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Effects of Two-Step Deposition and Thermal Treatment on the Frequency Response Characteristics of ZnO SAW Devices

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Abstract - Polycrystalline ZnO thin films are deposited on SiO_2/Si(100) substrate using RF magnetron sputtering. The film deposition performed in this research is composed of the following two procedures; 1^{st}-deposition for 30 min without oxygen at 100 W and 2^{nd}-deposition with oxygen in the range $O_2/(Ar+O_2)$ = 10~50 %. Deposited ZnO films reveal a strongly c-axis preferred-orientation (the corresponding texture coefficient ~ 100 %) as well as a high resistivity (> 10^7 Ωcm). It is also observed that the crystallite size of ZnO is noticeably increased by thermal-annealing. In addition, surface acoustic wave (SAW) devices are also fabricated by using a lift-off method, with the configuration of IDT/ZnO/SiO_2/Si(100). Frequency response characteristics (including S_{21}) of fabricated SAW devices are measured and device parameters including insertion losses and side-lobe rejection level estimated from frequency response are compared.

I. INTRODUCTION

Future wireless communication systems with expanded mobile capabilities are increasing on the rise. With the rapid progress of communication technology, there has been an increasing interest in developing thin-film band-pass filters, including surface acoustic wave (SAW) filters and film bulk acoustic resonators (FBARs) [1][2]. Generally, piezoelectric materials for SAW devices must possess favorable c-axis properties for preferred growth, for a high electromechanical coupling coefficient, as well as high resistivity for low insertion loss and little distortion in the frequency response. Among recently developed piezoelectric thin film materials, Polycrystalline ZnO has been considered as one of the promising materials for such device applications due to their desirable c-axis preferential growth nature as well as technological compatibility with the conventional silicon process. Especially, ZnO film is desirable for high frequency (a few hundreds MHz~GHz) SAW device applications when it is coupled with diamond or sapphire [3][4]. The sputtering method has widely been used to obtain the c-axis oriented ZnO film. Very often the impurities (Li, Cu, etc.) and/or the oxygen have been injected during deposition to increase the resistivity of ZnO [2][5][6]. Also, selection of substrate materials on which ZnO film is grown is very important for achieving a highly oriented and high-quality ZnO film since the differences in lattice parameters and crystalline structures between ZnO and substrate materials may significantly affect the growth behavior of ZnO film [7]. However, it seems that there exists a trade off between the c-axis preferred orientation and the electrical resistivity of ZnO. Also, little has been reported on practical applications of SAW devices, although research on thermal annealing effects of ZnO thin film has been reported for applications of ZnO films, and the studies have not mainly concentrated on their use in surface acoustic wave devices.

In this paper a new deposition technique for improving both the c-axis preferred orientation and the resistivity of ZnO film is proposed. In addition, the effect of thermal-annealing on the ZnO film is also discussed for enhancement of crytallinity. Furthermore, frequency response characteristics (including S_{21}) of fabricated SAW devices on two-step method and thermal annealing effects were measured and device parameters including insertion losses and side-lobe rejection level estimated from frequency response are compared.

II. EXPERIMENTAL

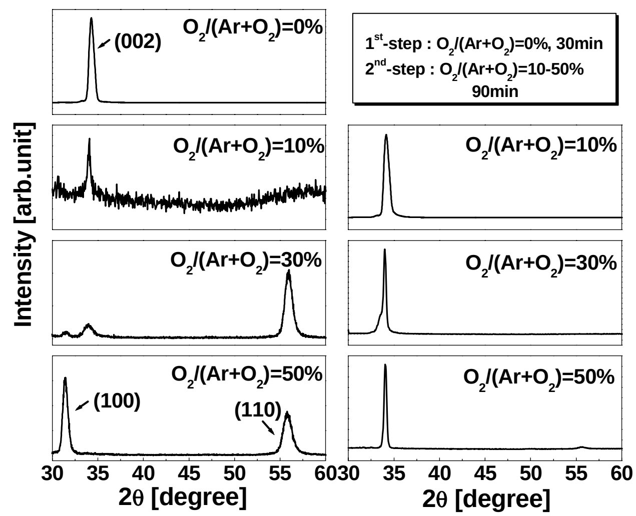
ZnO films were deposited on SiO_2/Si substrate using RF magnetron sputtering. During deposition the substrate was rotated at a low speed of 5 rpm to enhance the thickness uniformity of deposited films. The deposition method proposed in this research consisted of a 1^{st}-step deposition without addition of oxygen and a 2^{nd}-step deposition with addition of oxygen in the range $O_2/(Ar+O_2)$ = 10~50 %. In addition, thermal annealing on deposited ZnO films was performed by varying the temperature from RT to 800 °C. Details of deposition and thermal-annealing conditions are summarized in Table 1.

Table 1 Conditions for two-step deposition and thermal-annealing

	One-step method	Two-step method	
		1^{st}-step	2^{nd}-step
$O_2/(Ar+O_2)$ [%]	10~50	0	10~50
RF power [W]	100	100	100
Pressure [mTorr]	5	5	5
Sub-temp. [^oC]	200	200	200
Depo.-time [min]	120	30	90

Thermal annealing	
Annealing-temperature [^oC]	200, 400, 600, 800
Annealing-time [min]	120
Ambient	Air

Texture coefficient (TC) values for c-axis (002)-orientation and crystallite sizes of ZnO films were evaluated from the XRD (X-ray diffractometer, Bede D3 system) spectra. The I-V characteristics were measured to calculate the resistivity of ZnO, in the voltage range 0 ~ 1 V with a 0.01 V-step by using a pico-ampere meter/DC voltage source (HP 4140B). Raman spectra (Jobin Yvon T64000) were also monitored for all ZnO films to identify the oxygen-deficiency in the film. In addition, inter-digital transducer (IDT) patterns of Al with 2 μm-line width for SAW devices were formed on ZnO films by using a lift-off method. Frequency response characteristics (including S21) of fabricated SAW devices were measured by network analyzer(HP 8720C) and device parameters including insertion loss and side-lobe rejection level were estimated.



(a) (b)

Figure 1. XRD peak patterns of deposited ZnO films; (a) films prepared by one-step deposition and (b) films prepared by two-step deposition.

III. Results and discussion

A. Deposition and effects on thermal annealing of ZnO

Figures 1 (a) and (b) show the XRD peak patterns measured from the ZnO films deposited by using the conventional one-step method and the proposed two-step method, respectively. In the case of using the one-step deposition, the (002)-orientation peak was relatively most intense when the ZnO film was deposited without oxygen. However, as the amount of added oxygen was increased, the (002)-peak intensity was lessened and finally disappeared at $O_2/(Ar+O_2)$ = 50 %. It seemed that oxygen neutrals were embedded in lattice sites or interstitials during the growth of films, which caused a strain and change in the lattice constant and orientation of film [8]. On the other hand, as shown in Figure 1 (b), the ZnO films deposited using the proposed method revealed a highly (002)-oriented growth nature, irrespective of the $O_2/(Ar+O_2)$ ratio used in the 2^{nd}-deposition proceedure. It has generally been considered that the matching in the lattice parameter and crystal structure between the film and the substrate, on which the film is grown, may significantly affect the growth behavior of the film [9]. It should be noted that in the 1^{st}-deposition step of the proposed method, oxygen was not added. The 1^{st}-deposited thin ZnO layer was expected to exhibit a highly (002)-oriented behavior and consequently act as a good substrate for the subsequently-grown 2^{nd}-step ZnO film. This was believed to be one of important reasons why the ZnO films deposited using the two-step method revealed a (002)-preferred orientation with little affected by the oxygen.

Figure 2 shows the crystallite size, resistivity and TC on oxygen ratio from the ZnO films deposited by using the conventional one-step method and the proposed two-step method. The crystallite size estimated from the XRD results was depicted in Figure 2(a), as a function of the $O_2/(Ar+O_2)$ ratio. When the same $O_2/(Ar+O_2)$ ratio was used, the crystallite size of ZnO films deposited by the two-step method was almost twice higher than that of films deposited by the one-step method. This was also attributed due to the reduction of mismatching between the film and the substrate, as previously discussed in the two-step deposition case. In addition, the crystallite size was observed to decrease with increasing

$O_2/(Ar+O_2)$ ratio. Figure 2(b) shows the electrical resistivity measured for the ZnO films deposited by the one-step and two-step methods. In addition, Figure 2(c) shows the TC values for (002)-orientation estimated from the corresponding XRD peak patterns as shown in Figure 1. The film deposited using the one-step method without oxygen exhibited a low resistivity (almost 10^5 Ωcm) which is not suitable for using piezoelectric material, while the TC value was almost 100 %. As the $O_2/(Ar+O_2)$ ratio increased, the resistivity rapidly increased, however the TC value significantly decreased. The similar trend regarding the effect of oxygen on the change of resistivity was also observed for the films deposited using the two-step method. However, it should be noted that the (002) TC value was kept high and not decreased, regardless of the $O_2/(Ar+O_2)$ ratio. At a typical ratio of $O_2/(Ar+O_2)$ = 50 %, the ZnO film deposited using the two-step method showed the resistivity as high as approximately 2.5×10^9 Ωcm and at the same time revealed a high TC value of approximately 100 %.

Figure 3 shows the Raman spectra of ZnO films deposited by one-step and two-step methods. It has been known that the peak at 430 cm^{-1} originates from the E_2 mode of ZnO associated with wurtzite structure and the peak at 570 cm^{-1} is a contribution of E_1(LO) mode of ZnO associated with oxygen deficiency [10][11]. At the $O_2/(Ar+O_2)$ ratio of 10 % (see Figure 3(a)), the peak at 430 cm^{-1} was observed to be dominant for all the films. On the other hand, at the $O_2/(Ar+O_2)$ ratio of 50 % the peak at 430 cm^{-1} weaken for both films, but the peak at 570 cm^{-1} emerged very intense, especially for the film deposited by the two-step method. This indicates that a considerable amount of oxygen-vacancies is present in the ZnO film deposited by the two-step method.

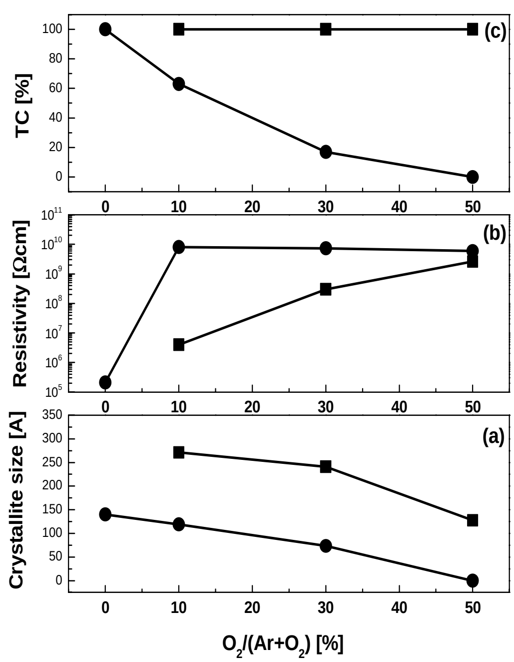


Figure 2. (a) The crystallite size, (b) (002) TC value and (c) resistivity of ZnO films prepared by one-step (●) and two-step (■) method, respectively, as a function of the $O_2/(Ar+O_2)$ ratio.

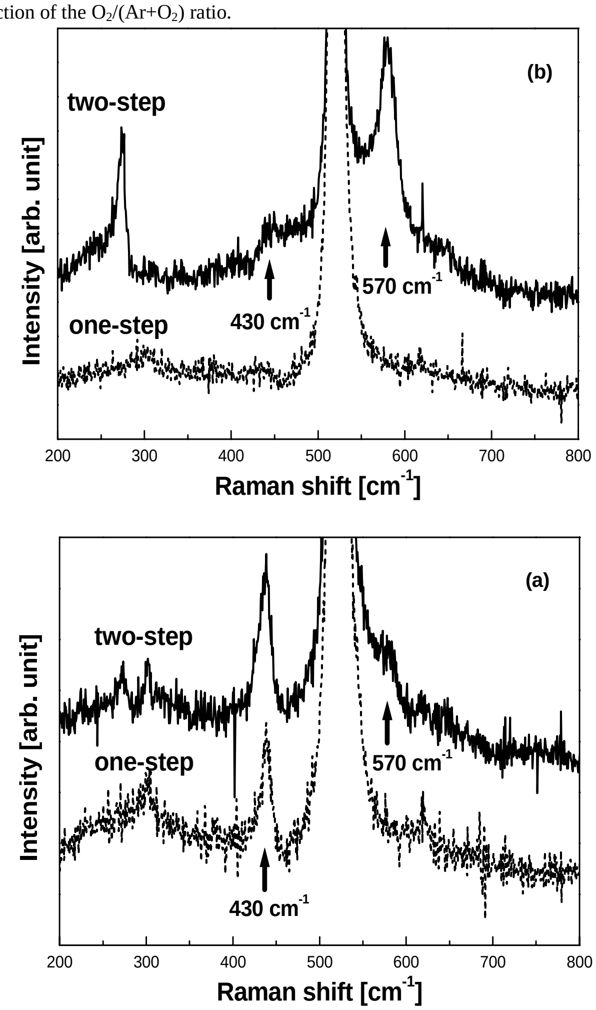


Figure 3. Raman spectra of ZnO films deposited using the one-step and two-step method : (a) $O_2/(Ar+O_2)$ = 10 % and (b) $O_2/(Ar+O_2)$ = 50 %.

Thermal annealing on the deposited ZnO films was performed in air at 200 °C ~ 800 °C for 120 min. The changes of crystallite size and resistivity were measured as a function of annealing temperature. As shown in Figure 4 (a) and (b), by increasing the annealing temperature the crystallite size monotonically increased and the resistivity significantly decreased. This was ascribed due to the enhanced carrier mobility which was resulted from the reduction of grain boundary defects [12][13]. It may also be noted that the change in the crystallite size and resistivity was larger for the film deposited at $O_2/(Ar+O_2)$ = 10 % by using the two-step method, compared with the other films. This was confirmed by analyzing the Raman spectra obtained from those films. Figure 5 indicates that for the two-step deposited films the peak related to the oxygen-vacancy (at 570 cm^{-1}) noticeably decreased as the annealing temperature increased, while it rarely changed for the one-step deposited films. It was previously reported that the oxygen deficiency could be reduced by thermal annealing since some of oxygen atoms in air combined with atomic Zinc in the ZnO film [7].

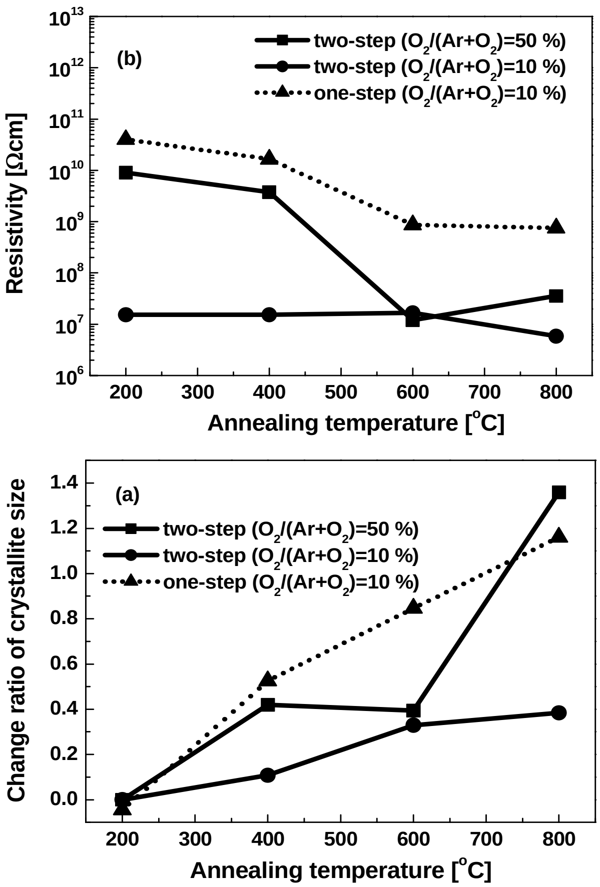


Figure 4. Effects of thermal annealing on the change of (a) crystallite size and (b) resistivity of ZnO films deposited by one-step and two-step methods.

In addition, the (002) TC values of ZnO films were observed to be scarcely changed after thermal annealing. Though thermal annealing at high temperature (600°C and 800°C) enhanced crystallinity of ZnO thin films, the cracks were observed all over the surfaces of the ZnO films. One can conclude that the cracks were attributed to thermal stress for the thermal annealing. Therefore, thermal-treated ZnO thin films at high temperature (600°C and 800°C) can not be used to SAW filter due to the crack in the ZnO thin films surface. In this study, for characterization of effects of SAW devices characteristics on thermal annealing ZnO films SAW devices were fabricated with ZnO thin films treated thermal annealing at 400°C. Also, SAW filters fabricated with ZnO film prepared by the two-step and conventional one-step deposition method are compared.

B. Fabrication and Characterization of SAW devices

SAW devices using ZnO films deposited by the conventional one-step and the proposed two-step are fabricated and their frequency response characteristics such as on the insertion loss and side-lobe rejection level are characterized to examine effects of thermal annealing and two-step deposition

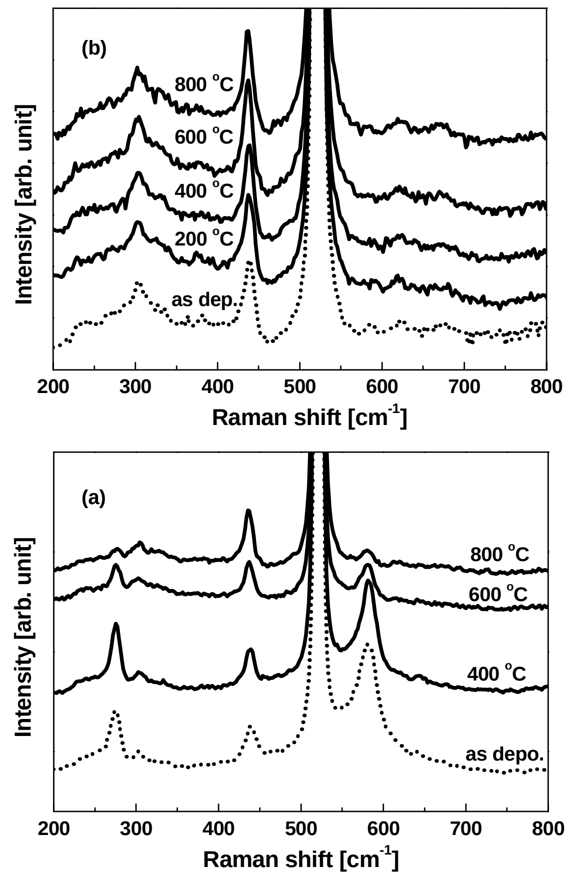


Figure 5. Effects of thermal annealing on the Raman spectra of ZnO films deposited by (a) one-step method and (b) two-step method.

method. As above mentioned, SAW filter are fabricated with using ZnO thin films which were prepared by one-step and two-step deposition method and treated thermal annealing at 400°C.

Figure 6 shows frequency response characteristics on SAW device fabricated by ZnO films deposited by one-step method at $O_2/(Ar+O_2)$ = 10 %. Figure 6(b) shows that by thermal annealing insertion loss and side-lobe rejection level are reduced to about 2~3 dB. However, large differences has not occurred in frequency response.

Figure 7 shows frequency response characteristics on SAW device fabricated by ZnO films deposited by two-step method at $O_2/(Ar+O_2)$ = 10 %. By thermal annealing of ZnO films(see Figure 7(b)), insertion loss and side-lobe rejection level are also reduced to about 3~4 dB as shown in Figure 6. However, by using the two-step deposition insertion loss are remarkably reduced to approximately above 10 dB (Figure 7(a) compared to Figure 6(a) and Figure 7(b) compared to Figure 6(b)). It is notice that improvement of (002) orientation and crystallinity result in reduction of insertion loss on fabricated SAW devices. Also, this is believed since improvement of material properties by the two-step deposition method becomes more conspicuous than that by thermal annealing.

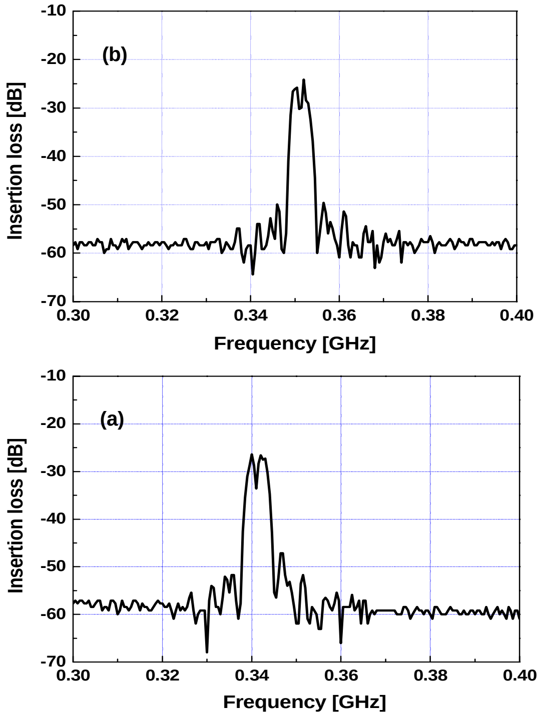


Figure 6. Frequency response on SAW device fabricated with ZnO films deposited by one step method at $O_2/(Ar+O_2)$ = 10 %: (a) as-dep. and (b) thermal annealing.

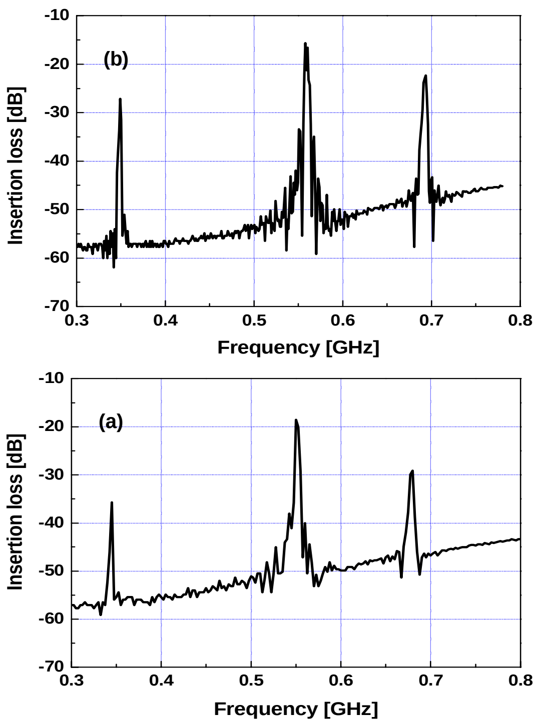


Figure 7. Frequency response on SAW device fabricated with ZnO films deposited by two step method at $O_2/(Ar+O_2)$ = 10 %: (a) as-dep. and (b) thermal annealing.

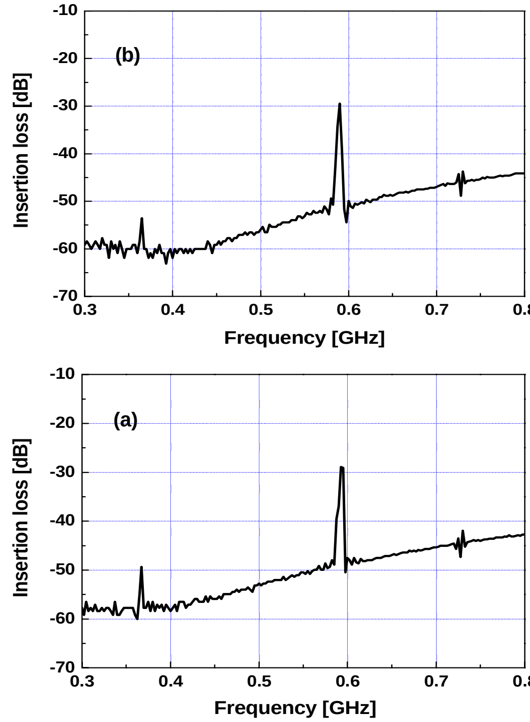


Figure 8. Frequency response on SAW device fabricated with ZnO films deposited by two step method at $O_2/(Ar+O_2)$ = 50 %: (a) as-dep. and (b) thermal annealing.

Figure 8 shows frequency response characteristics on SAW device fabricated with ZnO films deposited by the two-step method at $O_2/(Ar+O_2)$ = 50 %. Though little difference can be noticed in frequency response by thermal annealing of the ZnO thin film, side-lobe rejection level somewhat increases. Characteristic parameters of SAW devices, material parameters of ZnO films, and their deposition conditions are summarized in Table 2. From this table it can be observed that material properties were enhanced by thermal annealing and two-step deposition methods In addition, the table confirms that material properties of ZnO thin films are closely related to SAW devices characteristics. However, it is not clear yet why the center frequency of the SAW device fabricated using the two-step method is observed to be higher than that of the devices using the one-step method. This suggests that more studies are required on this subject.

IV. Conclusions

It was shown from this work that a highly resistive (> 10^9 Ωcm) and (002)-oriented (TC ~ 100 %) polycrystalline ZnO film could be achieved by using the proposed two-step deposition method. XRD peak patterns showed that, compared to the films deposited through the conventional one-step process, the films prepared using the two-step deposition method were grown along the (002) preferred-orientation even at a high $O_2/(Ar+O_2)$ ratio. It was also confirmed from the Raman spectra that the variations of crystallite size and electrical resistivity due to thermal annealing were closely related to the enhancement of crystallinity of ZnO films. In addition, the SAW devices fabricated using ZnO thin films deposited by two-step method show lower insertion loss than that of conventional one-step deposition method, and SAW devices fabricated using thermal annealing ZnO thin films also enhanced more than that of as-deposited ZnO thin films.

Table 2 Deposition condition, material properties of ZnO, and device characteristic parameter of fabricated SAW filter

Deposition conditions			SAW Device Parameters		Material Properties	
	$O_2/(Ar+O_2)$	Annealing	IL [dB]	SLR [dB]	D [ ]	TC [%]
One-step	10	×	26.4	32.4	118.2	63.2
	10	○	24.5	33.7	183.5	63.2
Two-step	10	×	18.7	32.7	283.7	100
	50	×	29.1	20.1	134.6	100
	10	○	15.8	38.0	299.2	100
	50	○	29.3	22.8	182.7	100

IL (insertion loss) / SLR (side-lobe rejection) / D (crystallite size) / TC (texture coefficient)

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