Plasma discharge characteristics of MgO protection layer deposited by using targets made of single crystals, and nano powders

[1]Choong Jin Yang*, [1]Eun Byeong Park, [2]Ho Sang Lee, [3]Jung Hoo Park
[1] Research Institute of Industrial Science & Technology(RIST), P. O. Box 135, Pohang, Korea 790330, [2]Samwha Chemical Industry, [3]Busan National University
054-279-6331, cjyang@rist.re.kr

Abstract

Improved properties of second electron emission was evaluated by assemble of 4 inch PDP panel. MgO protection layers were deposited and examined by using both pellets made of extremely pure MgO single crystals and sintered pellets using MgO nano powders. γ coefficients higher than 0.11 were obtained from the panel by adopting dopant-controlled MgO single crystals. Especially the MgO layer deposited by sintered pellets made of nano powders showed the higher γ coefficients at the operating voltage above 200 volts.

1. Introduction

MgO protection layer deposited using pellets made of various methods were concerned by industrial users in terms of second electron emission, luminance efficiency, and turn off/on voltages. The present study reports the emission characteristics of plasma discharge of PDP panels assembled by innovative MgO protection layers. The deposition targets were made of both the very pure MgO single crystals containing required dopant and sintered MgO pellets made of nano powders. Detailed plasma characteristics were compared with the panels adopting both the single and sintered MgO targets.

2. Experimental

In the present study two different types of MgO targets were employed. First of all, single crystal MgO pellets were grown by electrical melting technique. By using the electrical melting furnace, necessary main dopants and purity were controlled during melting. Basically, the purity of MgO precursor without dopant was controlled up to 99.8 %, and the purity was kept precisely after the melting. Ca, Si, Al, Fe, Cr, Cu, Ag, and Sc were doped during melting.

The second target was sintered MgO pellets. After the MgO single crystals were obtained, these crystals were crushed into desired particle size, and then additional dopants such as Sr, MgF_2, and Ba were mixed. In this step nanoparticles of MgO were also blended up to the desired content, i.e., 5~20 wt.%. The nanoparticles of MgO were synthesized by RF plasma combustion process developed in-house at RIST[1,2]. The size of MgO nano particles used in this study was about 100 nm in average.

3. Results and discussion

3-1. Synthesis of nano MgO powders

Fig. 1 shows the RF plasma facility equipped at RIST. Precursor of dry MgO powders obtained from the recovery of single crystals was used for the synthesis of nano MgO particles. The size of synthesized nanoparticles ranged from 20 to 100 nm., and the shape was cubic which was prominent when the particles become smaller.

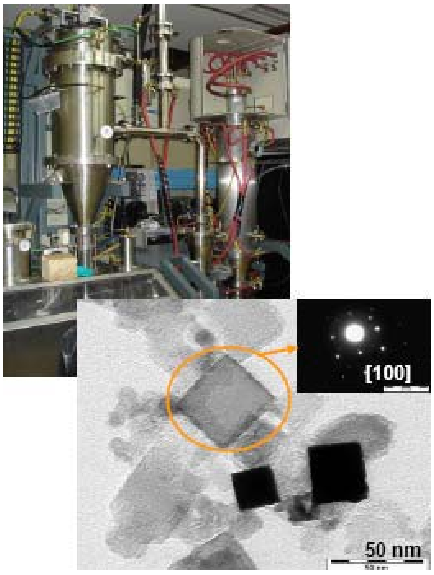

Fig.1. RF plasma process and synthesized nano MgO particles

3-2. Preparation of single crystal MgO targets

Commercial oxide powders of required elements were mixed before melting in the electric furnace. Especially the purity of each element was 99.5 %, and MgO was 99.8% pure. One ton capacity of pilot furnace was used for the melting of materials with current of maximum 1000 Amp. And voltage about 110 volt was applied during 15 hours of melting. Fig. 2 shows the electric melting furnace, single crystals grown in ingot, and MgO pellets cut for the target.

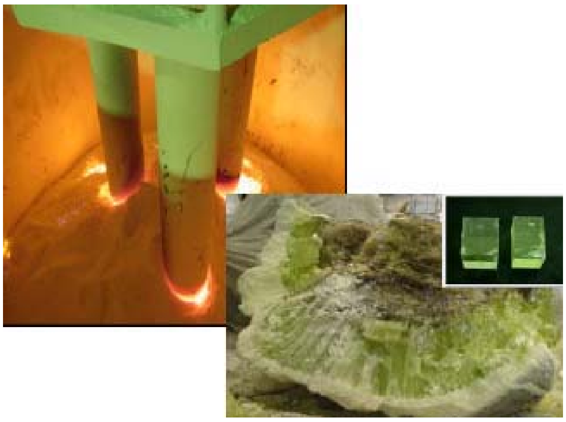

Fig. 2 Electric melting furnace of 1 ton capacity, and MgO single crystals grown after melting. The insert shows pellets after cutting.

Table 1 summarizes compositions of the batch melted. Although more than 10 times of batches were tested, however, typical results are summarized in this table. After the melts, MgO purity was improved due to refining of some elements such as Sc_2O_3, CaO and Fe_2O_3.

Table 1. Summary of compositions used in single crystal growing

Element	Unit (ppm)						
	MgO	CaO	SiO_2	Al_2O_3	Fe_2O_3	B_2O_3	Sc
Batch 2	99.80%	635	47	145	43	34	<100
Batch 1	99.88%	140	30	120	42	20	<100
After melt	99.91%	102	29	230	60	18	<100

3-2. Preparation of sintered muticrystal MgO pellets

As mentioned already, the precursor powders of sintered MgO pellets were the recoveries obtained after crushing the MgO single crystal, which is a unique technique in this study. During blending the compounds, additional doping elements such as BaO, Sc_2O_3, SrO, CeO_2, Li_2O_3, and Eu_2O_3, etc. were added further. More than 80 batches were tried to optimize the characteristics of sintered target. Nano MgO particles were also blended during mixing, if necessary. For the powder blending, ethanol was used as a dispersant, and PVB was used as a sintering agent. Before sintering, two-step axial pressure was applied for a uniform distribution of density. The first pressure was 75 kg/cm^2, and the next was 230 kg/cm^2. Then green pellets were hold at 1600 ℃ for 3~ 4 hours. However, an appropriate temperature increment was needed for several hours to reach the goal temperature. Table 2 summarizes some typical results of the batches tested. The numbers denote the weight of mixing. When the blended elements were precisely analyzed, Ca, Si, Al, and Sc were observed not to change, however, Sr and Ba were found to be changed quite a lot. This means that Sr and Ba were not soluble in MgO lattice due to their large atomic radius.

Table 2. Summary of compositions used for the blending of sintered pellets

Element wt/.%	Batch 35	Batch 44	Batch 45	Batch 53	Batch 75	Ref.
MgO	200	180	200	180	180	?
CaO	-	0.1	-	0.04	400	?
SiO_2	-	0.05	-	-	350	700
B_2O_3	-	0.04	-	0.04		?
Sc_2O_3	0.10	0.15	0.2	0.2		?
CeO_2	0.025	-	17.5	30		?
SrO				0.02		?
Al_2O_3	0.06	0.06	0.06	0.06		?
BaO				0.1		?
Nano	-	-	-	20		-

Nano MgO : CaO 350, SiO_2 20, Al_2O_3 170, Fe_2O_3 120, B_2O_3 10 (ppm)

Table 3 compares the elemental change before and after the sintering. Fe, Cr and Zr were observed which were not added at all. These may be from crucibles used during the experiments.

Table 3 Comparison of composition change before and after the sintering of target

Batch/melting		Composition(ppm)													
		Ca	Si	Al	Fe	Cr	B	Ni	Cu	Li	SrO	BaO	Ce	Sc	Zr
35	before	16	170	10	15	2	<1	<1	1.6	<1	<1	<1	6.1	<1	<1
	after	24	200	12	18	2	<5							<5	45
43	before	370	50	202	14	2	<5							500	
	after	425	42	185	7.0	<5	<5	<5	<5					656	97
44	before	400	43	200	12	2	<5							500	
	after	443	42	181	<5	<5	<5	<5	<5					621	83
45	before	40	53	195	17	2	<1							650	
	after	22	46	187	12	2	<5							800	60
53	before	157	45	13	12	2	<5				120	500		650	
	after	176	26	10	13	<5	13	<5	<5		56	112		651	43

Batch number 53 is the case of sintering which contains nano MgO particles of 20 wt. %. Those nano MgO also includes a little amount of CaO, SiO_2, Al_2O_3, Fe_2O_3 and B_2O_3 which were negligible. By adding nano MgO during compounding the pellets, sintering contraction along the axial direction of the pellet was taking place and found to be 13~26%, which is prominent compared with that of none-nano MgO pellet showing the contraction rate of only 4.5 % along the same direction. Fig. 3 shows the microstructure of pellets for Batch 43 and 53 which contains 20 wt. % nano MgO. The figure indicates a uniform size and shape of MgO grains for the sintered pellets made of nano MgO powders.

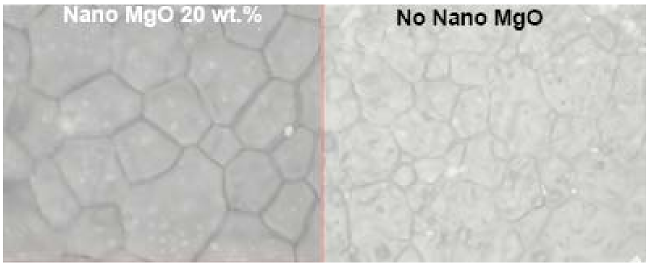

Fig. 3 Fe-SEM microstructures of sintered MgO pellets, (a) with nano, and (b) without nano MgO powders.

3-3. Characteristics of plasma discharge of single crystal and sintered MgO pellets

The evaluation of developed MgO pellets was carried out by assembling of 4 inch PDP panels. Typical plasma discharge characteristics are summarized in Table 4 where the targets of pure single crystal, sintered MgO without using nano MgO, and sintered MgO containing nano MgO are compared

Table 4. Comparison of plasma discharge between single crystal and sintered MgO target

sample	discharge volt.					
	V_{fM}	V_{fm}	V_{sM}	V_{sm}	Marg.	Driving Volt.
Ref.(Jap.)	235	230	190	142	40	210
Single cry #1	240	235	190	144	45	213
Single cry #2	241	238	194	145	44	216
Batch 75		230		142		208
Batch 53 nano MgO	235	231	185	142	45	208
Batch 44	235	233	190	142	43	212

sample	Current (㎃)	Lumi. (cdl/㎡)	Eff. (㏐/w)	Y	Response(㎲)	
					D/T	Jitter
Ref.(Jap.)	3.04	660	2.5	0.1	0.75	0.65
Single cry #1	3.15	668	2.5	0.09	0.8	0.7
Single cry #2	3.12	712	2.48	0.09	0.65	0.6
Batch 75		717	2.6	0.12	0.72	0.5
Batch 53 nano MgO	3.25	641	2.3	0.10	0.7	0.7
Batch 44	3.06	680	2.54	0.10	0.7	1.05

Basically sintered targets show the better property in terms of gamma coefficient and response time(delay time + Jitter width). Especially, the sintered targets containing nano MgO shows the best gamma coefficient and response time, and low driving voltage. However, discharge current tends to increase which result in a low luminance efficiency. At any rate, the targets of sintered MgO developed in this study showed the better properties than the Japanese reference target. Fig. 4 shows a comparison of typical discharge curves indicating delay time and Jitter width.

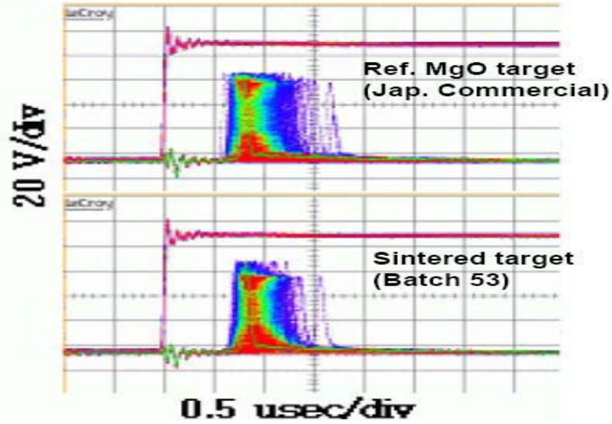

Fig. 4. Plasma discharge curves indicating a better delay time and Jitter width of developed MgO target.

Targets of the Batch 44, which contain 3 times more amount of Ca than single crystal target, show the better luminance efficiency with a lower discharge current. The highest gamma coefficient 0.12 and excellent luminance efficiency obtained from the Batch 75 target also can be understood on the basis of high content of Ca without any other impure elements.

By testing more than 80 batches of trial melting and compounding of sintered MgO pellets, we established elemental effects on plasma discharge characteristics in PDP panels as shown in Table 5.

Table 5. The effects of dopant on plasma discharge characteristics

Dopant	Effects	
	merit	demerit
SC_2O_3	response time ↑ gamma coefficient ↑ discharge current ↓	discharge voltage ↑
SrO	lumi. efficiency ↑ response time ↑ voltage margin ↑ discharge current ↓	gamma coefficient ↓
CeO_2	gamma coefficient ↑ lumi. efficiency ↑ discharge current ↓	response time ↓ discharge voltage ↑
SiO_2	response time ↑ discharge current ↓	lumi. efficiency ↓ discharge voltage ↑
SiO_2 Base CaO	response time ↑ gamma coefficient ↑ lumi. efficiency ↑	discharge current ↑
Fe_2O_3	response time ↑ gamma coefficient ↑ discharge voltage ↓	luminance ↓ (Cd/㎡)
Cr_2O_3	luminance ↑ (Cd/㎡)	response time ↓ gamma coefficient ↓
MgF_2	response time ↑ discharge current ↓	discharge voltage ↑

Particularly we have concentrated on enhancing the secondary electron emission behavior by developing the materials and process at the same time. As indicated in Table 5, the doping of specific element has trade-off effect on the plasma discharge characteristic.

Accordingly, the selection of doping element has to be based on the specific goal of panels which will be a function of panel size and economy. In this sense, we summarize the resultant effect of process and materials for preparation of MgO pellets on secondary electron emission behavior in Fig. 5. Gamma coefficients obtained from plasma discharge of 4 inch PDP panels are shown with respect to applied voltage. As the authors intended, MgO pellets made by returned MgO powder after crushing the single crystal grown by electric melting show a constant gamma coefficient higher than 0.11 as a function applied operating voltage. This process has a special meaning at the point of view that the returned MgO crushed powders show an additional application in term of materials recycling without any loss after preparation of single crystal MgO targets. Another purpose of this study was to find out the merit of nano MgO powders for improving the plasma discharge of protection layer of PDP panels. As shown in Fig. 5, the targets made of nano MgO powders denote a ever-increasing gamma coefficient up to 0.11 as a function increasing applied voltage. This means that when the commercial PDP, which is operated at higher than 230 volt, is taken into account, the MgO targets made of nano powders will be a good choice for improving the gamma coefficient in future.

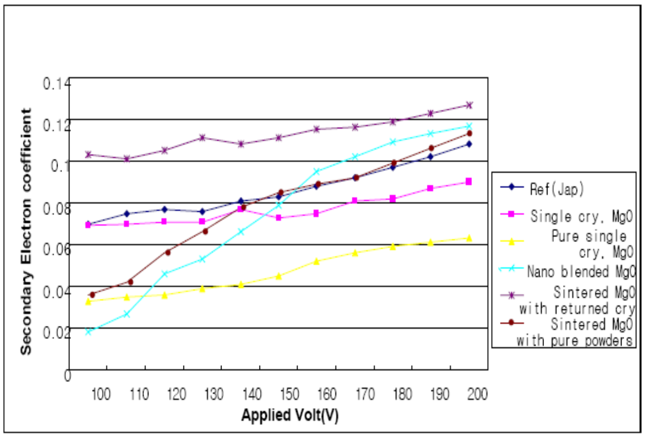

Fig. 5. Secondary electron emission behaviors of MgO protection layer deposited by various target, which were measured as a function of applied voltage.

4. Summary

Excellent plasma discharge characteristics were obtained from MgO targets made by sintered pellets using the recycled MgO powders crushed from single crystals. Both gamma coefficient higher than 0.11 and quick response time shorter than 1.22 μsec were observed without any prominent change as a function of applied voltage. The targets made of nano MgO powders which contains from 10 to 20 wt. % MgO also indicate a good gamma coefficient higher than 0.11 with a quick response time about 1.2~1.4 μsec all the time.

5. References

1. C. J. Yang, J. I. Park, Y. J. Lee, IMID' 2007 Technical Digest, Vol.**II**, p.1646(2007).
2. C. J. Yang, S.D. Choi, E. B. Park and J. I. Park, J. of Korean Ceramic Soc., Vol.**45**(7), p.380(2008).

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