

# Deposition of Sputtered NiO as a p-type Layer for Heterojunction Diodes with $\text{Ga}_2\text{O}_3$

Running title: Deposition of Sputtered NiO

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The characteristics of sputtered NiO for use in pn heterojunctions with  $\text{Ga}_2\text{O}_3$  were investigated as a function of sputtering parameters and post-deposition annealing temperature. The Oxygen/ Nickel and  $\text{Ni}_2\text{O}_3/\text{NiO}$  ratios, as well as the bandgap and resistivity, increased as a function of  $\text{O}_2/\text{Ar}$  gas flow ratio. For example, bandgap increased from 3.7 to 3.9 eV and the resistivity increased from 0.1 to 2.9  $\Omega\text{.cm}$  for  $\text{O}_2/\text{Ar}$  ratio increasing from 1/30 to 1/3. By sharp contrast, the bandgap and  $\text{Ni}_2\text{O}_3/\text{NiO}$  ratio decreased monotonically with post-deposition annealing temperatures up to 600°C, but the density of the films increased due to a higher fraction of NiO being present. Hydrogen is readily incorporated into the NiO during exposure to plasmas, as delineated by Secondary Ion Mass Spectrometry measurements on deuterated films. The band alignments of the NiO films were type II- staggered gap with both  $\alpha$ - and  $\beta$ - $\text{Ga}_2\text{O}_3$ . The breakdown voltage of NiO/ $\beta$ - $\text{Ga}_2\text{O}_3$  heterojunction rectifiers was also a strong function of  $\text{O}_2/\text{Ar}$  flow ratio during deposition, with values of 1350 V for 1/3 and 830V for 1/30.

## I. INTRODUCTION

NiO is a well-established p-type oxide <sup>(1)</sup> which has found renewed application as a component of p-n heterojunctions with  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> in high power rectifiers <sup>(2-17)</sup>. This provides a solution to the lack of a robust p-type doping capability in Ga<sub>2</sub>O<sub>3</sub> <sup>(3)</sup>, allowing the demonstration of high breakdown voltages and forward conduction currents with NiO/Ga<sub>2</sub>O<sub>3</sub> heterojunction rectifiers <sup>(2-17)</sup>. The NiO may also be used as edge termination or p-type guard rings in such devices <sup>(10)</sup>, but clearly the design parameters have not been optimized in terms of thickness, resistivity, stoichiometry and other parameters for the NiO used in these applications <sup>(4-17)</sup>.

There have been many studies of the properties of NiO deposited by a number of methods <sup>(18-32)</sup>, including e- beam evaporation <sup>(23)</sup>, reactive sputtering <sup>(24)</sup> or atomic layer deposition <sup>(26)</sup>, but few specifically related to the thin layers needed in NiO/Ga<sub>2</sub>O<sub>3</sub> devices, which must also typically undergo annealing treatments during device processing. It is well-established that the NiO bandgap varies from ~3.6-4 eV, depending on deposition conditions and that the p-type conductivity is a strong function of the O<sub>2</sub> partial pressure during deposition <sup>(18-32)</sup>.

In this work, we systematically vary the O<sub>2</sub>/ Ar ratio during multi-source NiO target magnetron sputtering and also perform post-deposition annealing up to 600°C. The composition, bandgap, density and surface roughness of the NiO films were measured for all these conditions and the effect of O<sub>2</sub>/Ar gas flow ratio on the breakdown voltage of NiO/ $\beta$ -Ga<sub>2</sub>O<sub>3</sub> heterojunction rectifiers was quantified. We also investigated the ease of incorporation of hydrogen during simulated plasma enhanced chemical vapor deposition processes. This work provides a framework for optimizing the incorporation of NiO into Ga<sub>2</sub>O<sub>3</sub> power devices.

## II. EXPERIMENTAL

The deposition was performed in a Kurt J. Lesker Multi-Source Sputter System using dual NiO targets. The 13.56 MHz rf power during deposition was 150W at a pressure of 3mTorr, producing a deposition rate of 12 nm/min (2 Å/sec). The gas flow ratios of O<sub>2</sub>/Ar were 1/3, 1/7, 1/10 or 1/30. The deposition temperature was nominally room temperature and was < 100°C. All of the NiO films were polycrystalline, as determined by x-ray diffraction. The NiO was deposited on to quartz slides for calibration of sputter rate, resistivity, composition, density and morphology and also on to β-Ga<sub>2</sub>O<sub>3</sub> structures consisting of 10 μm epi layers with doping 3x10<sup>16</sup> cm<sup>-3</sup> deposited by Hydride Vapor Phase Epitaxy onto a (001) oriented n<sup>+</sup> Sn-doped Ga<sub>2</sub>O<sub>3</sub> substrates for fabrication of heterojunction rectifiers whose processing sequence has been described in detail elsewhere<sup>(10)</sup>. The current-voltage (I-V) characteristics of these devices were recorded with a Tektronix 370-A curve tracer, 371-B curve tracer and Agilent 4156C parameter analyzer. The reverse breakdown voltage was defined as the bias for a reverse current reaching 0.1 A.cm<sup>2</sup>. Hall measurements were used to obtain carrier mobilities. In a few cases, we also deposited the NiO on to 1 μm thick α-polytype Ga<sub>2</sub>O<sub>3</sub> layers grown on sapphire substrates in order to compare band alignment of NiO on both α- and β-polytype samples before and after annealing up to 600°C under O<sub>2</sub> ambient. Finally, some of the NiO films were exposed to <sup>2</sup>H plasmas at 200°C for 30 min and then subsequently annealed at 300°C to simulate typical PECVD processes and contact annealing steps. The deuterium profiles in the samples were measured by Secondary Ion Mass Spectrometry (SIMS).

The compositional analysis was done by measuring the ratio of ratio of O/Ni transitions in X-Ray Photoelectron Spectroscopy (XPS) spectra. For the Ni<sub>2</sub>O<sub>3</sub>/ NiO phases, we were not able to separate the peaks of Ni<sup>2+</sup> and Ni<sup>3+</sup> because they are too close in energy. The XPS system was a Physical Instruments ULVAC PHI, with an Al x-ray source (energy 1486.6 eV, source power 300W), analysis size of 100  $\mu$ m diameter, a take-off angle of 50° and acceptance angle of  $\pm 7$  degrees. The electron pass energy was 23.5 eV for high-resolution scans. The bandgap was extracted from Tauc plots of absorbance obtained using UV-Vis (Perkin-Elmer Lambda 800 UV/Vis) spectrometer). The film density and surface roughness were obtained from X-Ray Reflectometry (XRR) measurements using an XRR measurements have been performed on the PANalytical Empyrean  $\omega/2\Theta$  diffractometer system. The acquired spectra were modeled with the freeware GenX (GenX) software package. Features of the XRR spectrum such as the critical angle, periodicity of oscillation and their amplitude are directly related to the density, thickness and roughness of the actual structure. The ratio of NiO/Ni<sub>2</sub>O<sub>3</sub> as obtained from X-Ray Diffraction (XRD) measurements. The XRD analysis was performed using a Brucker D8 Advance diffractometer. The crystalline structure of the NiO thin films was established by applying the standard XRD technique using Cu K $\alpha$  radiation ( $\lambda = 1.55418 \text{ \AA}$ ) in the range of  $2\theta = 25\text{--}80$  degrees. Band alignments were also determined by XPS.

### III. RESULTS AND DISCUSSION

Figure 1 shows the XPS spectrum of a typical NiO film. The different valence transitions for Ni present show the film is a mixture of NiO and Ni<sub>2</sub>O<sub>3</sub>. There are two different components of the oxygen peak in the composition calculation, namely 529.3eV

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as the main oxygen peak and 531eV which is associated with oxygen next to a Ni vacancy, which can be used to calculate the oxygen concentration in the material. There are also multiple components within the main Ni peak <sup>(34)</sup>.

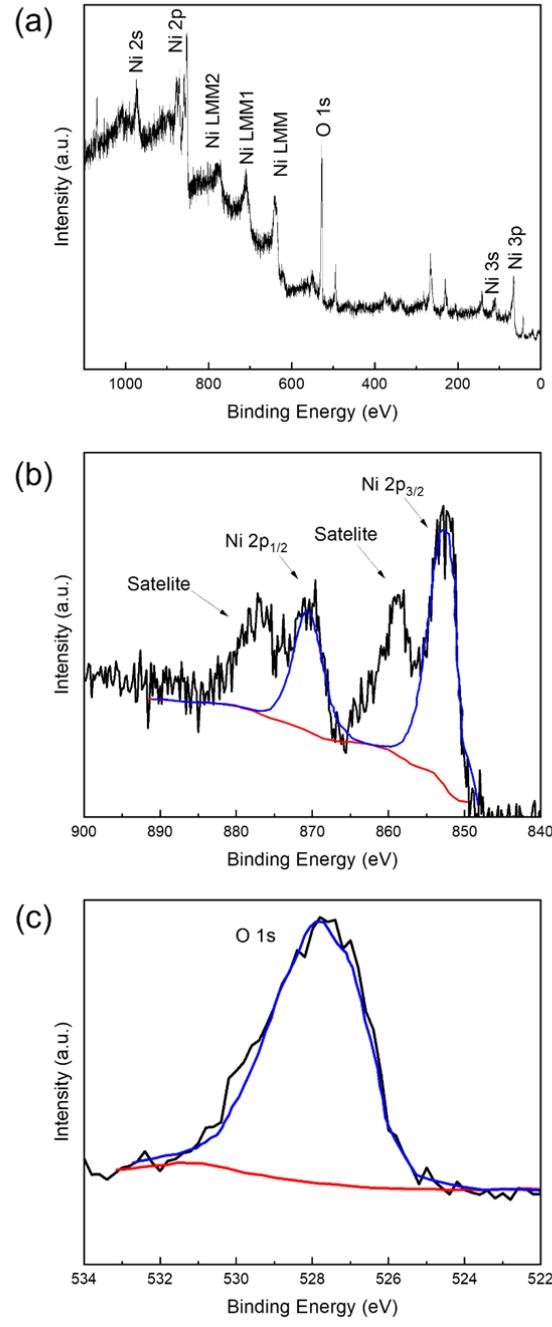


Fig. 1.(a) XPS survey spectrum of NiO film (b) expanded view of Ni 2p peaks region (c) expanded view of O 1s peak region

Figure 2 (a) shows the oxygen/ nickel ratio and  $\text{Ni}_2\text{O}_3/\text{NiO}$  as a function of  $\text{O}_2/\text{Ar}$  gas flow ratio. NiO films deposited at a higher ratio of oxygen gas flow have higher oxygen content related to the higher proportion of  $\text{Ni}_2\text{O}_3$ . Figure 2 (b) shows the energy bandgap and resistivity of NiO films as a function of oxygen/argon flow ratio. The stoichiometry of NiO is dependent upon the deposition parameters. The bandgap was obtained from the Tauc plot, where we obtained better fitting to the absorbance data assuming a square power dependence in the plot of  $(\alpha h\nu)^2$  as a function of the photon energy  $h\nu$ . This suggests the bandgap is direct<sup>(1)</sup>. In these relations,  $\alpha$  and  $h\nu$  are the absorption coefficient and photon energy, respectively. With a higher ratio of oxygen gas flow, the  $E_g$  and the resistivity both increase. The bandgap trend is in general agreement with past reports where the films were deposited by sputtering at elevated temperature (200 °C)<sup>(22)</sup>, but the resistivity in that case strongly decreased with increasing oxygen content due to an increase in carrier concentration<sup>(22)</sup>. The increase in hole concentration was ascribed to nickel vacancies and oxygen interstitials. However, in our films deposited near room temperature, the increasing oxygen content leads to a relatively small increase in resistivity, with no increase in oxygen interstitials and nickel vacancies. The hole concentration was  $2 \times 10^{18} \text{ cm}^{-3}$  for 1/3  $\text{O}_2/\text{Ar}$  gas ratio and  $2 \times 10^{19} \text{ cm}^{-3}$  for 1/30 gas ratio, with a mobility of  $<1 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$  in all cases. Table 1 summarizes the changes in bandgap and film composition as a function of  $\text{O}_2/\text{Ar}$  ratio during sputtering.

The stability of NiO film properties during post-deposition annealing was measured, since this is relevant to device processing sequences where Ohmic contacts have to be

annealed and dielectric films might be deposited for a variety of purposes, including surface passivation or encapsulation. Figure 3 shows the energy bandgap and  $\text{Ni}_2\text{O}_3/\text{NiO}$  ratio of NiO films as a function of post-deposition annealing temperatures. With increasing annealing temperature, the  $E_g$  decreases and more oxygen escapes from the film, corresponding to a decrease in the  $\text{Ni}_2\text{O}_3/\text{NiO}$  ratio.

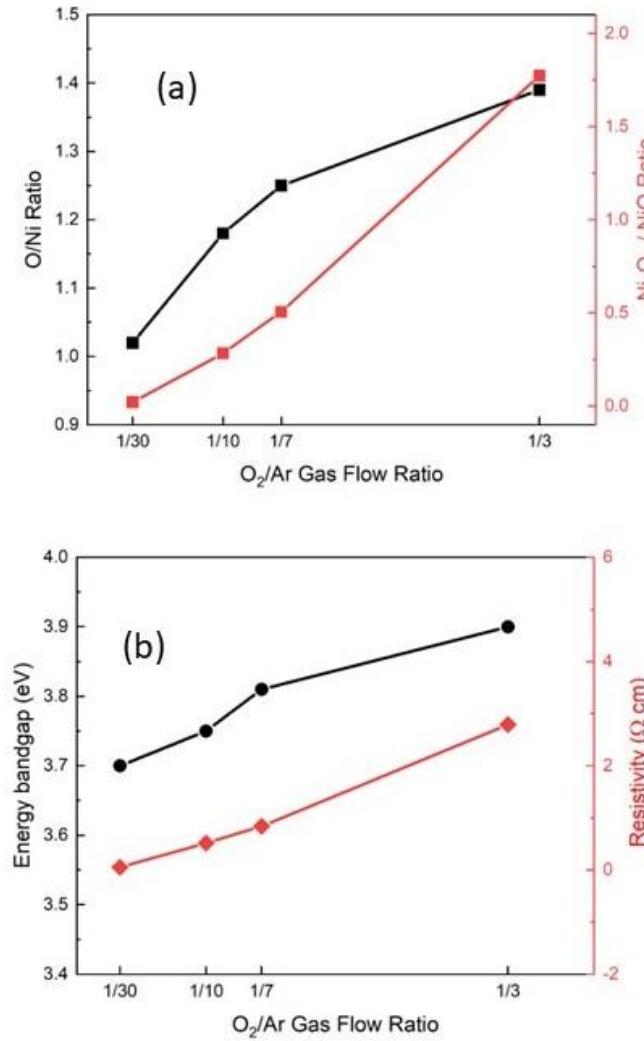


Fig 2. (a) Oxygen/ nickel ratio and  $\text{Ni}_2\text{O}_3/\text{NiO}$  ratio and (b) energy bandgap and resistivity as a function of  $\text{O}_2/\text{Ar}$  gas flow ratio.

**Table 1.** Composition and bandgap data for films as a function of O<sub>2</sub>/Ar flow rate during sputtering.

O <sub>2</sub> /Ar flow rate ratio	1/3	1/7	1/10	1/30
O (atomic %)	58.2	55.6	54.1	50.5
Ni (atomic %)	41.8	44.4	45.9	49.5
O/Ni	1.39	1.25	1.18	1.02
Ni <sub>2</sub> O <sub>3</sub> /NiO	1.77	0.51	0.28	0.02
E <sub>g</sub> (eV)	3.90	3.81	3.75	3.70

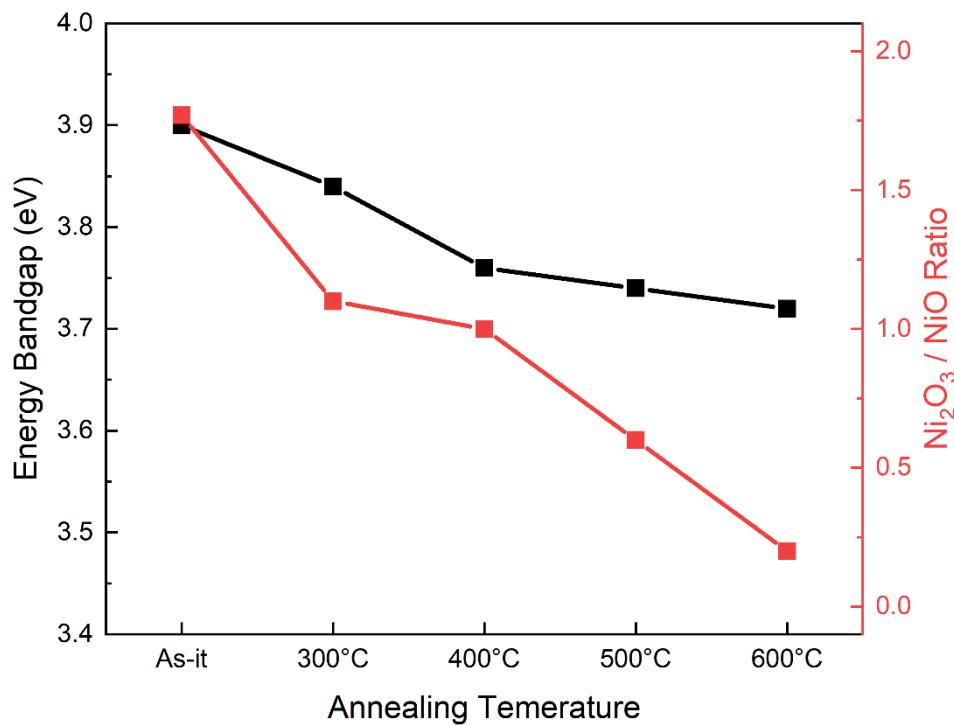


Fig. 3. Energy bandgap and Ni<sub>2</sub>O<sub>3</sub>/NiO ratio in NiO films as a function of annealing temperatures.

Another issue of interest during such simulations for process steps is the diffusion of hydrogen, which might occur during annealing in forming gas or during plasma enhanced chemical vapor deposition of dielectrics. In general, hydrogen is an unavoidable impurity in oxide semiconductor films deposited by sputtering, due to incorporation through reactions with residual gas species,  $\text{H}_2\text{O}$  and  $\text{H}_2$ , within the vacuum chamber<sup>(35)</sup>.  $\text{NiO}$  samples grown with 1/3  $\text{O}_2/\text{Ar}$  gas ratio were treated in  $\text{D}_2$ -plasmas for 30 min at a nominal temperature of 200 °C and were characterized with Secondary Ion Mass Spectrometry (SIMS) at EAG Laboratories before and after subsequent annealing at 300°C. Detection limits for SIMS for D were  $3 \times 10^{15} \text{ cm}^{-3}$ . Figure 4 (a) shows SIMS profiles of the  $^2\text{H}$  in the film after plasma exposure, with a very high concentration of deuterium incorporated. Subsequent annealing lowered this by approximately a factor of 5, as shown in Figure 4(b), but a high concentration remains ( $\sim 10^{20} \text{ cm}^{-3}$ ). Plots of the deuterium profiles before and after annealing are shown in Figure 4 (c). Since hydrogen is an important n-type dopant in a variety of conducting oxides<sup>(36, 37)</sup>, it could have substantial influence on the conductivity of the  $\text{NiO}$  and also it is known to influence the surface properties of  $\text{Ga}_2\text{O}_3$ <sup>(38, 39)</sup>. The resistivity of the sample changed from  $2.9 \Omega \cdot \text{cm}$  to  $1.6 \Omega \cdot \text{cm}$  after the plasm treatment.

In a similar vein, the band offset of  $\text{NiO}$  on  $\text{Ga}_2\text{O}_3$  is of interest to determine its ability as a hole injector. We have reported<sup>(17, 40)</sup> that this band alignment is type II staggered gap for  $\text{NiO}$  on both  $\beta\text{-Ga}_2\text{O}_3$ <sup>(17)</sup> and  $\text{GaN}$ <sup>(40)</sup>. The band alignments were determined from the standard X-Ray Photoelectron Spectroscopy (XPS) method<sup>(41-43)</sup>. We also measured this for  $\text{NiO}$  on the  $\alpha$ -polytype of  $\text{Ga}_2\text{O}_3$  to assess the generality of this trend in band alignment. Figure 5 shows band alignments for (a)  $\text{NiO}/\beta\text{-Ga}_2\text{O}_3$  and (b)

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NiO/ $\alpha$ -Ga<sub>2</sub>O<sub>3</sub> both as-deposited and different annealing temperatures and the same trends are seen for both polytypes. The band alignment remains as staggered gap across the entire annealing range.

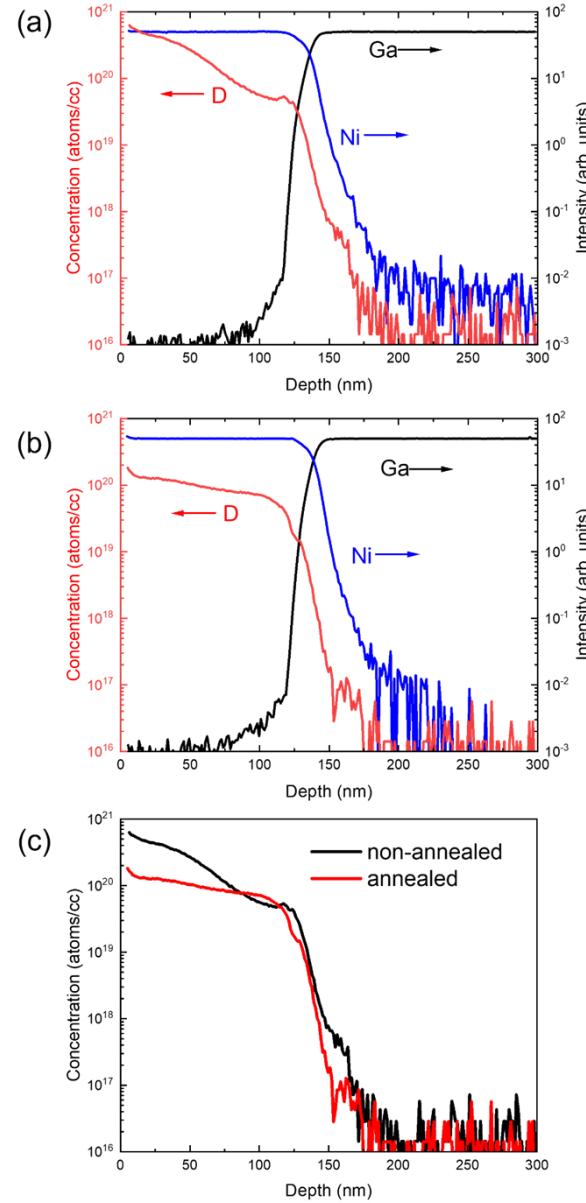


Fig. 4. SIMS profiles of deuterium in <sup>2</sup>H plasma exposed NiO at 200°C (a) as-exposed and (b) after subsequent annealing at 300°C.(c) shows a detailed comparison of the deuterium profiles before and after annealing.

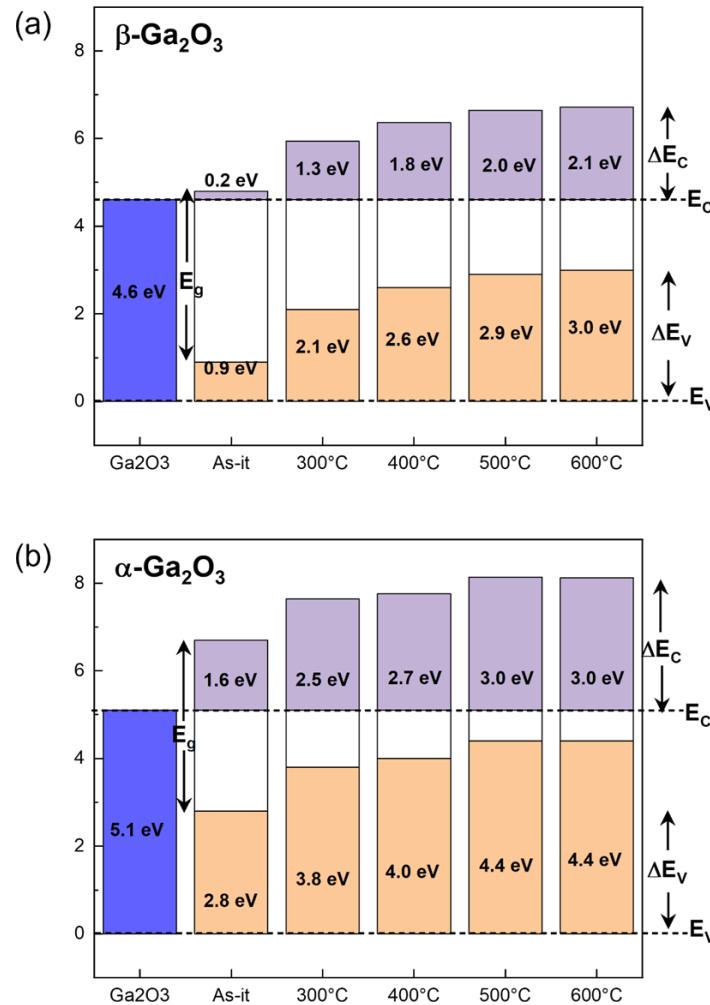


Fig 5. Band alignments for (a) NiO/  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> and (b) NiO/  $\alpha$ -Ga<sub>2</sub>O<sub>3</sub> for as-deposited and different annealing temperatures.

Figure 6 (a) shows that with the increase of annealing temperature, the density of the films obtained from XRR increases due to more NiO being formed relative to Ni<sub>2</sub>O<sub>3</sub>. The density of the former is 6.67 g/cm<sup>3</sup>, while the density of Ni<sub>2</sub>O<sub>3</sub> is 4.84 g/cm<sup>3</sup>. The roughness of the films also increases with increasing annealing temperatures. As shown in the XRD spectra of Figure 6 (b), the Ni<sub>2</sub>O<sub>3</sub> peak decreases with higher annealing

temperature so that the amount of  $\text{Ni}_2\text{O}_3$  decreases with higher annealing temperature, consistent with the decrease of film density. Table 2 summarizes the annealing temperature induced changes in bandgap, resistivity,  $\text{Ni}_2\text{O}_3/\text{NiO}$  ratio, density, surface roughness and band offsets. The increase of resistivity with annealing temperature is speculated may be due to an increased concentration of oxygen vacancies.

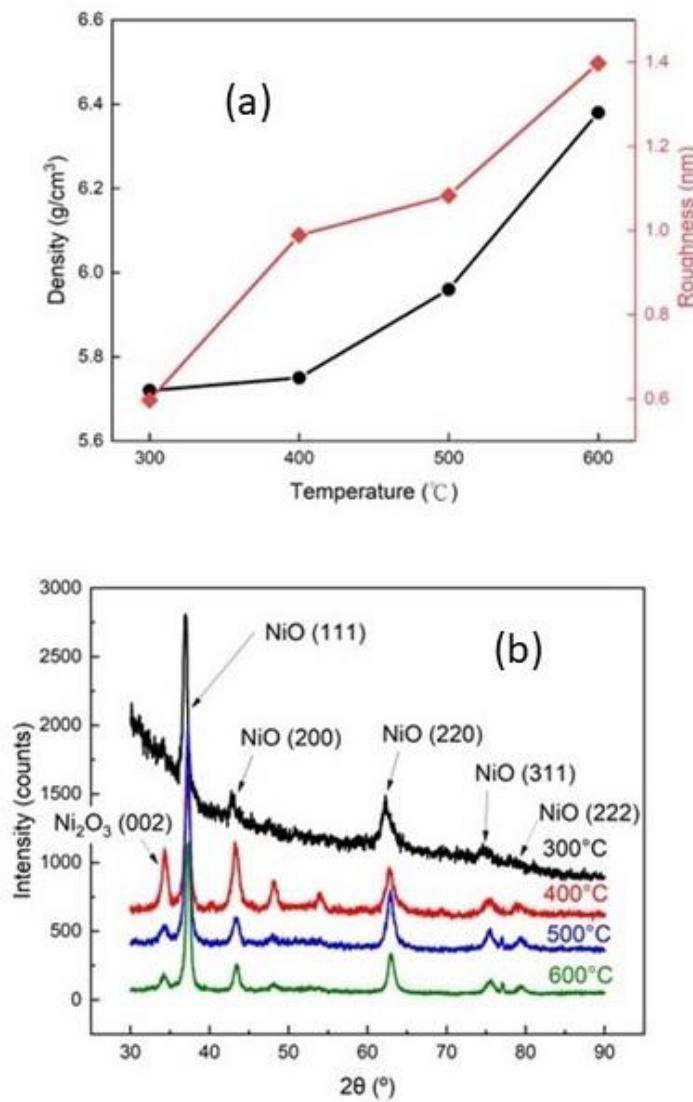


Fig. 6. (a) Film density and surface roughness and (b) XRD spectra as a function of different annealing temperatures.

**Table 2.** Composition, resistivity, band offsets, density and RMS roughness data for films as a function of post deposition annealing temperature.

Temperature (°C)	As-Dep	300	400	500	600
Ni <sub>2</sub> O <sub>3</sub> /NiO	1.77	1.08	1.01	0.63	0.19
Resistivity (Ω·cm)	2.79	5.6	> 10	> 10	> 10
E <sub>g</sub> (eV)	3.90	3.84	3.76	3.74	3.72
ΔE <sub>c</sub> (eV) (β-Ga <sub>2</sub> O <sub>3</sub> )	0.2	1.34	1.76	2.04	2.12
ΔE <sub>v</sub> (eV) (β-Ga <sub>2</sub> O <sub>3</sub> )	0.9	2.1	2.6	2.9	3
ΔE <sub>c</sub> (eV) (α-Ga <sub>2</sub> O <sub>3</sub> )	1.6	2.54	2.66	3.04	3.02
ΔE <sub>v</sub> (eV) (α-Ga <sub>2</sub> O <sub>3</sub> )	2.8	3.8	4	4.4	4.4
Density (g/cm <sup>3</sup> )	5.50	5.72	5.75	5.96	6.38
Roughness (nm)	-	0.597	1.083	0.989	1.397

To determine the effect of sputtering conditions on the performance of NiO/Ga<sub>2</sub>O<sub>3</sub> rectifiers, structures with a fixed NiO thickness of 240nm were fabricated, as shown in the schematic of Figure 7(a). With a higher ratio of oxygen gas flow, on-state resistance R<sub>on</sub> and V<sub>bi</sub> of the diode increased when O<sub>2</sub>/Ar ratio was decreased, as shown in Figure 7(b). Concurrently, the turn on voltage V<sub>on</sub> also decreased, as shown in Figure 7 (c). The ideality factor of the rectifiers was 2.7, indicating more than simply thermionic emission present. There was no systematic difference in barrier heights determined by I-V and C-V, indicating a low degree of interface inhomogeneity <sup>(44)</sup>. The V<sub>on</sub> is calculated from the slope of the forward I-V curves. This is due to the lower resistivity of the film under these conditions. Correspondingly, the on/off ratio and the breakdown voltage V<sub>B</sub> increased because of lower conductivity at high O<sub>2</sub>/Ar ratio, as shown in Figure 8(a) and (b), respectively. This shows the strong influence the deposition parameters of the NiO have

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on the device performance of NiO/Ga<sub>2</sub>O<sub>3</sub> rectifiers. The breakdown doesn't occur catastrophically, as defined by breakdown criteria of current reaching 0.1 mA/cm<sup>2</sup>. For 200  $\mu$ m diameter rectifiers, the reverse recovery time of  $\sim$ 21 ns was independent of temperature, with the  $I_{rr}$  monotonically increasing from 15.1 mA at 25°C to 25.6 mA at 250°C <sup>(45)</sup>.

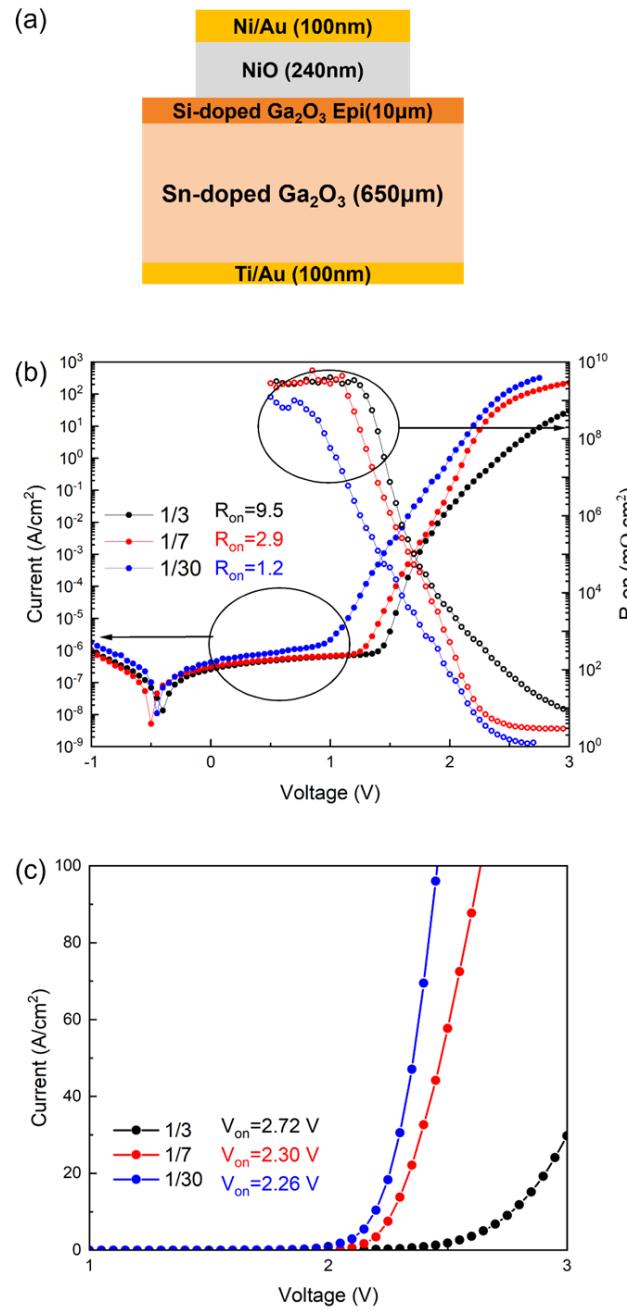


Fig 7. (a) Schematic of device structure (b) Forward current density and on-state resistance and (c) Forward turn-on voltage as a function of O<sub>2</sub>/Ar gas flow ratio during deposition

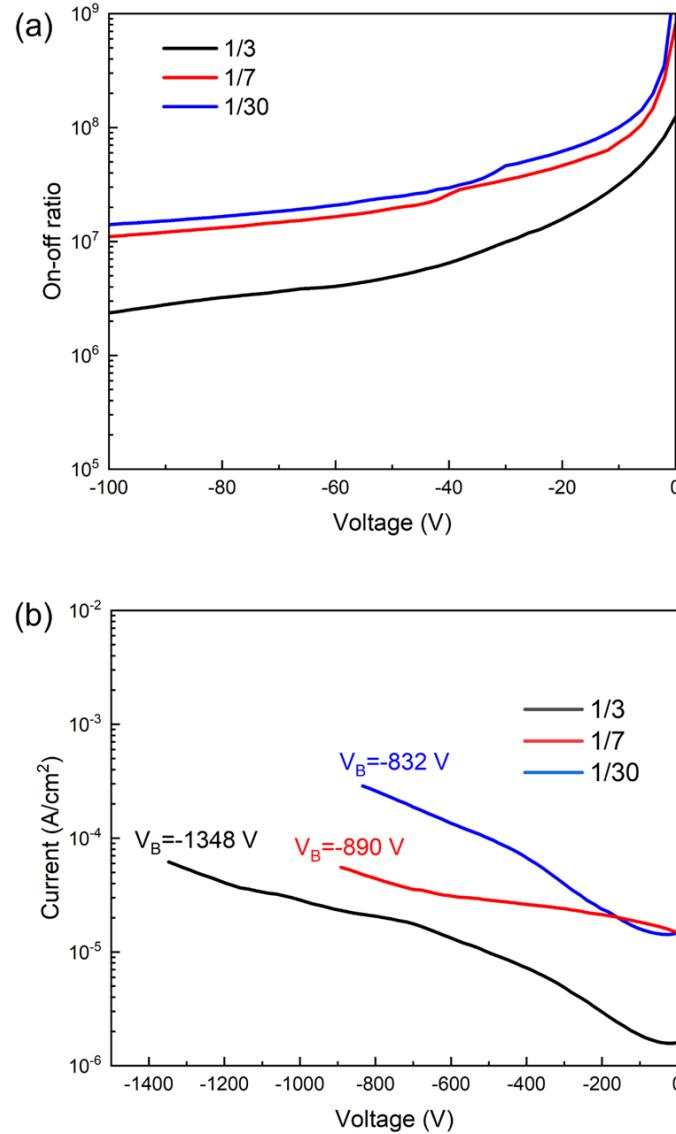


Fig. 8. (a) On-off ratio (b) Reverse I-V characteristics and associated breakdown voltages as a function of O<sub>2</sub>/Ar gas flow ratio during deposition.

## IV. SUMMARY AND CONCLUSIONS

The properties of thin NiO films deposited by magnetron sputtering and intended for use in heterojunction devices with  $\text{Ga}_2\text{O}_3$  were examined as a function of deposition parameters and post-deposition annealing temperature to understand the effect of these parameters on device performance of NiO/ $\beta$ - $\text{Ga}_2\text{O}_3$  rectifiers. The bandgap, resistivity, density and composition are all affected by  $\text{O}_2/\text{Ar}$  ratio during deposition. Post-deposition annealing up to 600°C also changes the film properties in a controlled fashion. High amounts of hydrogen are included during exposure to simulated PECVD conditions. The device performance of heterojunction rectifiers is optimized at lower  $\text{O}_2$  relative flow rates, due to a lower resistivity in the NiO films. With recent breakthroughs like use of  $(\text{Al}_x\text{Ga}_{1-x})_2\text{O}_3$  to enhance the bandgap in rectifiers<sup>(46)</sup>, there are now more options available to enhance the performance of these devices.

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## AUTHOR DECLARATIONS

### Conflicts of Interest

The authors have no conflicts to disclose.

## DATA AVAILABILITY

The data that supports the findings of this study are available within the article

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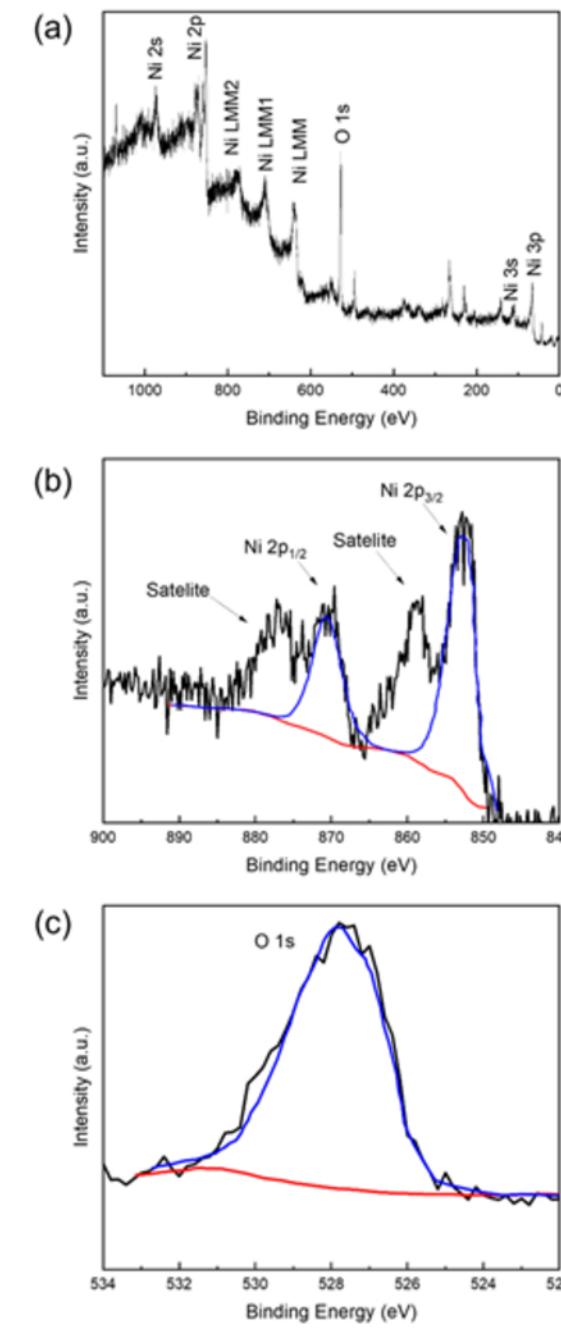
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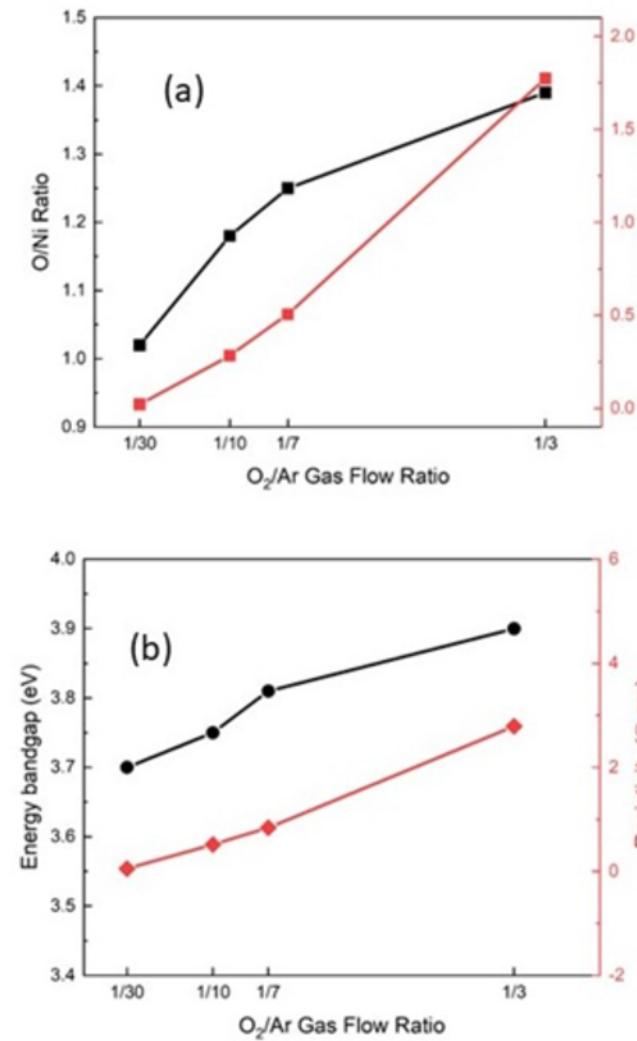
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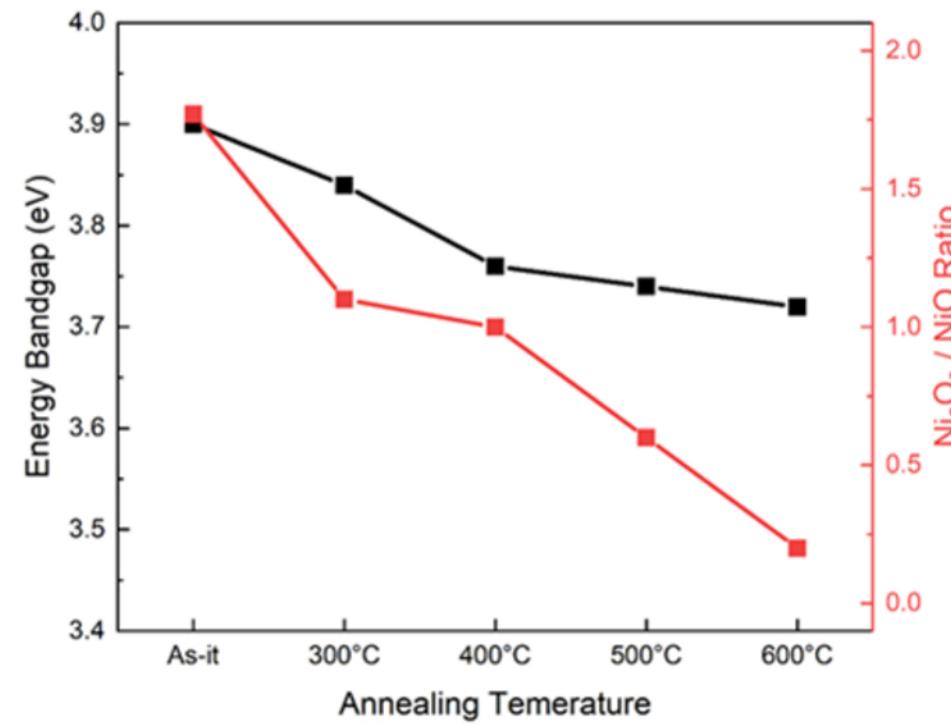


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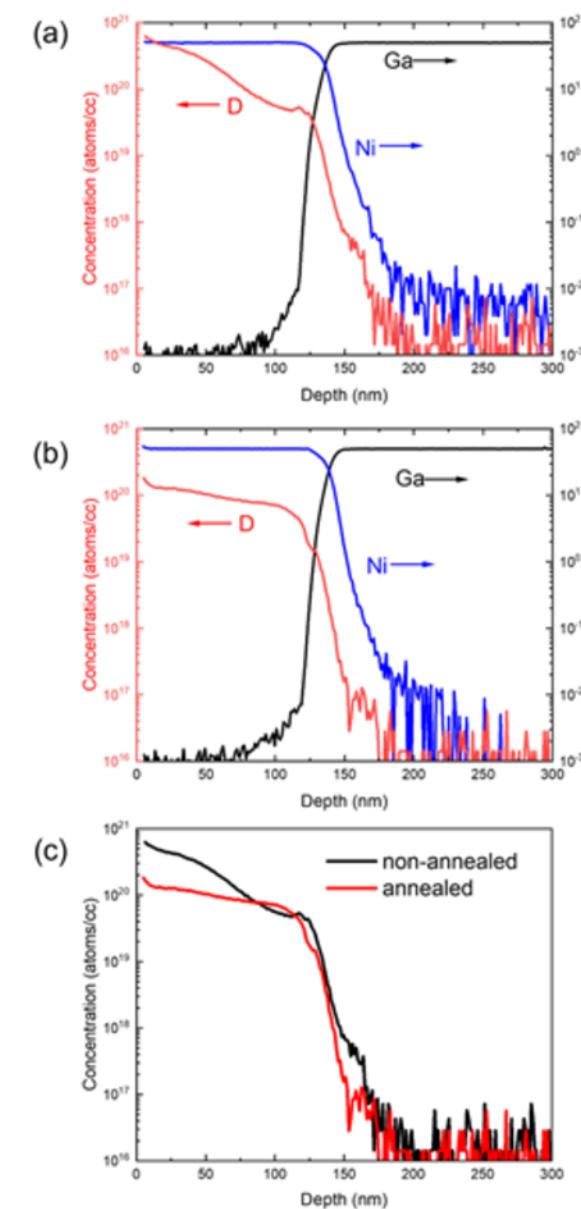


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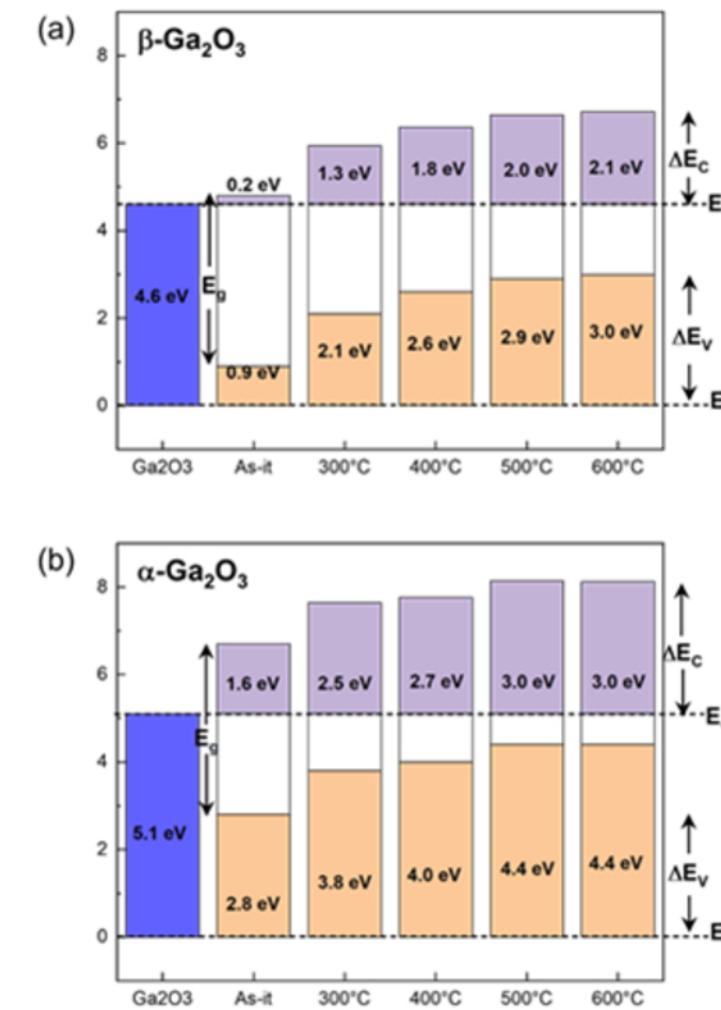
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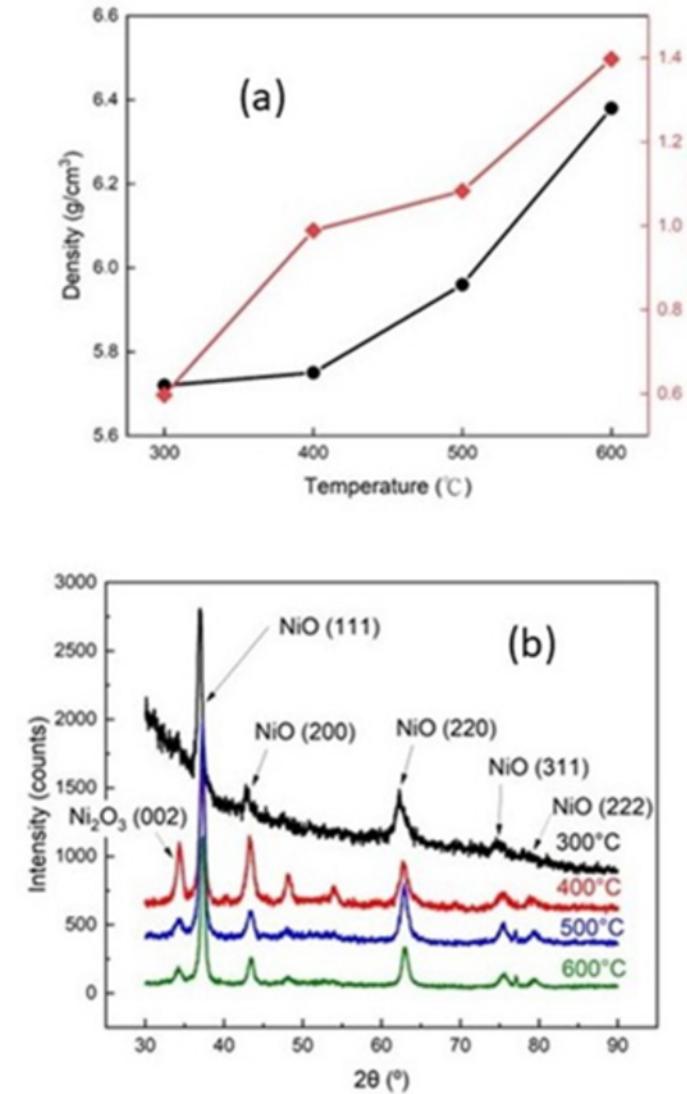
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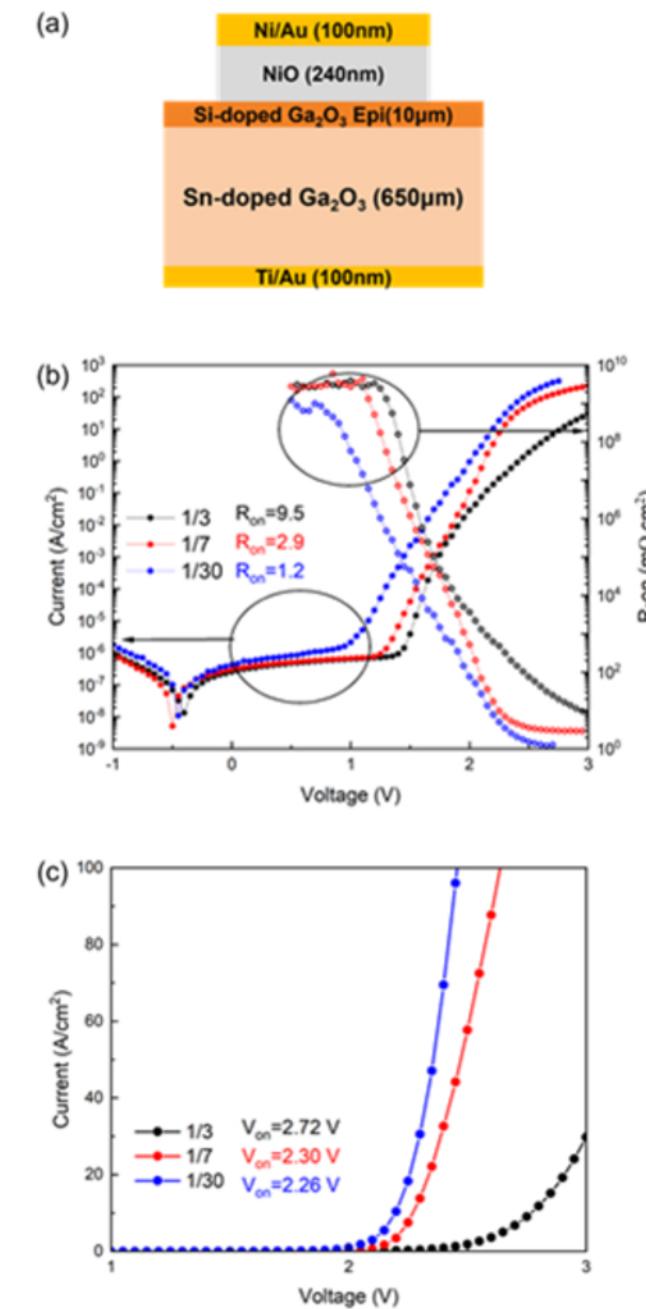
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