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Phase evolution and microwave dielectric properties of $(Bi_{1-x}Ln_x)_2MoO_6$ (Ln=Nd and La, $x \leq 0.3$) ceramics

Li-Xia Pang [a], Wei-Guo Liu [a], Di Zhou [b,c,*], Zhen-Xing Yue [d]

[a] Laboratory of Thin Film Techniques and Optical Test, Xi'an Technological University, Xi'an 710032, Shaanxi, China
[b] Electronic Materials Research Laboratory, Key Laboratory of the Ministry of Education & International Center for Dielectric Research, Xi'an Jiaotong University, Xi'an 710049, Shaanxi, China
[c] Department of Materials Science and Engineering, University of Sheffield, S1 3JD, UK
[d] State Key Laboratory of New Ceramics and Fine Processing, Department of Materials Science and Engineering, Tsinghua University, Beijing 100084, China

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ABSTRACT

$(Bi_{1-x}Ln_x)_2MoO_6$ (Ln=Nd and La) ceramics were prepared by conventional solid state reaction method. Solid solutions crystallizing in a monoclinic structure with a space group *P*21/*c* were obtained in the $(Bi_{1-x}La_x)_2MoO_6$ ($x \leq 0.3$) ceramics. For the $(Bi_{1-x}Nd_x)_2MoO_6$ ceramics, tetragonal Nd_2MoO_6 phase with a space group $I4_1/acd$ was detected when x value reached 0.2 besides the Bi_2MoO_6 solid solution. Temperature stability and quality factor Qf value at microwave frequencies were improved by appropriate substitution for Bi^{3+} by Nd^{3+} and/or La^{3+} in Bi_2MoO_6 system. When x=0.2, for both $(Bi_{1-x}Nd_x)_2MoO_6$ and $(Bi_{1-x}La_x)_2MoO_6$ ceramics, Qf values reached maximum about 30,200 GHz and 32,900 GHz, respectively. TCF value shifted towards zero with substitution of Bi^{3+} by Nd^{3+} and/or La^{3+}. When x=0.3, TCF values of $(Bi_{1-x}Nd_x)_2MoO_6$ and $(Bi_{1-x}La_x)_2MoO_6$ ceramics were −70 and −85 ppm/°C, respectively. Relation between crystal structure, microstructure and microwave dielectric properties were discussed.

1. Introduction

The development of high-speed and high-frequency digital devices, which are capable of transferring large amounts of information in a short time is one of the main strategic drivers of the consumer electronics market. Meanwhile, demands for miniaturization and performance enhancement of microwave devices have increased with the development of the modern communication system. Thus, there have been increasing investigations on microwave dielectric material with a high-quality factor (Qf value) to decrease the noise and insertion loss selectivity and a near-zero temperature coefficient of resonant frequency (TCF value) for achieving excellent thermal stable microwave devices [1]. At the same time, different microwave devices need materials with different permittivities. For example, as microwave substrates, the use of low relative permittivity (ε_r) materials lower than that of alumina (ε_r=9.8) can reduce the signal propagation delay sufficiently to offer immense potential for high-frequency applications [2,3], while a large dielectric constant (ε_r) is also required for the microwave dielectric material to miniaturize the microwave resonators because the size of the device is inversely proportional to $\varepsilon_r^{1/2}$ [4].

A great number of excellent microwave dielectric materials were developed, such as $Sr_{2-x}Ca_xAl_2SiO_7$ [5], ZnO-Nb_2O_5 [6], Bi(Nb, Ta, Sb)O_4 [7–9], Li_2O-Nb_2O_5-TiO_2 [10], (Zr, Sn)TiO_4 [11], BaO-TiO_2-Nb_2O_5 [12], and the $(A_1A_2)(B_1B_2)O_3$ complex perovskite system [13]. While most of them need high sintering temperatures above 1200 °C. Nowadays, investigations on new compounds with intrinsically lower sintering temperatures have attracted much more attention [14–17]. In the Bi_2O_3-MoO_3 system, six compounds with pure phase were reported and all of them were glass-free low-firing microwave dielectric materials. [18] The Bi_2MoO_6 ceramic with a monoclinic structure was reported to possess a low sintering temperature (750 °C), a high Qf value (16,700 GHz), and a negative TCF value (−114 ppm/°C). TCF value could be tailored to near zero by ceramic composites, such as Bi_2MoO_6-TiO_2 composites, and a temperature stable low-firing microwave dielectric material was obtained in our previous work [19]. Substitution is another good way to modify TCF values of microwave dielectrics [20–23]. Rare earth cations, such as La^{3+} and Nd^{3+}, have the same chemical valence and similar ionic radii to Bi^{3+}, so solid solutions

* Corresponding author at: Electronic Materials Research Laboratory, Key Laboratory of the Ministry of Education & International Center for Dielectric Research, Xi'an Jiaotong University, Xi'an 710049, Shaanxi, China.
E-mail address: zhoudi1220@gmail.com (D. Zhou).

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might be formed in the $(Bi_{1-x}Ln_x)_2MoO_6$ (Ln=Nd and La) system. According to literatures' reports, both Nd_2MoO_6 and La_2MoO_6 can crystallize as tetragonal crystal structure with a $I4_1/acd$ space group [24,25]. The La_2MoO_6 is a kind of optical materials which can be activated with the trivalent rare earth ions [26], and the Nd_2MoO_6 ceramic, which can be densified well at 1350 °C, is a microwave dielectric material with a permittivity (ε_r) of 13.8, a Qf value of 66,400 GHz, and a TCF value of −53 ppm/°C [27]. In the present work, $(Bi_{1-x}Ln_x)_2MoO_6$ (Ln=Nd and La, $x \le 0.3$) ceramics were prepared by conventional solid state reaction method, and sintering behavior, phase evolution, microstructure and microwave dielectric properties were investigated in detail.

2. Experimental procedure

Proportionate amounts of reagent-grade Bi_2O_3 (>99%, Shu-Du Powders Co. Ltd., Chengdu, China), La_2O_3 (>99.99%, Sinopharm Chemical Reagent Co., Ltd, Shanghai, China), Nd_2O_3 (>99.99%, Sinopharm Chemical Reagent Co., Ltd, Shanghai, China), and MoO_3 (>99%, Fuchen Chemical Reagents, Tianjin, China) were prepared according to the stoichiometric compound formulation of $(Bi_{1-x}Ln_x)_2MoO_6$ (Ln=Nd and La, x=0.0, 0.1, 0.2, and 0.3). Powders were mixed and milled for 4.5 h using a planetary mill (Nanjing Machine Factory, Nanjing, China). The mixtures were dried and calcined at 550–650 °C for 5 h. The calcined powders were ball milled again for 5 h, and then the dried powders were pressed into cylinders (10 mm in diameter and 4–5 mm in height) in a steel die with 5 wt% PVA binder addition under a uniaxial pressure of 20 MPa. Samples were sintered at temperatures from 830 to 1030 °C for 2 h.

Densities of the sintered specimens were measured by Archimedes method. To examine phase structure, sintered ceramics were crushed into powders. Room temperature X-ray diffraction (XRD) measurement was performed by using a XRD with Cu Kα radiation (Rigaku D/MAX-2400 X-ray diffractometry, Tokyo, Japan). The ss-fired samples were observed by using scanning electron microscopy (SEM) (JSM-6460, JEOL, Tokyo, Japan) to examine grain morphology. Dielectric properties at microwave frequencies were measured according to the $TE_{01\delta}$ dielectric resonator method with a network analyzer (HP 8720 Network Analyzer, Hewlett-Packard) and a temperature chamber (Delta 9023, Delta Design, Poway, CA). Temperature coefficient of resonant frequency TCF was calculated with the following formula:

$$TCF = \frac{f_T - f_{T_0}}{f_{T_0} \times (T - T_0)} \times 10^6 \text{ ppm/°C}, \quad (1)$$

where f_T and f_{T0} are the $TE_{01\delta}$ resonant frequencies at temperature T and T_0 (room temperature), respectively.

3. Results and discussion

Fig. 1 presents X-ray diffraction patterns of the $(Bi_{1-x}Nd_x)_2MoO_6$ and $(Bi_{1-x}La_x)_2MoO_6$ ceramics. Almost all the diffraction peaks of the samples (except $(Bi_{0.7}Nd_{0.3})_2MoO_6$ ceramics) can be indexed on a monoclinic cell, which was similar with parent Bi_2MoO_6. It indicates that solid solutions, which crystallized as single monoclinic phase structure with space group $P21/c$, were obtained in $(Bi_{1-x}La_x)_2MoO_6$ ($x \le 0.3$) and $(Bi_{1-x}Nd_x)_2MoO_6$ ($x \le 0.2$) ceramics. The diffraction peaks (080) and (002) separated from each other with the substitution of Bi^{3+} by Nd^{3+} and/or La^{3+}, because the cell parameters varied with substitution. In the $(Bi_{0.7}Nd_{0.3})_2MoO_6$ ceramics, a small amount of tetragonal Nd_2MoO_6 phase with space group $I4_1/acd$ was detected besides the Bi_2MoO_6 solid solution phase. As we know, both pure Nd_2MoO_6 and La_2MoO_6 crystallize as tetragonal crystal structure with $I4_1/acd$ space group [24,25], which is quite different from that of Bi_2MoO_6, so solid solutions couldn't be obtained in the whole range of $(Bi_{1-x}Ln_x)_2MoO_6$ (Ln=Nd and La). According to Shannon's result, ionic radius of La^{3+} (1.160 Å) is much closer to that of Bi^{3+} (1.170 Å), and the ionic radius of Nd^{3+} (1.109 Å) is much smaller [28]. Thus, the solid solubility of $(Bi_{1-x}La_x)_2MoO_6$ is a bit higher than that of $(Bi_{1-x}Nd_x)_2MoO_6$, as the case here.

Fig. 2 shows the bulk densities and relative densities of the $(Bi_{1-x}Nd_x)_2MoO_6$ and $(Bi_{1-x}La_x)_2MoO_6$ ceramics as a function of sintering temperature. Bulk densities of $(Bi_{1-x}Ln_x)_2MoO_6$ (Ln=Nd and La) ceramics decreased with the contents of Nd^{3+} and/or La^{3+} due to their smaller atomic weights than that of Bi. For parent Bi_2MoO_6 ceramic, its bulk density reached maximum value and relative density kept above 96% when sintering temperature ≥850 °C. The sintering temperature increased with the substitution of Bi^{3+} by Nd^{3+} and/or La^{3+}. For both $(Bi_{0.7}Nd_{0.3})_2MoO_6$ and $(Bi_{0.7}La_{0.3})_2MoO_6$ ceramics, they could not be densified well unless the sintering temperatures were higher than 970 °C. Meanwhile, the bulk densities and relative densities would decrease when sintering temperatures went further, for all the $(Bi_{1-x}Ln_x)_2MoO_6$ (Ln=Nd and La) ceramics, which might result from the Bi volatilization at higher sintering temperatures. So the sintering

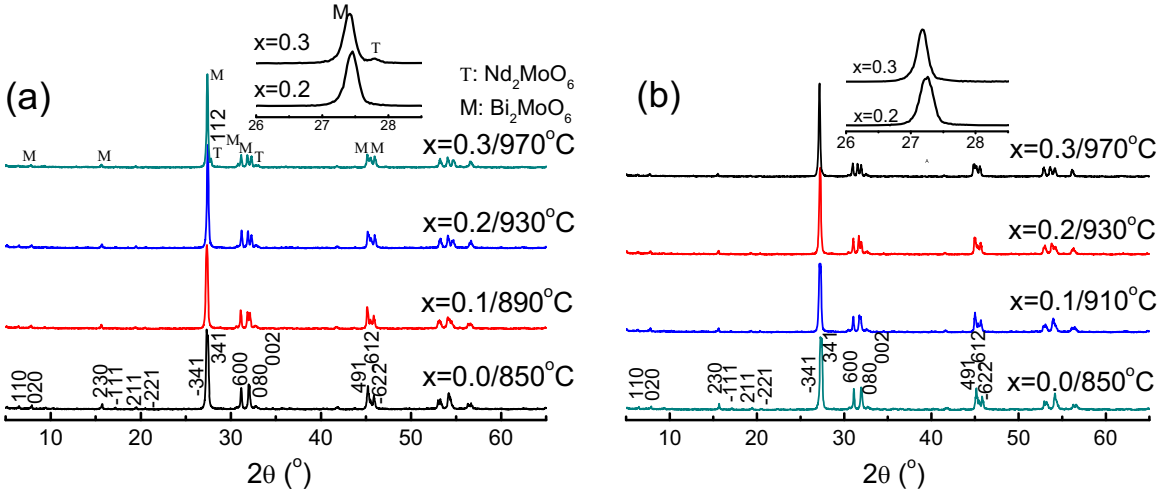

Fig. 1. X-ray diffraction patterns of (a) $(Bi_{1-x}Nd_x)_2MoO_6$ and (b) $(Bi_{1-x}La_x)_2MoO_6$ ceramics.

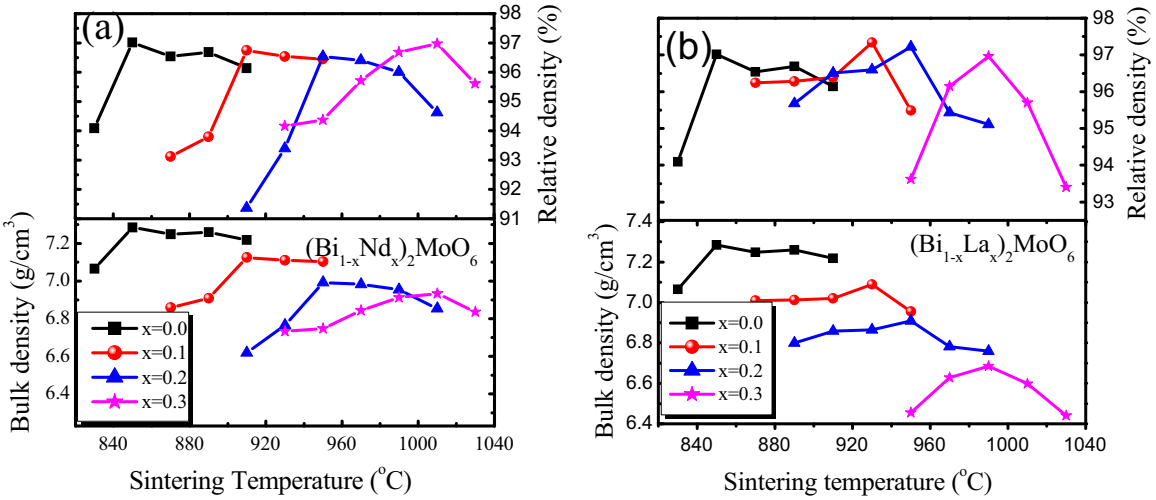

Fig. 2. Bulk densities and relative densities of the $(Bi_{1-x}Nd_x)_2MoO_6$ (a) and $(Bi_{1-x}La_x)_2MoO_6$ (b) ceramics as a function of sintering temperature.

temperatures could not be too high to densify $(Bi_{1-x}Ln_x)_2MoO_6$ (Ln=Nd and La) ceramics.

Fig. 3 presents SEM images of as-fired surfaces of the $(Bi_{1-x}Nd_x)_2MoO_6$ and $(Bi_{1-x}La_x)_2MoO_6$ ceramics. All the ceramics had been well densified with little pores inside. The gains were uniform and there was no secondary phase grains in all $(Bi_{1-x}La_x)_2MoO_6$ ($x \leq 0.3$) ceramics and $(Bi_{0.9}Nd_{0.1})_2MoO_6$ ceramic. In the $(Bi_{0.8}Nd_{0.2})_2MoO_6$ and $(Bi_{0.7}Nd_{0.3})_2MoO_6$ ceramics, a small amount of secondary phase grains with octahedral shape were detected in the grain boundaries, which might be tetragonal Nd_2MoO_6 grains according to the XRD results above. It means that the secondary phase appeared in $(Bi_{0.8}Nd_{0.2})_2MoO_6$ ceramic, but the amount was too small to be detected by XRD experimental. On the other hand, the secondary phase grains gathered to the surface of the sample, so they can only be observed on the surface from SEM image.

Microwave dielectric properties of $(Bi_{1-x}Nd_x)_2MoO_6$ and $(Bi_{1-x}La_x)_2MoO_6$ ceramics were shown in Fig. 4. Permittivity decreased gradually with the substitution for Bi^{3+} by Nd^{3+} and/or La^{3+} because of their smaller dielectric polarizabilities of Nd^{3+} (5.01 Å^3) and La^{3+} (6.07 Å^3) than Bi^{3+} (6.12 Å^3) [29]. Permittivity decreased from 31.5 of pure Bi_2MoO_6 ceramic to 22.6 at x=0.3 for $(Bi_{1-x}Nd_x)_2MoO_6$ ceramics and to 20.7 at x=0.3 for $(Bi_{1-x}La_x)_2MoO_6$ ceramics, respectively. Permittivity of the $(Bi_{1-x}Nd_x)_2MoO_6$ ceramic was a little larger than that of $(Bi_{1-x}La_x)_2MoO_6$ ceramic with the same x value, which might be attributed to the influence of the secondary phase Nd_2MoO_6.

It is shown in Fig. 4(b) that Qf value first increased remarkably

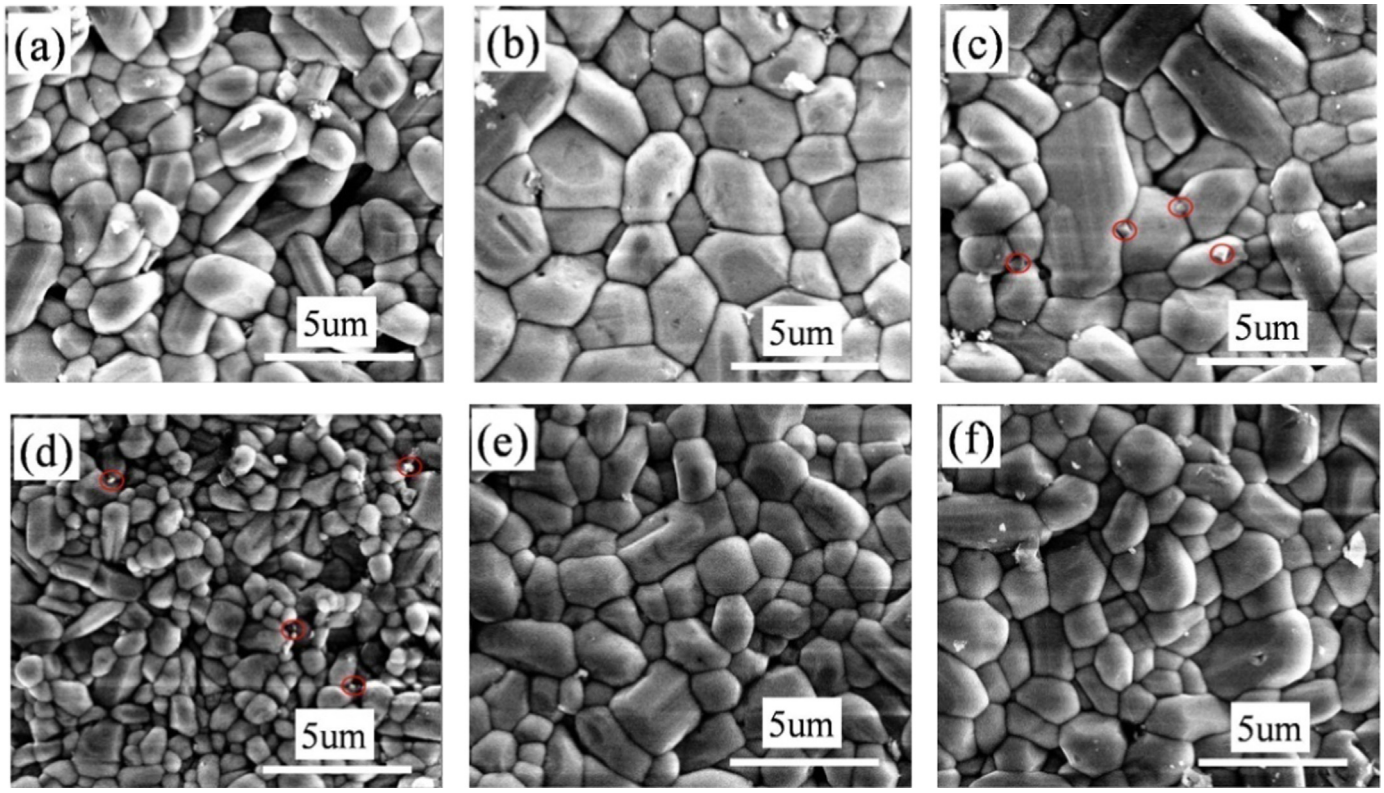

Fig. 3. SEM images of as-fired surface of (a) Bi_2MoO_6 sintered at 850 °C, (b) $(Bi_{0.9}Nd_{0.1})_2MoO_6$ sintered at 910 °C, (c) $(Bi_{0.8}Nd_{0.2})_2MoO_6$ sintered at 930 °C, (d) $(Bi_{0.7}Nd_{0.3})_2MoO_6$ sintered at 970 °C, (e) $(Bi_{0.9}La_{0.1})_2MoO_6$ sintered at 890 °C, and (f) $(Bi_{0.8}La_{0.2})_2MoO_6$ sintered at 930 °C.

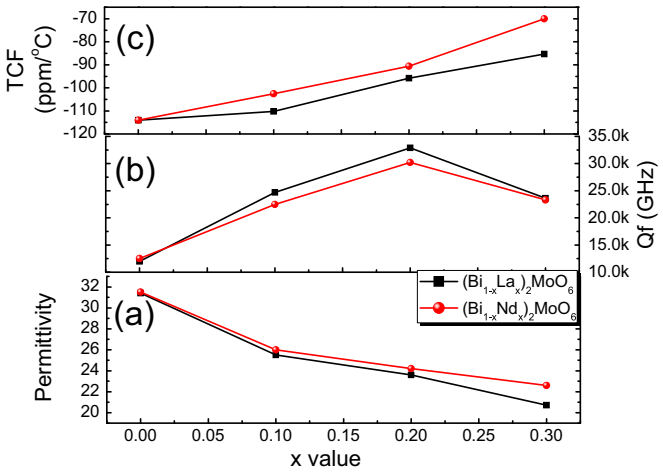

Fig. 4. Microwave dielectric properties of the $(Bi_{1-x}Nd_x)_2MoO_6$ and $(Bi_{1-x}La_x)_2MoO_6$ ceramics: (a) permittivity; (b) Qf value; (c) TCF value.

Table 1
Sintering Temperatures and microwave dielectric properties of several typical microwave dielectrics.

Sample	S.T (°C)	Permittivity	Qf value (GHz)	TCF (ppm/°C)	Reference
$SmAlO_3$	1650	20.4	65,000	−74	[31]
$LaAlO_3$+0.25 wt% CuO	1460	20.7	48,000	−80	[33]
$BaTe_2O_6$	650	21	50,300	−51	[32]
$NdAlO_3$	1650	22.3	58,000	−33	[31]
$LiNb_3O_8$	1075	24	58,000	−96	[30]
$(Bi_{0.8}La_{0.2})_2MoO_6$	930	23.6	32,900	−96	This work
$(Bi_{0.8}Nd_{0.2})_2MoO_6$	930	24.2	30,200	−91	This work
$Li_2ZnTi_3O_8$	1075	25.6	72,000	−11.2	[34,35]
$Li_2MgTi_3O_8$	1075	27.2	42,000	+3.2	[34,35]

S.T.: Sintering Temperature.

and then decreased slightly with the increase of substitution amount. Maximum Qf values were achieved at x=0.2, and the maximum Qf values were 30,200 GHz in $(Bi_{0.8}Nd_{0.2})_2MoO_6$ ceramics and 32,900 GHz in $(Bi_{0.8}La_{0.2})_2MoO_6$ ceramics, respectively. Decrease in Qf value in x=0.3 sample might be caused by the escape of Bi at high sintering temperature. TCF value of the $(Bi_{1-x}Ln_x)_2MoO_6$ (Ln=Nd and La) ceramics shifted towards zero with the substitution for Bi^{3+} by Nd^{3+} and/or La^{3+}, as shown in Fig. 4(c). When x=0.3, the TCF values of $(Bi_{1-x}Nd_x)_2MoO_6$ and $(Bi_{1-x}La_x)_2MoO_6$ ceramics were −70 and −85 ppm/°C, respectively. It means that both temperature stability and Qf value can be improved by appropriate substitution of Bi^{3+} by Nd^{3+} and/or La^{3+} in Bi_2MoO_6 system. Table 1 presents the sintering temperatures and microwave dielectric properties of several typical microwave dielectrics with permittivity 20–24 [30–33]. Comparing with these dielectrics, the $(Bi_{1-x}Ln_x)_2MoO_6$ (Ln=Nd and La) ceramics were nontoxic and processed a lower sintering temperature, which makes it possible for low temperature co-fired ceramics technology.

4. Conclusions

Microwave dielectric ceramics, $(Bi_{1-x}Nd_x)_2MoO_6$ and $(Bi_{1-x}La_x)_2MoO_6$, were prepared by conventional solid state reaction method. Solid solutions with monoclinic structure were obtained for $(Bi_{1-x}La_x)_2MoO_6$ ceramics when $x \leq 0.3$. While in the $(Bi_{1-x}Nd_x)_2MoO_6$ ceramics, tetragonal Nd_2MoO_6 phase with space group $I4_1/acd$ appeared when $x \geq 0.2$ besides the Bi_2MoO_6 solid solution phase. With the increase of substitution amount, sintering temperature of the $(Bi_{1-x}Nd_x)_2MoO_6$ and $(Bi_{1-x}La_x)_2MoO_6$ ceramics increased from 850 °C to about 970 °C, and permittivity decreased from 31.5 to 22.6 and 20.7, respectively. Both temperature stability and Qf value were improved. Maximum Qf values were 30,200 GHz in $(Bi_{0.8}Nd_{0.2})_2MoO_6$ ceramics and 32,900 GHz in $(Bi_{0.8}La_{0.2})_2MoO_6$ ceramics, respectively. TCF value of $(Bi_{1-x}Ln_x)_2MoO_6$ (Ln=Nd and La) ceramics shifted towards zero with the substitution of Bi^{3+} by Nd^{3+} and/or La^{3+}. When x=0.3, TCF values of $(Bi_{1-x}Nd_x)_2MoO_6$ and $(Bi_{1-x}La_x)_2MoO_6$ ceramics were −70 and −85 ppm/°C, respectively.

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