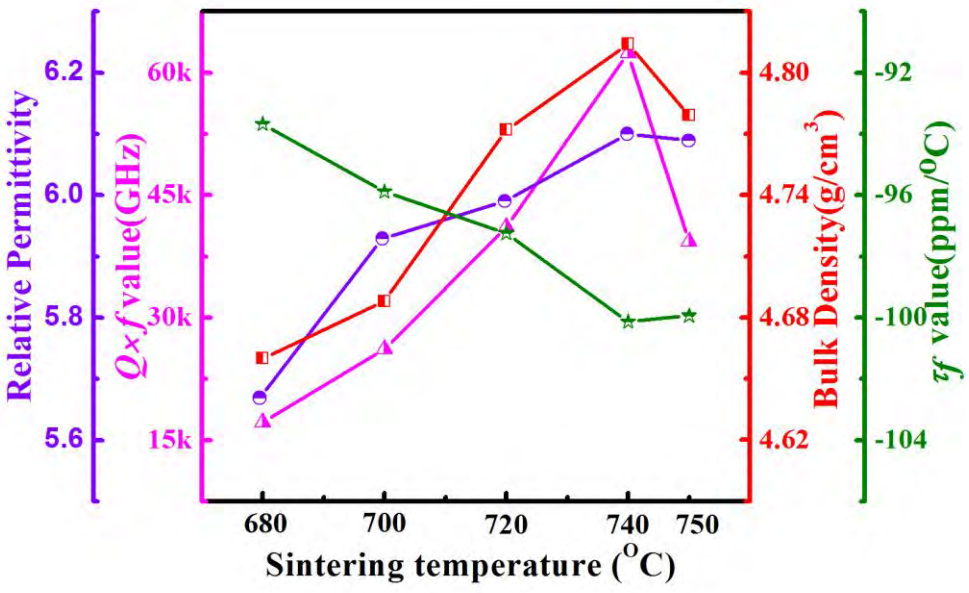
Graphicalabstract



Highlights

- $CaWO_4$-$2Li_2WO_4$ low-firing material was prepared by solid state reaction method.
- $CaWO_4$-$2Li_2WO_4$ ceramic exhibited good properties of $Q \times f$ = 62400 GHz, ε_r =6.1 and τ_f = -100.1 ppm/°C.
- $CaWO_4$-$2Li_2WO_4$ ceramic has a chemical compatibility with Ag, indicating its application in LTCC devices.

Abstract

A low firing microwave dielectric ceramic with the composition of $CaWO_4$-$2Li_2WO_4$ was prepared by the solid-state reaction method. The phase composition, microstructure and microwave dielectric properties of ceramics were studied by X-ray diffraction (XRD), scanning electron microscopy (SEM) and network analyzer. The $CaWO_4$-$2Li_2WO_4$ ceramics sintered at 740 °C for 4 h possessed the superior comprehensive performance with relative permittivity (ε_r) of 6.10, quality factor ($Q \times f$) of 62400 GHz and temperature coefficient of resonant frequency (τ_f) of -100.1 ppm/°C. The $CaWO_4$-$2Li_2WO_4$ dielectric ceramics were chemically compatible with silver (Ag) electrode, indicating that $CaWO_4$-$2Li_2WO_4$ ceramics might be a promising candidate for low temperature co-fired ceramic (LTCC) devices.

Keywords: $CaWO_4$-$2Li_2WO_4$; LTCC; Microwave dielectric properties; Ceramics

1. Introduction

Microwave dielectric materials are widely used to prepare many components, such as dielectric resonators, filters, and waveguides, etc [1,2]. Recently, with the rapid development of commercial wireless technologies, the demands for microwave

dielectric ceramics with excellent performance and low cost have been greatly increased. Low-temperature co-fired ceramics (LTCC) technology has attracted much interest due to the benefits provided for the fabrication of miniature multilayer devices [3,4]. For the application of LTCC devices, chemical compatibility between the ceramics and the inner metal electrodes is necessary and the firing temperature should be below the melting point of electrode materials (such as silver, <961 ºC). Therefore, the microwave dielectric materials used in LTCC field should possess several characteristics, such as a low sintering temperature (<961 ºC), low relative permittivity (ε_r<10), high quality factor ($Q \times f$ >50000 GHz) values and near zero temperature coefficient of resonant frequency (τ_f) [5-9].

Recently, many microwave dielectric ceramics have attracted much scientific and commercial attention due to their good microwave dielectric properties, such as $ZnAl_2O_4$ [10], $BaAl_2Si_2O_8$ [11], and $Mg_{1.975}Mn_{0.25}SiO_4$ [12]. However, high sintering temperatures (≥1100 ºC) restricted their practical applications in LTCC devices. It is generally known that the most effective and inexpensive way to reduce the sintering temperature of ceramics was liquid-phase sintering by adding glasses or low melting point oxides. However, the added glasses may react with the matrix, producing a lot of amorphous phase in materials, which seriously degrade the microwave dielectric properties of ceramics [13-16]. Consequently, searching for materials with intrinsically low firing temperatures is still a cutting-edge field of scientific research, such as Li_2O-rich, MoO_3-rich and WO_3-rich systems [17-19].

In the present work, the $CaWO_4$-$2Li_2WO_4$ ceramic was prepared by the conventional solid-state reaction method. Furthermore, the sintering behavior, microstructure, microwave dielectric properties and chemical compatibility with Ag of the $CaWO_4$-$2Li_2WO_4$ ceramics were investigated.

2. Experimental procedure

$CaWO_4$-$2Li_2WO_4$ ceramics were synthesized by the conventional solid-state reaction methods from high purity oxide powders of Li_2CO_3 (≥99%, Sinopharm Chemical Reagent Co., Ltd, Shanghai, China), $CaCO_3$ (≥99%, Sinopharm Chemical Reagent Co., Ltd., Shanghai, China) and WO_3 (≥99%, Sinopharm Chemical Reagent Co., Ltd, Shanghai, China). Stoichiometric proportion of the above raw materials was milled in alcohol using zirconia balls for 4 h and dried. The powders were calcined at 600 °C for 4 h. And then the mixtures were milled in the same way as the raw powders. After drying, the powders were mixed with 5 wt% polyvinyl alcohol (PVA) and granulated. Subsequently, the granulated powders were uniaxially pressed into pellets with 12 mm in diameter and 6 - 7 mm in thickness under the uniaxial pressure of 200 MPa. Finally, the pellets were sintered at 680 - 750 ºC for 4 h in air at a heating rate of 5 ºC/min and then cooled to room temperature in furnace.

The crystal structure of the samples was investigated by an X-ray diffraction (XRD) ($CuK_{\alpha 1}$, 1.54059 Å, Model X'Pert PRO, PANalytical, Almelo, Holland) operated at 40 kV and 40 mA with the scanning range of 15 - 70 º. Microstructure observation of the sintered samples was performed by a scanning electron microscopy (SEM). The elementary analysis was performed by an energy dispersive spectrometer (EDS). The bulk density of the sintered ceramics was measured by the Archimede's method using the distilled water as a medium. The relative permittivity (ε_r) and quality factor ($Q \times f$) values at microwave frequencies were measured by the $TE_{01\delta}$ shielded cavity method [20] in the frequency range of 10 - 13 GHz using a Network Analyzer (ModelN5230A, Agilent Co., CA, 10 MHz - 40 GHz) and a temperature chamber (DELTA9039, Delta Design, USA). The outer and inner diameters of the $TE_{01\delta}$ shielded cavity were 39 mm and 25 mm, respectively. The support of the cavity

was the single crystal quartz with 4.8 mm in diameter and 3.97 mm in thickness, which had a superior comprehensive performance with relative permittivity (ε_r) of 4.4, quality factor ($Q \times f$) of 1400000 GHz and temperature coefficient of resonant frequency (τ_f) of 9 ppm/°C [21]. The temperature coefficients of resonant frequency (τ_f) values of ceramics were calculated by the following formula:

$$\tau_f = \frac{f_T - f_0}{f_0(T - T_0)} \quad (1)$$

where f_T and f_0 are the $TE_{01\delta}$ resonant frequencies at the measuring temperature T (85 °C) and T_0 (25 °C), respectively.

3. Results and discussion

The room-temperature X-ray diffraction patterns (XRD) of the $CaWO_4$-$2Li_2WO_4$ samples calcined at 600 °C and sintered at 700 - 750 °C for 4 h are shown in Figure 1. The aim of calcination for ceramics is to synthesize the main phase of ceramics, which avoid the crack induced by the large shrinkage of ceramics in sintering process. In this work, when the calcined temperature was < 600 °C, the sample did not form the desired phase ($CaWO_4$ and Li_2WO_4). As the calcination temperature was > 600 °C the sample was too densified to crush. Therefore, the powder was calcined at 600 °C. All peaks can be indexed in terms of Li_2WO_4 (PDF card # 00-012-0760) and $CaWO_4$ (PDF card # 00-007-0210), and no other phase was observed. The $CaWO_4$ and Li_2WO_4 phases were marked in Fig. 1(b), Fig. 1(c), respectively. It can be concluded that no chemical reaction between Li_2WO_4 and $CaWO_4$ occurred. However, the intensity of diffraction peaks for Li_2WO_4 differed greatly as the sintering temperature increased from 700 °C to 750 °C, as shown in Fig. 1. When the sintering temperature increased from 700 °C to 720 °C, the intensity of diffraction peaks increased, indicating that the relative content of Li_2WO_4 increased with increasing the sintering

temperature. However, no obvious peaks varied with further increasing the sintering temperature.

Figure 2 demonstrates the scanning electron microscopy (SEM) pictures of the $CaWO_4$-$2Li_2WO_4$ ceramics sintered at different temperatures (700 - 750 °C) for 4 h. There are two kinds of grains, one is large grain (marked "I" in Fig. 2) and the other is small grain (marked "II" in Fig. 2). When the sintering temperature was 700 °C, there are many pores in ceramics, which agreed well with the analysis of the bulk density. When the sintering temperature increased to 740 °C, the grain size of ceramics increased and the samples became denser. Due to the evaporation of Li, some abnormal grains were observed as the sintering temperature was 740 °C. The evaporation of Li is a dynamic process from inside to outside at high temperature, which leads to the excessive content of Li_2O on the surface of ceramic. However, during the sintering process, the enrichment of Li will form more liquid-phase on the surface of samples, the flow of liquid-phase will promote the fusion and accelerate the growth of grains. Therefore, the grain size of the ceramic surface is larger.

Table 1 lists the energy dispersive spectrometer (EDS) analysis of the $CaWO_4$-$2Li_2WO_4$ ceramics sintered at 740 °C for 4 h. It can be seen that the region "I" was Li_2WO_4 and the region "II" was $CaWO_4$. Lithium could not be detected by EDS because of its light atomic mass. In large grain size region, W and O elements were detected, which indicated that this region was Li_2WO_4 grains. In small grain size region, the ratio between Ca and W atomics was close to 1:1, showing that this grain was $CaWO_4$ grains.

Figure 3 shows the bulk density (ρ), relative permittivity (ε_r), quality factor ($Q \times f$) and temperature coefficient of resonant frequency (τ_f) of $CaWO_4$-$2Li_2WO_4$ ceramics as a function of the sintering temperature (680 - 750 °C). As shown in Fig.

3(a), when the sintering temperature was 680 ºC, the bulk density of ceramics was ~ 4.66 g/cm^3. With increasing the sintering temperature, the bulk density increased, reached maximum value (4.82 g/cm^3) at 740 ºC, and then declined with further increasing the sintering temperature. It was reported that the relative permittivity (ε_r) was primarily determined by the composition, grain size and the density [22]. Among them, the density had a great effect on the relative permittivity (ε_r). The relationship between the relative permittivity (ε_r) and the sintering temperature shows a similar trend to that of the bulk density, as show in Fig. 3(b). As the sintering temperature increased from 680 ºC to 750 ºC, the relative permittivity (ε_r) was increased from 5.70 to 6.10, and then decreased to 6.09. As well known, the quality factor ($Q\times f$) values are mainly affected by the densification, average grain size and secondary phases of ceramics [23,24]. The $CaWO_4$-$2Li_2WO_4$ ceramics were mainly influenced by the densification of ceramics. When the sintering temperature increased from 680 ºC to 750 ºC, the quality factor ($Q\times f$) values increased because the ceramics gradually became denser. The ceramics reached a maximum value of 62400 GHz as the sintering temperature was 740 ºC. Thereafter, the $Q\times f$ values decreased with further increasing the sintering temperature because of the abnormal grain growth. The temperature coefficients of resonant frequency (τ_f) for $CaWO_4$-$2Li_2WO_4$ ceramics did not change remarkably and remained a stable value in the range from -93.7 ppm/ºC to -100.1 ppm/ºC as the sintering temperature increased from 680 ºC to 750 ºC.

To evaluate the chemical compatibility between the $CaWO_4$-$2Li_2WO_4$ ceramics and silver electrode, $CaWO_4$-$2Li_2WO_4$ ceramics were co-fired with 20 wt% Ag powders at 740 °C for 4 h and analyzed to detect interactions between the low-fired samples and electrodes. Figure 4 illustrates the X-ray diffraction pattern (XRD) and the backscattered electron micrograph (BSEM) image of $CaWO_4$-$2Li_2WO_4$ ceramics

co-fired with 20 wt% Ag powders at 740 °C for 4 h. XRD showed that no second phases were formed except $CaWO_4$, Li_2WO_4 and Ag. In addition, there was no obvious diffusion phenomenon between the Ag and matrix grains, demonstrating that no chemical reaction between the $CaWO_4$-$2Li_2WO_4$ ceramics and Ag occurred, as shown in the inset of Fig. 4. These results indicated that $CaWO_4$-$2Li_2WO_4$ ceramics have a good chemical compatibility with Ag electrode.

The comparison of microwave dielectric properties between the $CaWO_4$-$2Li_2WO_4$ ceramics and previous material systems is illustrated in Table 2. The Magnesium-based [25] and Manganese-based [26,27] compounds demonstrated low relative permittivity (ε_r) and high quality factor ($Q\times f$) values. But higher sintering temperatures (≥1150 ºC) restricted their further applications. By contrast, $CaWO_4$-$2Li_2WO_4$ ceramics could be sintered at 740 ºC and exhibited good microwave dielectric properties with low relative permittivity (ε_r = 6.10) and high quality factor values ($Q\times f$ = 62400 GHz). Especially, $CaWO_4$-$2Li_2WO_4$ ceramics could be co-fired with Ag, indicating that it has great potential application in LTCC devices.

4. Conclusions

$CaWO_4$-$2Li_2WO_4$ ceramics has been investigated as a promising microwave dielectric material for LTCC applications. $CaWO_4$-$2Li_2WO_4$ ceramics sintered at 740 °C for 4 h exhibited low relative permittivity (ε_r) of 6.10, high quality factor ($Q\times f$) value of 62400 GHz, and temperature coefficient of resonant frequency (τ_f) of -100.1 ppm/°C. Particularly, $CaWO_4$-$2Li_2WO_4$ ceramics have a good chemical compatibility with Ag electrode. Therefore, $CaWO_4$-$2Li_2WO_4$ ceramics might be an attractive promising candidate for LTCC application.

Acknowledgements

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Figure Captions:

Fig.1. XRD patterns of the $CaWO_4$-$2Li_2WO_4$ samples calcinded at (a) 600 ºC and sintered at: (b) 700 ºC, (c) 720 ºC, (d) 740 ºC, (e) 750 ºC for 4 h.

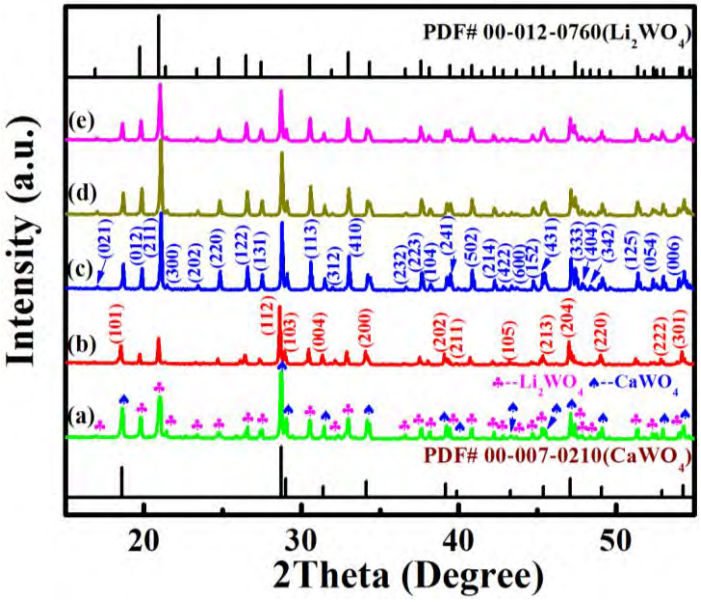

Fig.1

Fig.2. Scanning electron emission (SEM) micrographs of the $CaWO_4$-$2Li_2WO_4$ ceramics sintered at: (a) 700 ºC, (b) 720 ºC, (c) 740 ºC, (d) 750 ºC for 4 h.

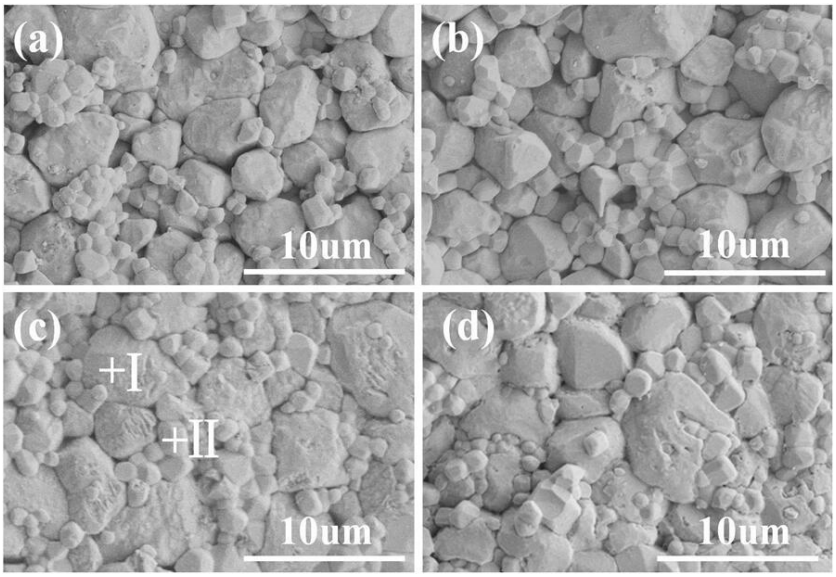

Fig.2

Fig.3. Bulk density (ρ), relative permittivity (ε_r), quality factor ($Q\times f$) and temperature coefficient of resonant frequency (τ_f) of the $CaWO_4$-$2Li_2WO_4$ ceramics as a function of the sintering temperature.

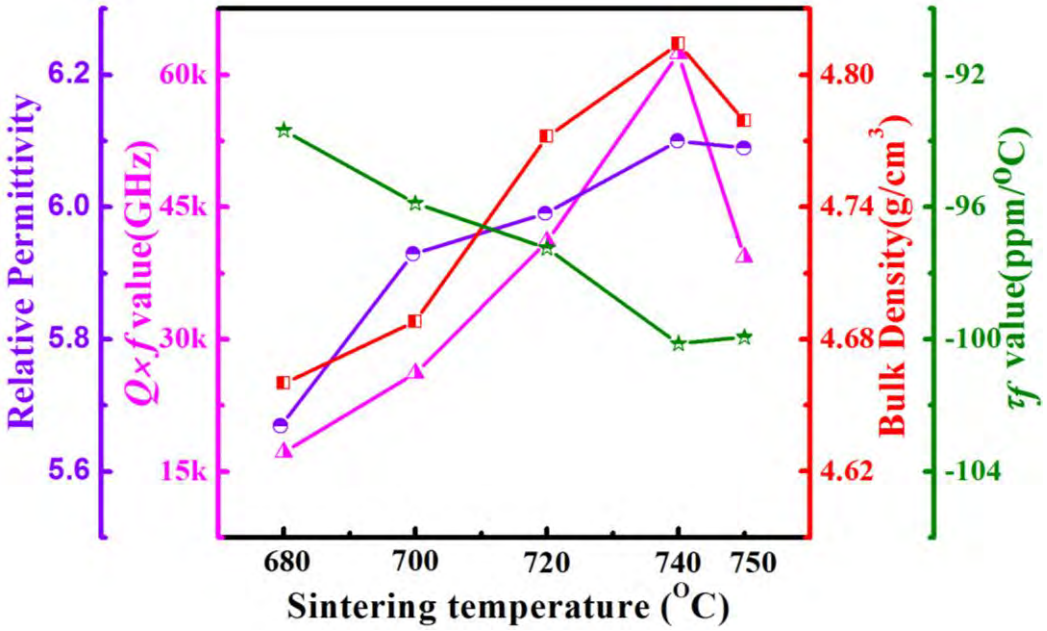

Fig.3

Fig.4. X-ray diffraction pattern (XRD) and backscattered electron micrograph (BSEM) image of the $CaWO_4$-$2Li_2WO_4$ ceramics co-fired with 20 wt% Ag powders at 740 °C for 4 h.

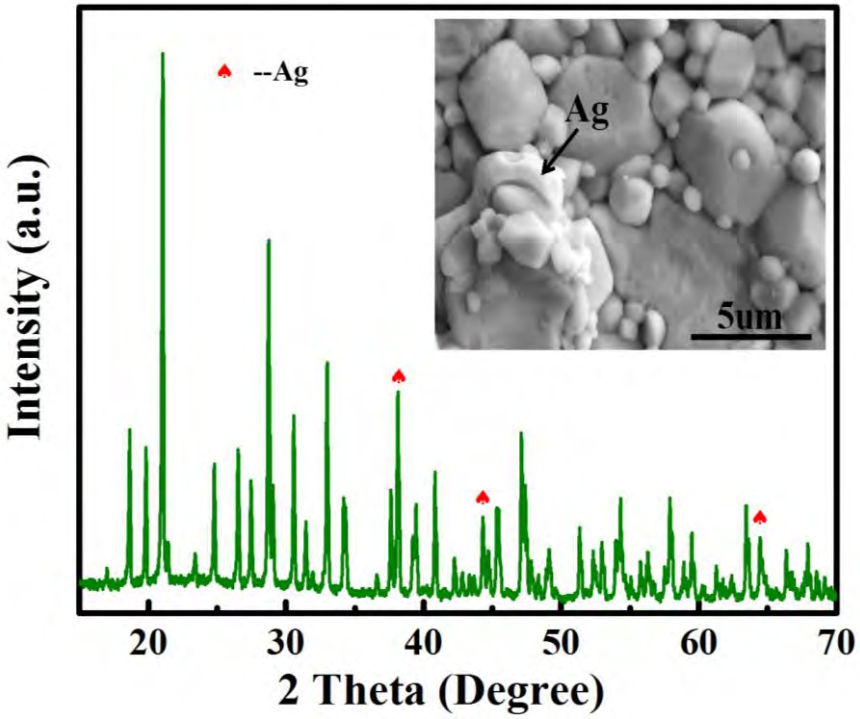

Fig.4

Table Captions:

Table 1 Energy dispersive spectrometer (EDS) analysis of the $CaWO_4$-$2Li_2WO_4$ ceramics sintered at 740 ºC for 4 h, which corresponds to the SEM image in Fig. 2 (c).

Table 1

Region	Atomic(%)		
	Ca K	W M	O K
Ⅰ	—	27.98	72.02
Ⅱ	12.27	14.86	72.87

Table 2 The comparison of microwave dielectric properties between the $CaWO_4$-$2Li_2WO_4$ ceramics and the previous material systems.

Ceramic composition	Sintering temperature (°C)	ε_r	$Q \times f$ (GHz)	τ_f (ppm/°C)	Ref.
$Mn_2P_2O_7$	1150	7.34	23850	-96.0	[25]
α-$Mg_2P_2O_7$	1150	6.10	38100	-746.0	[25]
$(Mg_{0.4}Zn_{0.6})_2SiO_4$	1250	6.60	95600	-60.0	[26]
$(Ca_{1-x}Mg_x)SiO_3$	1290	6.49	62420	-43.3	[27]
$CaWO_4$-$2Li_2WO_4$	680	5.70	17200	-93.7	
$CaWO_4$-$2Li_2WO_4$	700	5.93	26100	-95.9	
$CaWO_4$-$2Li_2WO_4$	720	5.99	41100	-97.2	Our work
$CaWO_4$-$2Li_2WO_4$	740	6.10	62400	-100.1	
$CaWO_4$-$2Li_2WO_4$	750	6.09	39300	-99.9	