

# Effects of Dy<sub>2</sub>O<sub>3</sub> on the Dielectric Property, Phase Composition, and Microstructure of the Low Temperature Sintered AlN Ceramics

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## Abstract

The effects of Dy<sub>2</sub>O<sub>3</sub> on the phase composition, microstructure, and the dielectric property, especially, dielectric constant, of AlN ceramics sintered at low-temperature (1650°C), were investigated systematically. X-ray diffraction (XRD) was employed to identify the phase compositions of the ceramics during the sintering. Scanning electronic microscopy (SEM) was used to observe the microstructures of the AlN ceramics. The results show that the dielectric constant changes with the amount of Dy<sub>2</sub>O<sub>3</sub>. When the amount of Dy<sub>2</sub>O<sub>3</sub> is less than 0.93 wt.%, the dielectric constant increases with the increased amount of Dy<sub>2</sub>O<sub>3</sub>. The dielectric constant of the AlN ceramics is higher than 200 when the amount of Dy<sub>2</sub>O<sub>3</sub> is 0.93 wt.%. However, when the amount of Dy<sub>2</sub>O<sub>3</sub> is more than 0.93 wt.%, the dielectric constant decreases with the increased amount of Dy<sub>2</sub>O<sub>3</sub>. The dielectric constant of the AlN ceramics with 1.87 wt.% Dy<sub>2</sub>O<sub>3</sub> is only about 15, which closed to the theoretical value of pure AlN ceramics. The analysis illuminated that the grain phases changed from Al<sub>2</sub>O<sub>3</sub>-rich aluminate to AlDyO<sub>3</sub>, which existed at triple pockets with the increased amount of Dy<sub>2</sub>O<sub>3</sub>, the former can improve the dielectric constant greatly due to ion relaxation polarization.

**Keywords:** Dy<sub>2</sub>O<sub>3</sub>, dielectric properties, phase composition, microstructure, AlN ceramics

## 1. Introduction

AlN has received growing interests in the electronics industry because of the need for smaller and more reliable integrated circuits and the need for higher voltage devices for power applications (Luo & Zhang, 2005). AlN ceramics are used as electronics materials because of its excellent properties, such as high resistivity, high thermal conductivity, low dielectric constant and thermal expansion coefficient close to that of silicon.

However, it is difficult to sinter AlN ceramics due to its high covalent bonding (Sheppard, 1990). High temperature (e.g. 1900°C) and/or high pressures are required in most of cases (Kume & Yasuoka, 2005). A common solution to lower down sintering temperature is to add sintering additives to improve liquid-phase sintering (Qiao & Zhou, 2003; Qiu & Hotta, 2006). Especially, using multiple component additives (Qiao & Zhou, 2003; Watari & Hwang, 1999; Boey & Tok, 2002) was the effective route to lower down sintering temperature. Apart from enhancing densification, the additives could also affect the dielectric properties since these additives mainly existed in grain boundaries as a second phase (Hagen & Yu, 2002; Kume & Yasuoka, 2005; Kume & Yasuoka, 2005). Using nanopowders as raw materials is another novel resolution. The sinterability is found to be improved greatly by lowering down the size (Panchula & Ying, 2003; Qiu & Hotta, 2006). However, there are some opposite opinions on using nanopowders due to their high agglomeration (Suehiro & Hirotsaki, 2003).

When AlN ceramics were used as substrate and package materials for integrated circuits (ICs), not only dielectric loss but also dielectric constant should be necessary low for the sake of shortening signal delay time. As far as high pure AlN ceramics with few or only one additive are concerned, their dielectric constants are almost same to one another. However, when multiple component additives were used for low-temperature sintering, the complicated microstructure of AlN ceramics was always unavoidable. Under this condition, the dielectric constant and dielectric loss varied significantly. To date, many literatures paid much more attention on

the dielectric loss, especially for high pure AlN ceramics (Kume & Yasuoka, 2005; Hagen & Yu, 2002), but less on dielectric constant. Since the dielectric constant is equally important to substrate and package materials, it is significant to explore the effects of those sintering aids on the dielectric constant.

Dy<sub>2</sub>O<sub>3</sub> is one of rare earth oxides that are typically used as sintering aids for AlN. It has been reported that Dy<sub>2</sub>O<sub>3</sub> is effective for improving the densification and thermal conductivity (Du & Qin, 2007). In this study, AlN with 0-1.87 wt.% Dy<sub>2</sub>O<sub>3</sub> sintered at 1650°C were prepared, and the effects of Dy<sub>2</sub>O<sub>3</sub> addition on the phase composition, microstructure and dielectric properties of AlN ceramics are investigated.

## 2. Experimental

The starting materials were commercially available CaO, Y<sub>2</sub>O<sub>3</sub>, Dy<sub>2</sub>O<sub>3</sub> (analytical purity), AlN with micrometer size powder (Tokuyama soda, Japan, grade H, D<sub>50</sub>=2.55 μm, oxygen content 0.85 wt.%) and high purity Al<sub>2</sub>O<sub>3</sub> (purity 99.99%, Xinmeiyu, China, D<sub>50</sub>=0.6 μm). Samples were prepared through conventional ceramic fabrication process. With polyvinylbutyral (PVB) solution (5%-10%, ethanol as solvent) as binder, AlN powder and additives were planetary milled using ethanol as mixing medium (mass ratio of raw powders: ball: ethanol = 1:2.2:1.2) for 6 hours. After drying, the powder mixture was uniaxially die-pressed into 20 mm diameter and 3 mm thick pills at 10 MPa for 1 min. The compositions of the samples are shown in Table 1. The green bodies were placed into Al<sub>2</sub>O<sub>3</sub> crucibles which is full of carbon powders. Then the crucibles were covered with lids to form a 'sealed' environment. These green bodies were embedded within carbon powders and sintered under a weak reducing atmosphere formed by carbon powder. Sintering process was as follows: firstly sintering at 1650°C for 3h; then cooled to annealing temperature (1600°C) for 5 minutes, and soaking for 6 h at 1600°C; finally, the samples are cooled to room temperature, naturally.

In order to understand the formation of the liquid during sintering, for samples DO1, DO2 and DO3, Al<sub>2</sub>O<sub>3</sub> were used to replace AlN in D1, D3 and D5, which added as one of the reactants to examine the phases in the sintering.

Table 1. Chemical compositions of the starting materials

sample	AlN (wt.%)	Y <sub>2</sub> O <sub>3</sub> (wt.%)	CaO (wt.%)	Dy <sub>2</sub> O <sub>3</sub> (wt.%)	Al <sub>2</sub> O <sub>3</sub> (wt.%)
D1	93.46	3.74	2.80	0	0
D2	93.46	3.74	2.33	0.47	0
D3	93.46	3.74	1.87	0.93	0
D4	93.46	3.74	1.40	1.40	0
D5	93.46	3.74	0.93	1.87	0
DO1	0	37.4	28.0	0	34.6
DO2	0	37.4	18.7	9.3	34.6
DO3	0	37.4	9.3	18.7	34.6

The densities and water absorbability of the sintered pellets were measured using Archimedes's method and the data was obtained by averaging 3-5 readings. The fracture surfaces of the pellets were observed by scanning electron microscopy (JSM-6700F, Japan). The XRD patterns were recorded with a X'Pert-Pro MPD diffractometer (PANalytical, Holland) using Cu K $\alpha$  radiation ( $\lambda=1.5406 \text{ \AA}$ ). To measure dielectric property of the AlN samples, machining and polishing and then coating Ag were performed. Dielectric properties were measured from 1 Hz-10 MHz at room temperature by using Novocontrol Concept, Broad Band Dielectric Spectrometer.

## 3. Results and Discussion

### 3.1 Physical Properties

Figure 1 shows the water absorbability and density of the samples with different amount of Dy<sub>2</sub>O<sub>3</sub>. It can be observed that the water absorbability of all of the samples is less than 0.1% regardless of the amount of Dy<sub>2</sub>O<sub>3</sub>, which means that all the samples were almost fully densified. The water absorbability depends on the amount of

$Dy_2O_3$ . When the amount of the  $Dy_2O_3$  is less than 0.93%, the water absorbability decreased with increasing amount of  $Dy_2O_3$ . When the amount of the  $Dy_2O_3$  is more than 0.93%, the water absorbability then increased with increasing amount of  $Dy_2O_3$ . In contrast to water absorbability, the density shows different trend, it increased with the increase of the amount of  $Dy_2O_3$ .

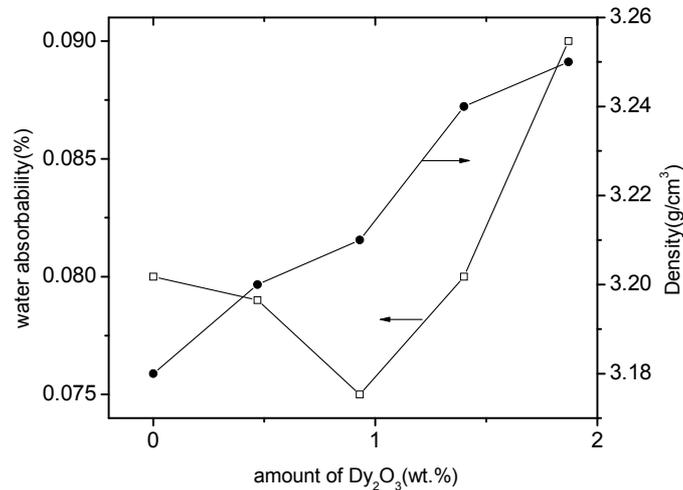


Figure 1. Densification and porosity of AlN ceramics with various amount of  $Dy_2O_3$

### 3.2 Microstructure and Phase Composition

Figure 2 shows SEM photographs of fracture surfaces of sintered samples of D1, D3 and D5. The grain sizes of all three samples are about  $2\mu m$ , which is close to the grain size of the raw materials, so the grain size did not change during sintering. Because liquid phase has a good wettability to AlN grain, sample D3 with 0.93 wt.%  $Dy_2O_3$  was densified fully, in whose image a plate-like second phase covers the AlN grain to form continuous network. In contrast, although sample D1 without  $Dy_2O_3$  also densified under the help of the good wetting second phase, the small amount of second phase is limited and could only improve the contact between AlN grains. Thus, no obviously continuous network was found in sample D1. As far as D5, almost all grains were found to contact with each other. The phases in D5 includes isotropic AlN grains as the main phase and grain boundary phases existing at the triple pockets which indicates that those grain boundary phases poorly wetted the AlN grains.

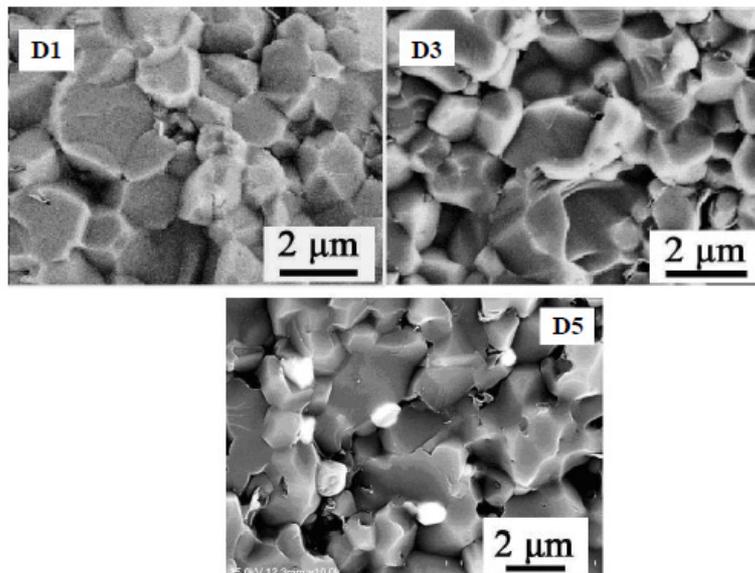


Figure 2. SEM photographs of fracture surfaces of sintered samples

Figure 3 illustrates XRD patterns of the sintered samples D1, D3 and D5. AlN is identified as a main phase for all of samples. It has been suggested (Du & Qin, 2007; Yoshikawa & Katsuda, 2005) that Al<sub>2</sub>O<sub>3</sub>-rich phase, as well as R-β-alumina phase wetted the AlN grain better than Al<sub>2</sub>O<sub>3</sub>. Therefore, Al<sub>2</sub>O<sub>3</sub>-rich phase tended to exist within two-grain boundary to form network structure while AlDyO<sub>3</sub> existed at the triple pockets to form isolated island, as illustrated in Figure 4. The amount of Dy<sub>2</sub>O<sub>3</sub> influenced the morphology of the second phases. When the amount of Dy<sub>2</sub>O<sub>3</sub> was low (such as sample D1), only small amount of the wetting second phase with Al<sub>2</sub>O<sub>3</sub>-rich aluminate dispersed among the AlN grains as discrete two-grain boundary phase (Figure 4a). As the amount of Dy<sub>2</sub>O<sub>3</sub> increased (sample D3), a large amount of wetting second phase (Al<sub>2</sub>O<sub>3</sub>-rich aluminate) formed and diffused through the AlN grains, as a result of which a thin wetting continuous second phase penetrated the ceramics (Figure 4b). Apart from Al<sub>2</sub>O<sub>3</sub>-rich aluminate, there was still small amount of AlDyO<sub>3</sub> which poorly wetted the AlN grains and existed at triple pockets. When the amount of Dy<sub>2</sub>O<sub>3</sub> was high, there was only AlDyO<sub>3</sub> but not Al<sub>2</sub>O<sub>3</sub>-rich aluminate, as the main second phase. What's more, most of these AlDyO<sub>3</sub> grains existed at triple pockets due to its poorly wetting the AlN grain (Figure 4c).

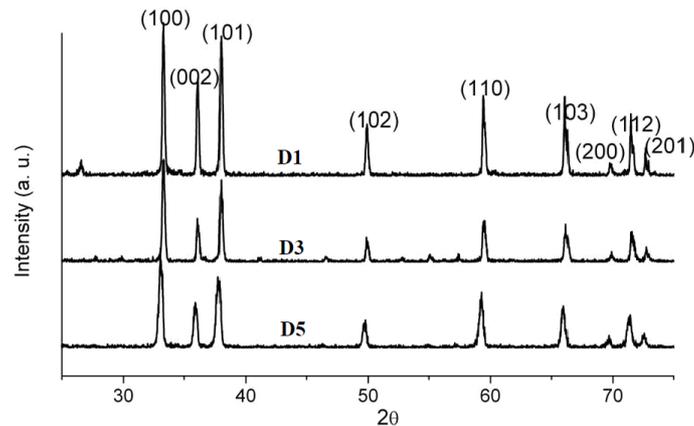


Figure 3. XRD patterns of D1, D3 and D5

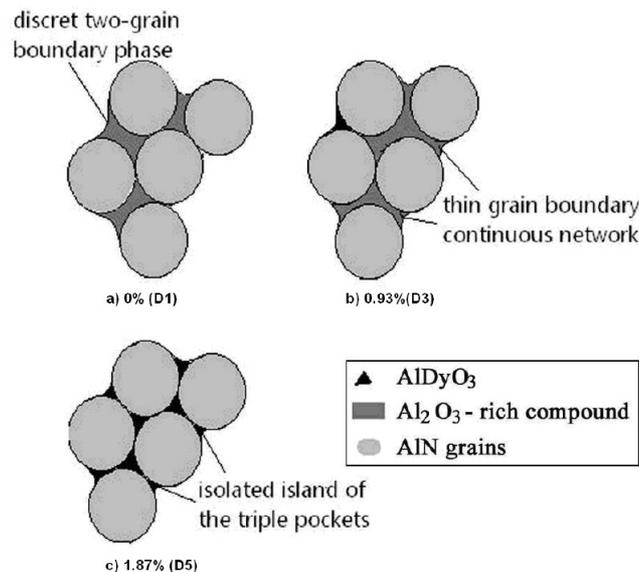


Figure 4. Schematic diagrams of the microstructure of the AlN ceramics with each amount of added Dy<sub>2</sub>O<sub>3</sub>

### 3.3 Dielectric Properties

The dielectric properties of samples D1-D5 is shown in Figure 5. All the samples have almost the same electric conductivity and dielectric loss. But the dielectric constant of the samples is different greatly. Dielectric constant

of D3 is higher than 200, but D5 is only about 15. This illustrates the dielectric constant increased with the increase of  $\text{Dy}_2\text{O}_3$  when the content of  $\text{Dy}_2\text{O}_3$  was low, after adding more  $\text{Dy}_2\text{O}_3$ , the dielectric constant decreased with the increase of  $\text{Dy}_2\text{O}_3$ .

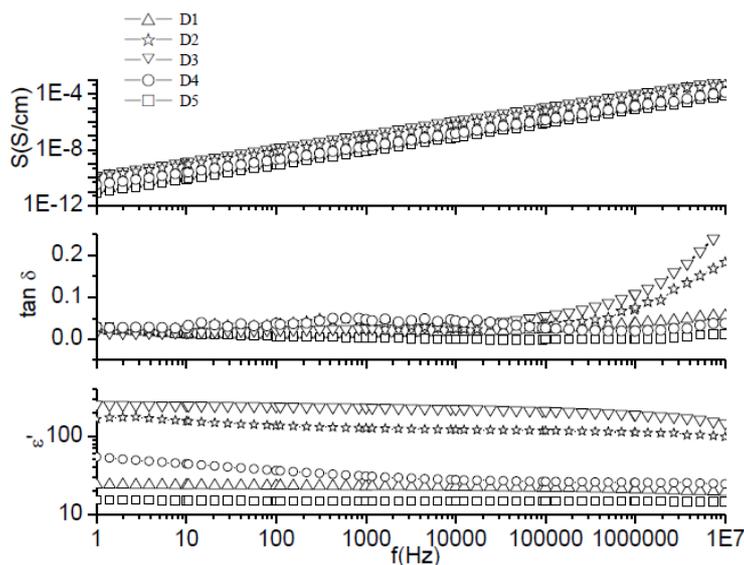


Figure 5. Dielectric constant, dielectric loss and electric conductivity of AlN ceramics D1-D5

It can be explained from the microstructure of the ceramics above. The dielectric constant could be deduced according to Lichtenecker's mixture formulae which related with the dielectric constant and volume of each phase, as well as microstructure in ceramics. Since the structure of  $\text{Al}_2\text{O}_3$ -rich aluminate was much more relaxed than that of AlN grain and  $\text{AlDyO}_3$ , the dielectric constant would be much higher than the latter due to ion relaxation polarization. When the content of  $\text{Dy}_2\text{O}_3$  was low, only limited  $\text{Al}_2\text{O}_3$ -rich aluminate was formed as discrete and relative thick two-grain boundary phase, under which condition both electric conductivity and dielectric constant were low. When the  $\text{Dy}_2\text{O}_3$  was added, an increased amount of  $\text{Al}_2\text{O}_3$ -rich aluminate existed among AlN grains to form thin grain boundary layer to improve the dielectric constant. As a result of increased amount of grain boundary layers, electric conductivity also increased greatly. This kind of ion relaxation polarization was further proved by dielectric loss: the  $\tan \delta$  rapidly increased as the frequency increased in high frequency band. When  $\text{Dy}_2\text{O}_3$  was added to a certain value,  $\text{AlDyO}_3$  formed as the second phase. The  $\text{Al}_2\text{O}_3$ -rich aluminate network among grain boundary disappeared and isolated  $\text{AlDyO}_3$  emerged at the triple pockets. As a result, the dielectric constant was lowered down again.

In order to explore the phase change explicitly, several additional experiments (Table 1) were also conducted by using  $\text{Al}_2\text{O}_3$  reacted with  $\text{Dy}_2\text{O}_3$ -CaO- $\text{Y}_2\text{O}_3$  in the same sintering procedure. The XRD patterns of the sintered samples (DO<sub>1</sub>-DO<sub>3</sub>) are shown in Figure 6. In the  $\text{Al}_2\text{O}_3$ - $\text{Dy}_2\text{O}_3$  system, depending on the amount of  $\text{Dy}_2\text{O}_3$ , there are two kinds of compounds:  $\text{Al}_2\text{O}_3$ -rich (Dy, Y)-aluminate and  $\text{Al}(\text{Dy}, \text{Y})\text{O}_3$ . The chemical formula of (Dy, Y)-aluminate are mainly  $\text{Al}_2\text{O}_3$ -rich phase:  $(\text{Dy}, \text{Y})_3\text{Al}_2[\text{AlO}_4]_3$  and  $\text{DyCaAl}_3\text{O}_7$ . The present results demonstrated that as the content of  $\text{Dy}_2\text{O}_3$  increased, the grain phases changed from  $\text{Al}_2\text{O}_3$ -rich phase, i. e., (Dy, Y)-aluminate (sample DO<sub>1</sub>) to multi-phases, (Dy, Y)-aluminate and  $\text{Al}(\text{Dy}, \text{Y})\text{O}_3$  (sample DO<sub>2</sub>) and finally changed to  $\text{Al}(\text{Dy}, \text{Y})\text{O}_3$  (sample DO<sub>3</sub>). This observation was consistent with literature (Yoshikawa & Katsuda, 2005) in which the ratio of  $\text{AlSmO}_3/\text{Sm-}\beta\text{-alumina}$  (a kind of  $\text{Al}_2\text{O}_3$ -rich phase) as grain boundary phases raised as the increased amount of  $\text{Sm}_2\text{O}_3$ . Some researchers (Yoshikawa & Katsuda, 2005; Bondar & Toropov, 1966; Nakano & Watari, 2003) suggested that  $\text{Al}_2\text{O}_3$ -rich phase was easily formed at the grain boundary.

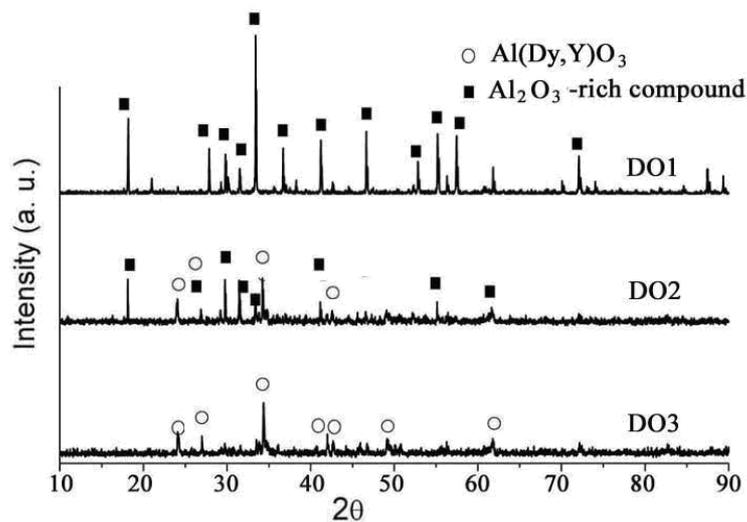


Figure 6. XRD patterns of DO1, DO2 and DO3

During sintering, these  $\text{Al}_2\text{O}_3$ -rich liquid phase would gradually migrated from interior to the surfaces of ceramic which resulted that the liquid phase gradually formed continuous network through the ceramic body. When the amount of  $\text{Dy}_2\text{O}_3$  increased to 0.93%, there was a large amount of liquid  $\text{Al}_2\text{O}_3$ -rich aluminate formed which rapidly migrated through the ceramic. There were also some  $\text{Al}(\text{Dy}, \text{Y})\text{O}_3$  formed at the same time. At the highest amount of  $\text{Dy}_2\text{O}_3$ ,  $\text{AlDyO}_3$  would instead formed primarily.

#### 4. Conclusion

$\text{AlN}$  ceramics with dielectric constant only of 15, close to the theoretical value of pure  $\text{AlN}$  ceramics, was prepared at low-temperature of  $1650^\circ\text{C}$  with the additive of  $\text{Dy}_2\text{O}_3$ . It was found that the amount of  $\text{Dy}_2\text{O}_3$  (0 - 1.87 wt.%) has significant effects on the composition, microstructure and dielectric constant of low temperature sintered  $\text{AlN}$  ceramics. The composition of the second phases varied from  $\text{Al}_2\text{O}_3$ -rich aluminate phase to  $\text{AlDyO}_3$  phase with increasing the amount of  $\text{Dy}_2\text{O}_3$ . The different wettability of the second phases resulted in dissimilarity of both the microstructure and the dielectric properties of these samples. The  $\text{AlN}$  ceramics with 0.93 wt.%  $\text{Dy}_2\text{O}_3$  additive contained both  $\text{Al}_2\text{O}_3$ -rich aluminate phase and  $\text{AlDyO}_3$  phase, whose wetting behavior to the  $\text{AlN}$  grain were good and poor, respectively. The large amount of wetting  $\text{Al}_2\text{O}_3$ -rich aluminate phase contributed to a high dielectric constant value (higher than 200). Sample D5 with 1.87 wt.% of  $\text{Dy}_2\text{O}_3$  contains only  $\text{AlDyO}_3$  as a second phase at the triple pockets, yielding the lowest dielectric constant among five samples.

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