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# Microwave dielectric properties and compatibility with silver of low-fired $Li_2MgTi_3O_8$ ceramics with $Li_2O-MgO-B_2O_3$ frit

Yan BAO[1], Guo-hua CHEN[1,2], Mei-zhen HOU[1], Yun YANG[1], Zuo-peng HAN[1], Kai-neng DENG[1]

1. School of Materials Science and Engineering, Guilin University of Electronic Technology, Guilin 541004, China;
2. Guangxi Key Laboratory of Information Materials, Guilin University of Electronic Technology, Guilin 541004, China

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**Abstract:** The effects of $Li_2O-MgO-B_2O_3$ (LMB) glass additive on the sintering characteristics, phase purity, microstructure, and microwave dielectric properties of $Li_2MgTi_3O_8$ ceramics were investigated. The experimental results demonstrate that the addition of LMB glass effectively lowers the sintering temperature of $Li_2MgTi_3O_8$ ceramic from 1025 °C to 875 °C and induces no obvious degradation of the microwave dielectric properties. Typically, the 1.5% LMB glass-added $Li_2MgTi_3O_8$ ceramic sintered at 875 °C for 4 h shows excellent microwave dielectric properties of $Q\times f$=45403 GHz, $\varepsilon_r$=25.9 and $\tau_f\approx0$ °C$^{-1}$. The dielectric ceramic exhibits stability against the reaction with the Ag electrode, which indicates that the ceramics could be applied in multilayer microwave devices requiring low firing temperatures.

**Key words:** microwave ceramics; dielectric properties; glass; low-temperature sintering; compatibility

## 1 Introduction

Recently, low temperature co-fired ceramic (LTCC) multilayer devices have been investigated to reduce the device size [1−6] to meet the demands of the miniaturization of microwave communication systems such as mobile systems. Silver has been widely used as the internal electrode in the multilayer devices because of its high conductivity and low cost [7−11]. The melting temperature of Ag is low. Therefore, for the fabrication of the multilayer devices, it is important to develop microwave dielectric ceramics that have low sintering temperatures and can be co-fired with Ag [12−14].

More recently, the microwave dielectric properties of $Li_2MgTi_3O_8$ ceramics have been investigated by GEORGE and SEBASTIAN [15]. They reported that this ceramic sintered at 1075 °C has good dielectric properties of relative permittivity $\varepsilon_r$=27.2, $Q\times f$=42000 GHz and resonant frequency $\tau_f=3.2\times10^{-6}$ °C$^{-1}$. However, the sintering temperature of $Li_2MgTi_3O_8$ ceramic is too high to be applicable to LTCC. So, it is necessary to reduce the sintering temperature of this material. It is well known that the addition of a small amount of the lithium-based glass having very low melting temperature often makes it possible to decrease the sintering temperature of many microwave dielectric materials. For example, using 2.0% $Li_2O-ZnO-B_2O_3$ glass, the $ZnTiNb_2O_8$ dielectric can be sintered at 875 °C and obtain good microwave dielectric properties with $\varepsilon_r$=31.8, $Q\times f$=25.013 GHz and $\tau_f=-62\times10^{-6}$ °C$^{-1}$ [16]. GEORGE and SEBASTIAN [17] reported that the lithium magnesium zinc borosilicate glass-doped $Li_2MgTi_3O_8$ ceramic could be sintered at 925 °C, and showed the microwave dielectric properties of $\varepsilon_r$=24.5, $Q\times f$=44000 GHz, $\tau_f=0.2\times10^{-6}$ °C$^{-1}$. Further investigations are still required for lowering its sintering temperature to less than 900 °C and enhancing the microwave dielectric properties so that they could be well co-fired with Ag electrode and improve the quality of devices.

In the present study, $Li_2O-MgO-B_2O_3$ (LMB) system glass was selected as the sintering aids. The LMB glass was chosen because the elements in the glass are Li and Mg, which are also present in the parent material ($Li_2MgTi_3O_8$). This may avoid the formation of secondary phase in the final product as far as possible

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**Corresponding author:** Guo-hua CHEN; Tel: +86-773-2291434; Fax: +86-773-2191903; E-mail: chengh@guet.edu.cn
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[17]. LMB glass additive was made and added to $Li_2MgTi_3O_8$ ceramics in order to investigate the possibility using LMB as a low temperature sintering additive. Furthermore, its effect on the sintering behavior, microstructure and microwave dielectric properties of the $Li_2MgTi_3O_8$ ceramics was investigated. Meanwhile, the compatibility of the LMB glass-added ceramic with Ag electrode was also evaluated.

## 2 Experimental

Specimens of the $Li_2MgTi_3O_8$ ceramics were prepared by a conventional solid-state ceramic route from the high-purity oxide powders (>99.9%, Guo-Yao Co. Ltd., Shanghai, China) of $Li_2CO_3$, MgO and $TiO_2$. Stoichiometric amounts of the powder samples were mixed and ball milled using zirconia balls in ethanol medium for 24 h. The resultant slurry was then dried. The $Li_2MgTi_3O_8$ ceramic powders were calcined at 900 °C for 4 h. The LMB ($30Li_2O-10MgO-60B_2O_3$) glass was prepared from the high purity oxide chemicals of $Li_2CO_3$, MgO and $H_3BO_3$ (99.5%, Guo-Yao Co. Ltd., Shanghai, China). The glass batch filled in an uncovered corundum crucible was melted at 950 °C. The melt was homogenized for 1 h, then quenched and powdered. The glass softening point was 500 °C and the medium grain size of LMB glass powder was about 5 μm. After subsequent ball-milling of $Li_2MgTi_3O_8$ with 0−3.0% LMB, the powders were uniaxially pressed into disks of 12 mm in diameter and 6 mm in thickness under the pressure of about 100 MPa. The pure $Li_2MgTi_3O_8$ pellets were sintered at 1000−1100 °C for 4 h in air and the ceramic pellets added with LMB were sintered at 850−900 °C for 4 h in air.

The crystal structure and phase purity of the powdered samples were analyzed using X-ray diffraction (XRD, D8-ADVANCE, Bruker, Germany) with Cu $K_\alpha$ radiation. The bulk densities of the sintered samples were measured by the Archimedes method. The relative densities of various LMB doped $Li_2MgTi_3O_8$ samples were calculated using the bulk densities divided by their corresponding theoretical densities with the following formula [18]:

$$D=\frac{w_1+w_2}{w_1/D_1+w_2/D_2} \tag{1}$$

where $w_1$ and $w_2$ are the mass fractions of $Li_2MgTi_3O_8$ and LMB glass respectively; $D_1$ and $D_2$ are the theoretical densities of $Li_2MgTi_3O_8$ ceramic and LMB glass, respectively.

The microstructure observation of the samples was performed using scanning electron microscopy (SEM, JEOL JSM−5610LV, Tokyo, Japan). The microwave dielectric properties were measured by a Vector Network Analyzer (N5230C, Agilent Technologies) [19]. The temperature coefficient of resonant frequency ($\tau_f$) was measured in the temperature range of 25−75 °C using the following equation:

$$\tau_f=(f_{75}-f_{25})/(50\times f_{25}) \tag{2}$$

where $f_{75}$ and $f_{25}$ are the resonant frequencies at 75 °C and 25 °C, respectively.

To check the chemical compatibility of the ceramic with the silver powder, 20% powdered silver was mixed and homogenized with the 1% LMB glass-added $Li_2MgTi_3O_8$ ceramic powder [20], and then the mixture was pressed into pellets and fired at 875 °C for 4 h to achieve equilibrium. In addition to the XRD analysis, micro-structural study and line scan were conducted via scanning electron microscopy (SEM, JSM−5610, JEOL, Tokyo, Japan) coupled with energy-dispersive X-ray spectroscopy (EDS).

## 3 Results and discussion

The room-temperature XRD patterns recorded for the pure $Li_2MgTi_3O_8$ ceramic sintered at 1025 °C for 4 h are shown in Fig. 1. All the diffraction peaks of the ceramic sample could be indexed as a cubic structure (*P*4332) $Li_2MgTi_3O_8$ (PDF 89—1308) without any secondary phase, which agreed well with those reported by KAWAI et al [21]. The theoretical density of the $Li_2MgTi_3O_8$ ceramic calculated from XRD data is about 3.50 g/cm$^3$ [15]. The SEM image of the surface of $Li_2MgTi_3O_8$ ceramic sintered at 1025 °C is also shown in Fig. 1. The dense microstructure of $Li_2MgTi_3O_8$ ceramic sintered at 1025 °C for 4 h with only few pores existing can be confirmed by SEM.

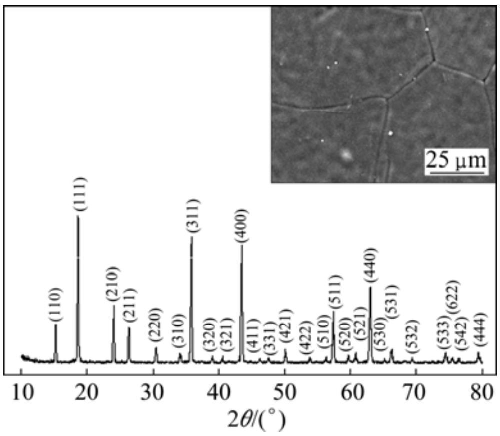


**Fig. 1** XRD pattern of $Li_2MgTi_3O_8$ ceramic sample sintered at 1025 °C for 4 h (Inset shows SEM image of as-sintered $Li_2MgTi_3O_8$ sample)

Figure 2 presents the bulk densities and relative densities of $Li_2MgTi_3O_8$ ceramics as a function of

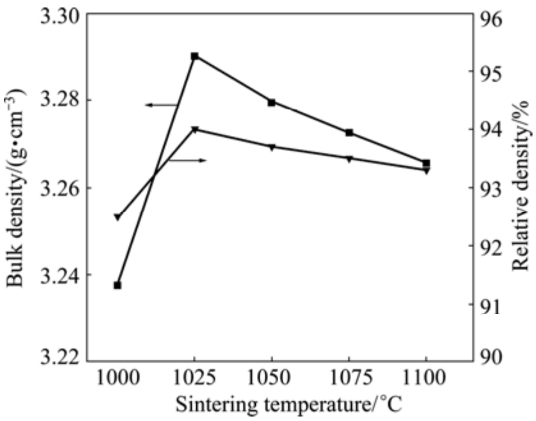


**Fig. 2** Bulk densities and relative densities of $Li_2MgTi_3O_8$ ceramics as function of sintering temperature

sintering temperature. As the sintering temperature increases from 1000 to 1025 °C, the bulk density increases from 3.2375 to 3.2900 g/cm$^3$, which is equivalent to a relative density of about 94.0% of the theoretical density. On further increase in the sintering temperature, the densities of $Li_2MgTi_3O_8$ ceramics slightly decrease. This indicates that the densification temperature of the $Li_2MgTi_3O_8$ ceramic is 1025 °C. The microwave dielectric properties of $Li_2MgTi_3O_8$ ceramics are shown in Fig. 3. The relative permittivity versus sintering temperature for $Li_2MgTi_3O_8$ ceramics has a trend similar to that of the density. As the sintering temperature increases to 1025 °C, the relative permittivity reaches a saturated value of 25.7. The $Q\times f$ value of $Li_2MgTi_3O_8$ ceramics reaches the maximum of 53995 GHz at $f$=5.76 GHz. The $\tau_f$ values of $Li_2MgTi_3O_8$ ceramics are found to be $3.28\times10^{-6}$, $3.34\times10^{-6}$, $4.36\times10^{-6}$, $4.33\times10^{-6}$ and $3.75\times10^{-6}$ °C$^{-1}$ when sintered at 1000, 1025, 1050, 1075 and 1100 °C, respectively. This means that the $\tau_f$ values do not change remarkably with the increase in sintering temperature. In general, the $Li_2MgTi_3O_8$ ceramics sintered at 1025 °C have better

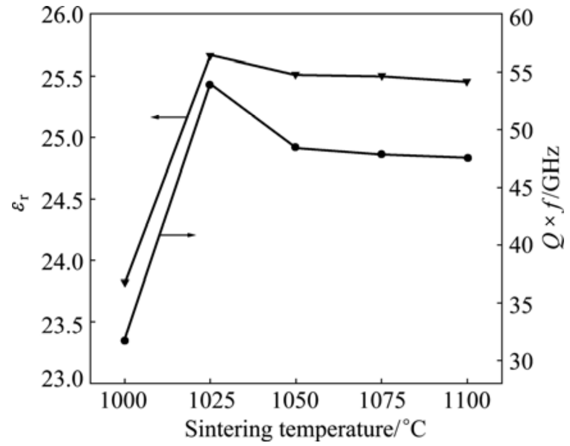


**Fig. 3** Relative permittivity $\varepsilon_r$ and $Q\times f$ of $Li_2MgTi_3O_8$ ceramics as function of sintering temperature

microwave dielectric properties of $\varepsilon_r$=25.7, $Q\times f$=53995 GHz, $\tau_f$=$3.34\times10^{-6}$ °C$^{-1}$. The obtained dielectric properties are different from the results reported by GEORGE and SEBASTIAN [15], which may be due to various raw materials and preparation technology.

To further reduce the sintering temperature of the $Li_2MgTi_3O_8$ ceramics to below 900 °C, a small amount of LMB glass was added into the $Li_2MgTi_3O_8$ samples. Due to the liquid phase effect, the addition of LMB glass can efficiently lower the sintering temperature from 1025 to 875 °C. Figure 4 shows the XRD patterns of the pure $Li_2MgTi_3O_8$ ceramics sintered at 1025 °C and the LMB glass-added ceramics sintered at 875 °C, respectively. It can be found that the XRD patterns are very similar to that of the pure ceramic except for one peak attributed to an unknown second phase with LMB glass ≥2% which is shown in Fig. 4.

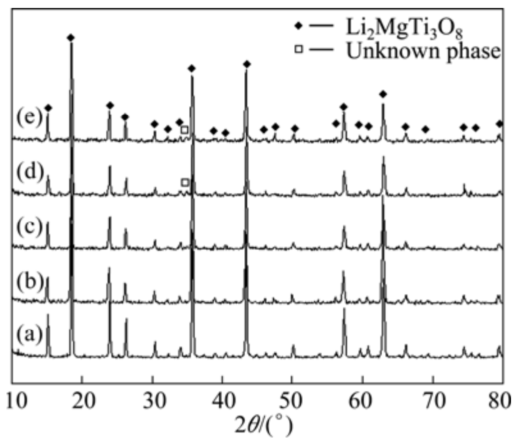


**Fig. 4** XRD patterns of pure $Li_2MgTi_3O_8$ ceramic sintered at 1025 °C for 4 h (a), 0.5% (b), 1.0% (c), 2.0% (d) and 3.0% (e) LMB glass-added $Li_2MgTi_3O_8$ ceramic sintered at 875 °C for 4 h

Figure 5 illustrates the SEM images of the $Li_2MgTi_3O_8$ ceramics added with different amounts of LMB glass sintered at 875 °C for 4 h. For the LMB glass added samples, dense microstructures are formed. The pure $Li_2MgTi_3O_8$ sample sintered at 1025 °C shows a dense microstructure with the average grain size of about 100 μm (see Fig. 1). A reduced grain growth is observed since the sintering aid promotes the densification of ceramics while inhibits the grain growth, so the grain sizes of the LMB glass-added samples are smaller than those of pure $Li_2MgTi_3O_8$ ceramic [22]. Besides, the smaller grain sizes can also be due to the fact that the sintering temperature is decreased by about 150 °C. In general, the LMB glass-added ceramics have a relatively dense microstructure along with the coexistence of large and small grain sizes. However, the grains grow with increasing the amount of LMB additive from 1% to 3% (Figs. 5(b)−(d)). Probably because of too much LMB

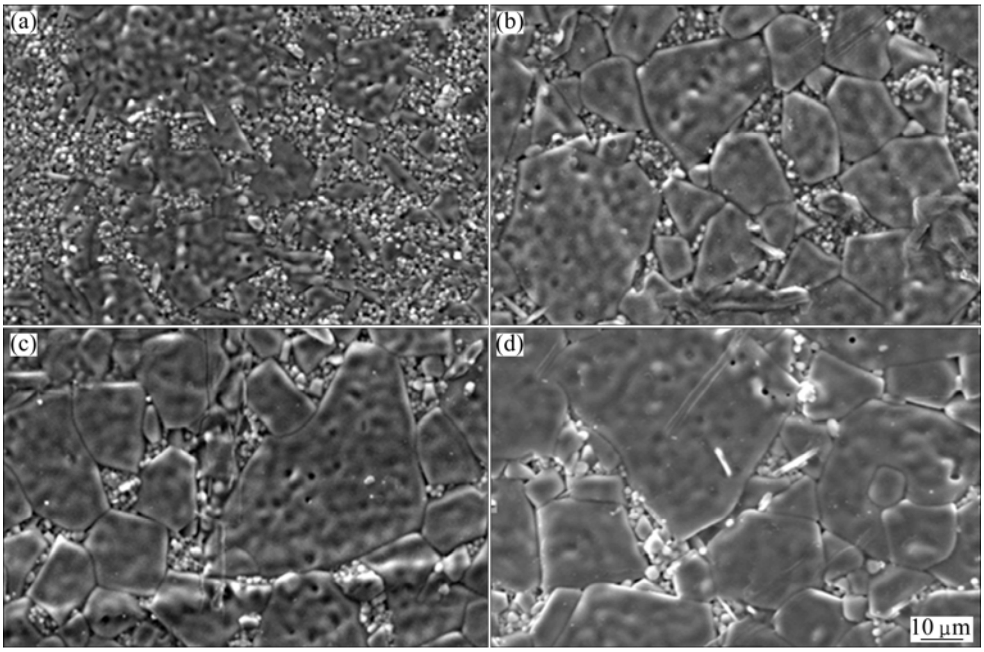


**Fig. 5** SEM images of LMB glass added $Li_2MgTi_3O_8$ sintered at 875 °C for 4 h: (a) 0.5%; (b) 1.0%; (c) 2.0%; (d) 3.0%

glass additive, the LMB glass phase existed in liquid phase state promotes excessively the growth of grains, which is well in agreement with the research results reported by LIU et al [22].

Figure 6 shows the relative densities and microwave dielectric properties of the pellets with the addition of 0.5%−3% LMB glass sintered at 875 °C for 4 h. From Fig. 6(a), it can be seen that the densities of all the LMB glass added samples were higher than that of pure ceramic (3.29 g/cm$^3$) only except for the 0.5% LMB glass added sample, which means that the LMB glass is a very effective low-temperature sintering additive. The relative density obviously increases and then decreases slightly with the increase in LMB glass content. The permittivity changes in a manner similar to the relative density and decreases slightly as the LMB content is increased from 1% to 3%. It is interpreted that a high density would lead to a high relative permittivity owing to lower porosities. The relative permittivity decreases with increasing LMB content (>1%), which may be attributed to the increase of LMB liquid phase with lower $\varepsilon_r$ value. In addition, the unknown secondary phase would hinder the permittivity.

It is verified that many factors affect the microwave dielectric loss and these factors can be divided into two aspects: the intrinsic loss and the extrinsic loss. Intrinsic losses are mainly caused by lattice vibration modes while extrinsic losses are dominated by the second phases, oxygen vacancies, grain sizes and densification or porosity [23]. Some investigations also reported that the $Q\times f$ value is independent of the density and the porosity for a theoretical density higher than 90%. The relative

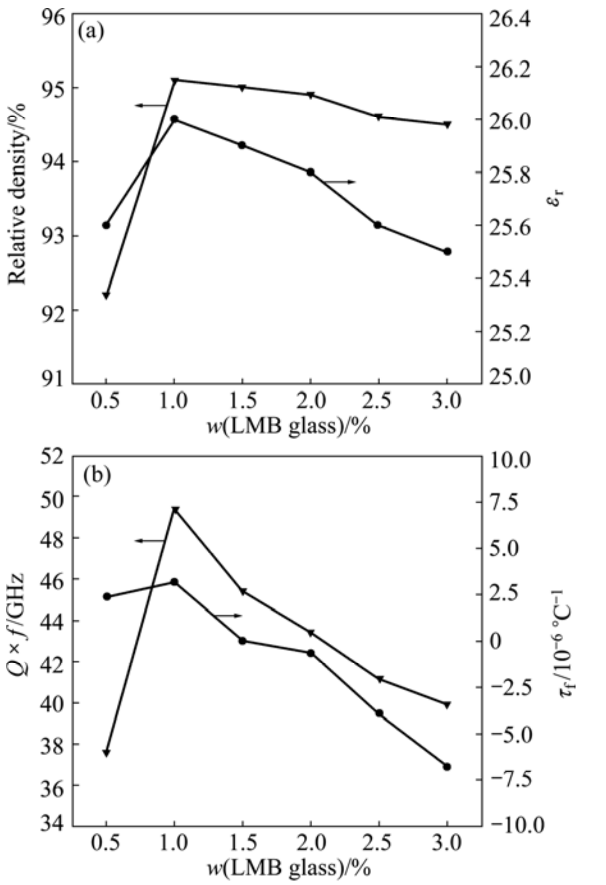


**Fig. 6** Relationship of relative density and permittivity (a), $Q\times f$ values and $\tau_f$ (b) of LMB glass added $Li_2MgTi_3O_8$ ceramic sintered at 875 °C for 4 h

density plays an important role in controlling dielectric loss, which is observed in other microwave dielectric materials [24]. The $Q\times f$ values of $Li_2MgTi_3O_8$ ceramics with various contents of LMB glass addition at 875 °C for 4 h are shown in Fig. 6(b). It can be seen that the $Q\times f$ values have the same tendency as the relative density shown in Fig. 6(a). For the specimens, with the increase in LMB addition, the $Q\times f$ values sharply increase and reach a maximum at 1% LMB, and then obviously decrease. This result may be due to the excessive low $Q\times f$ values LMB glass phase as well as the unknown secondary phase precipitated in the ceramics when the amount of LMB is more than 1%. As a whole, the $\tau_f$ value decreases from $2.36\times10^{-6}$ °C$^{-1}$ to $-6.8\times10^{-6}$ °C$^{-1}$ with increasing the addition of LMB because of negative $\tau_f$ value of LMB glass (see Fig. 6(b)). Optimum properties, i.e. $\varepsilon_r$=25.9, $\tau_f\approx0$ °C$^{-1}$ and $Q\times f$=45403 GHz ($f$=8.12 GHz), are achieved for the 1.5% LMB glass-added $Li_2MgTi_3O_8$ ceramic sintered at 875 °C for 4 h. It is found that near zero $\tau_f$ and good microwave dielectric properties can be easily obtained by adjusting the amount of LMB additive in the ceramic samples.

In order to evaluate the chemical compatibility of $Li_2MgTi_3O_8$ ceramic with silver electrode, mixtures of 1% LMB glass added $Li_2MgTi_3O_8$ ceramic powders with 20% Ag powders were co-fired and analyzed to detect interactions between the low-fired ceramic samples and electrodes [25]. The XRD pattern of $Li_2MgTi_3O_8$ ceramics added with 1% LMB co-fired with Ag at 875 °C for 4 h is presented in Fig. 7. The XRD result reveals hardly any new phase formation after firing. The reaction of low-fired $Li_2MgTi_3O_8$ ceramics with Ag electrodes does not occur.

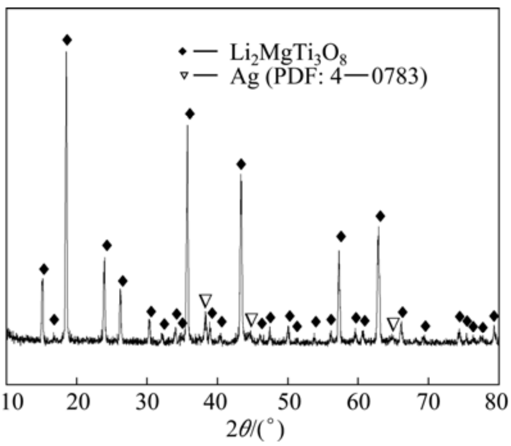


**Fig. 7** XRD pattern for 1% LMB glass added $Li_2MgTi_3O_8$ ceramic mixed with 20% Ag sintered at 875 °C for 4 h

Figure 8 shows the SEM image and EDS line scanning (from $A$ to $C$) between the silver electrode layer (from $A$ to $B$) and 1% LMB glass-added $Li_2MgTi_3O_8$ co-fired at 875 °C for 4 h. Four elements of Mg, O, Ag and Ti can be detected as shown in Fig. 8(b). However, it is difficult to detect lithium (Li) and boron (B) ions by EDS due to its detection limit. The silver profile sharply decreases to near zero level at the interface shown in Fig. 8(b), which indicates that the reaction between low fired $Li_2MgTi_3O_8$ ceramics and silver electrode does not occur. Therefore, $Li_2MgTi_3O_8$ ceramics with LMB glass additive could be selected as a promising candidate for low temperature co-fired ceramics application.

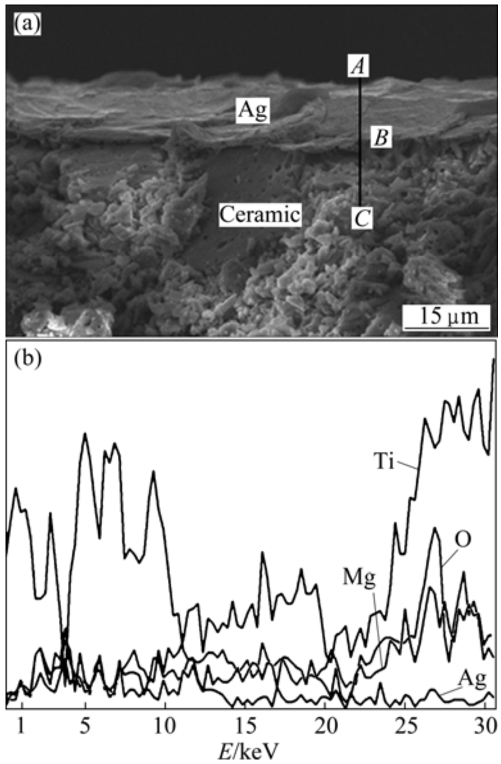


**Fig. 8** SEM image (a) and EDS line scanning (b) (from $A$ to $C$) between silver electrode layer (from $A$ to $B$) and 1% LMB glass-added $Li_2MgTi_3O_8$ ceramic, co-fired at 875 °C for 4 h

## 4 Conclusions

1) The LMB glass-added $Li_2MgTi_3O_8$ ceramic materials are prepared by the conventional solid-state ceramic route. The effects of LMB glass addition on the sinterability and microwave dielectric properties of $Li_2MgTi_3O_8$ dielectric ceramics were investigated for LTCC applications.

2) The sintering temperature of $Li_2MgTi_3O_8$ ceramics can be reduced from 1025 to 875 °C when a small amount of LMB glass is added. The addition of LMB glass induces only a limited degradation of the microwave dielectric properties. Typically, a dense $Li_2MgTi_3O_8$ ceramic doped with 1.5% LMB is obtained when sintered at 875 °C for 4 h, which shows microwave dielectric properties of $\varepsilon_r$=25.9, $Q\times f$=45403 GHz and $\tau_f\approx0$ °C$^{-1}$.

3) The materials developed in the present investigation are excellent in terms of sintering temperature, dielectric properties, and cost of raw

materials compared with other available high-quality dielectric ceramic materials. This material can be compatible with Ag electrodes, which makes it a promising candidate for LTCC application.

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# 添加 $Li_2O$−MgO−$B_2O_3$ 玻璃的 $Li_2MgTi_3O_8$ 陶瓷的微波介电性能及其与 Ag 共烧相容性


包 燕[1]，陈国华[1,2]，侯美珍[1]，杨 云[1]，韩作鹏[1]，邓开能[1]

1. 桂林电子科技大学 材料科学与工程学院，桂林 541004；

2. 桂林电子科技大学 广西信息材料重点实验室，桂林 541004



**摘 要**：研究添加 $Li_2O$−MgO−$B_2O_3$ 玻璃对 $Li_2MgTi_3O_8$ 陶瓷的烧结特性、相纯度、微观组织和微波介电性能的影响。结果表明：添加少量的玻璃能有效地将陶瓷的烧结温度从 1025 °C 降低到 875 °C，且没有恶化陶瓷的微波介电性能。添加 1.5% 玻璃的陶瓷在 875 °C 烧结 4 h 后具有优良的微波介电性能能，其介电常数 $\varepsilon_r$=25.9，品质因数 $Q\times f$= 45403 GHz，谐振频率温度系数 $\tau_f\approx0$。陶瓷和 Ag 电极共烧几乎不发生化学反应，表现为良好的化学相容性。所制备的陶瓷可望用于低温烧结的多层微波器件。



**关键词**：微波陶瓷；介电性能；玻璃；低温烧结；相容性



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