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ORIGINAL RESEARCH ARTICLE

Effects of Annealing on Co/Au and Ni/Au Schottky Contacts on β-Ga_2O_3

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Abstract

We have investigated the thermal stability of Co/Au and Ni/Au Schottky contacts to Sn-doped $(\bar{2}01)$ β-Ga_2O_3 substrates. Current–voltage (*I–V*) and capacitance–voltage (*C–V*) measurements were conducted after sequential annealing treatments totaling > 400 h at 300°C and > 150 h at 500°C in vacuum. For both sets of contacts, the average Schottky barrier heights (SBHs) calculated from the *I–V* measurements displayed no significant changes and remained within a narrow range throughout the anneals at 300°C. The SBHs calculated from the *C–V* measurements also remained largely constant for 300°C anneals, except for a modest increase for both contacts for anneal times > 350 h. For 500°C anneals, the *I–V* characteristics for Ni/Au and Co/Au contacts displayed continual degradation in the electrical behavior after 12 h and 24 h, respectively. Characterizations using scanning transmission electron microscopy, energy dispersive x-ray spectroscopy, and scanning electron microscopy of the samples annealed at 500°C revealed considerable changes in film morphology, interdiffusion, and phase segregation within the contacts. The results suggest the potential for Co/Au or Ni/Au Schottky contacts to be used in Ga_2O_3 devices at temperatures below, or possibly up to, 300°C, whereas higher temperatures will require modified metallization schemes.

Keywords Schottky contacts · thermal stability · gallium oxide · dewetting · agglomeration

Introduction

Silicon-based semiconductors have been the dominant semiconductor platform for decades. However, wide band gap semiconductors, such as silicon carbide (SiC) and gallium nitride (GaN), are replacing silicon in applications in which devices need to operate at elevated temperatures, high voltages, and/or higher powers.[1] Beta-gallium oxide (β-Ga_2O_3) is an emerging ultra-wide band gap (E_G ~ 4.8 eV) semiconductor that has attracted much interest due to its high Baliga's figure of merit for power devices and the availability of melt-grown bulk single crystals. Schottky barrier diodes (SBDs), which can operate at higher switching speeds than *p–n* junctions, are of particular interest for power devices.[2] For stable operation of Ga_2O_3 SBDs, there is a need for Schottky contacts to Ga_2O_3 that operate at high temperatures for long periods of time, since the devices can generate heat or become hot from the surrounding environment.

To date, there have been limited studies of the long-term thermal stability of Schottky contacts to Ga_2O_3. Most reports on the effects of temperature on Ga_2O_3 Schottky contacts describe the electrical characteristics as a function of temperature taken from current–voltage–temperature measurements[3] for short heating exposures (e.g., long enough to reach a stable temperature and to conduct the measurements). For example, metal oxides, such as PtOx and IrOx, displayed high Schottky barrier heights (SBHs) on Ga_2O_3 and stable electrical characteristics for limited times at operating temperatures up to 180°C.[4] Lyle et al.[5] investigated the short-term annealing effect of Au/Ti contacts annealed for 10 min from 50°C to 250°C on different orientations. With regard to elemental metals, Fares et al.[6] measured vertical Ga_2O_3 diodes with W/Au and Ni/Au Schottky contacts as a function of temperatures between 25°C and 500°C. The W contacts were more stable than the Ni/Au contacts, but showed evidence of Ga migration through the contact after 500°C device operation. Hong et al.[7] studied the effects of low-temperature post-annealing performed in N_2 gas on Ni/Au Schottky contacts at 100°C and 200°C for 5 min, resulting in the SBH increasing from

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1.07 eV to 1.14 eV after 200°C annealing. Ahn et al.[8] explored the temperature-dependent characteristics of Ni/Au and Pt/Au Schottky diodes. They noted that the ideality factor decreased and the SBH increased with an increase in temperature.

In this study, we investigated the electrical properties of Co/Au and Ni/Au Schottky contacts on single-crystal *n*-type β-Ga_2O_3 substrates as a function of extended annealing time at 300°C and 500°C in air. Co and Ni were chosen because of their high work functions (5 eV and 5.25 eV, respectively), and previous work by our group that showed near-ideal Schottky diode characteristics with an ideality factor (n) of ~ 1.05 for these metals on (100) β-Ga_2O_3.[3] In addition, positive free energies of reaction to form metal oxides (NiO, Co_3O_4, and CoO) from Ga_2O_3 were calculated at 25–500°C using the FactSage Database.[9] This analysis indicates that Ni or Co should not reduce Ga_2O_3 to form Ni-oxide or Co-oxide at the interface, although it does not preclude the possibility of other reactions and reaction products. Electrical properties of the contacts were determined from current–voltage (*I*–*V*) and capacitance–voltage (*C*–*V*) measurements conducted after sequential annealing treatments. Selected samples were characterized using high-resolution transmission electron microscopy (TEM), energy dispersive x-ray (EDX) spectroscopy, and scanning electron microscopy (SEM) to investigate changes in the morphology, microstructure, and chemical composition of the contacts after annealing.

Experimental

A single-crystalline, Sn-doped ($N_D = 2 \times 10^{18}$ cm^{-3}), ($\bar{2}01$) *n*-type β-Ga_2O_3 wafer grown by the edge-defined film-fed method, purchased from Novel Crystal Technology, Japan, was used in this study. Two 10 mm × 10 mm pieces were cut using a dicing saw and used as substrates. The substrates were ultrasonicated in acetone, isopropyl alcohol, and deionized (DI) water for 10 min each followed by a 10% hydrochloric acid clean for 5 min, rinsed in deionized water, and immersed for 5 min in boiling H_2O_2 at 85°C; this process follows the recipe described in Ref.10. The samples were blown dry in nitrogen after each step. All the metals were deposited onto unheated substrates by electron-beam evaporation via a Kurt Lesker PVD 75 deposition system with a base pressure of ~ 2.0 × 10^{-7} Torr. Ohmic contacts were formed using Ti/Au (20 nm/100 nm) on the backside of the substrate; a gap separating the contacts into two halves allowed the ohmic contacts to be tested electrically during the annealing series. The contacts were then annealed at 475°C for 1 min in N_2 in an AG Heat Pulse rapid thermal annealer. Schottky contacts were deposited through a shadow mask on the frontside of the substrates to form circular Schottky contacts with diameters of 125 μm, 250 μm, and 500 μm, and 20-nm-thick Co on one half and 20-nm-thick Ni on the other half. The Schottky contacts were then coated with 80 nm of Au to serve as a capping layer.[11] Cross-section and top-view schematics of the diode structure are shown in Fig. 1.

A resistively heated furnace with a mullite tube was used for the experimental anneals (as opposed to the processing anneals for the ohmic contacts). After loading the samples onto a quartz boat and inserting them into the furnace, the tube was evacuated to a pressure of 4.1 × 10^{-5} Torr for anneals at either 300°C or 500°C. Initial anneal times were 6 h, and then the times were increased to 12 h, 24 h, or 48 h. *I*–*V* measurements were conducted at room temperature using an Agilent 4155C semiconductor parameter analyzer with a Signatone S-1160-4 N probe station. *C*–*V* measurements were taken using an HP 4284A LCR meter at 1 MHz and a bias voltage range from − 5 V to 0 V. Before each use, a zero-correction calibration was performed on an open and closed circuit using two probes. The 125-μm-diameter contacts were selected for electrical measurements, since they were generally found to exhibit less noise in the *C*–*V* measurements than other contact diameters.

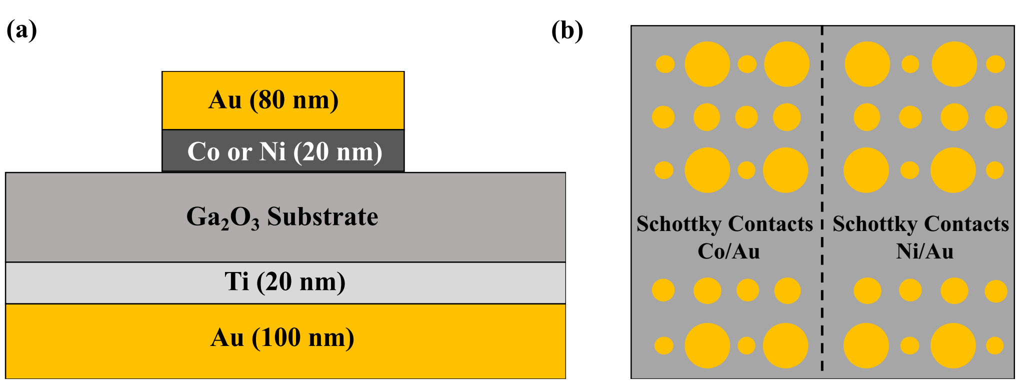

Fig. 1 (a) Cross-section schematic and (b) top view of Schottky diode vertical structure.

To understand the chemical, microstructural, and morphological changes that may occur during annealing, the samples were investigated using a variety of techniques. The focused ion beam-assisted lift-out using a Nova 600 and a Helios 650 was used to prepare the samples for subsequent characterization in a Themis 200 S/TEM. Pt was deposited as a capping layer to mitigate damage during TEM sample preparation. Plan view and cross-sections of the as-deposited and post-annealed samples were analyzed with SEM and STEM imaging, respectively. The SEM images were collected with an Everhart–Thornley detector and a backscattered electron detector in a Quanta 600. Cross-section images and elemental maps were characterized in STEM using high-angle annular dark-field (HAADF) imaging and energy-dispersive x-ray spectroscopy (EDX) on the as-deposited (i.e., pre-annealed) and post-annealed (at 500°C) Co/Au and Ni/Au contacts.

Results and Discussion

For moderately doped semiconductors, current transport is expected to be due to thermionic emission over the Schottky barrier. The *I–V* relationship can then be expressed by:

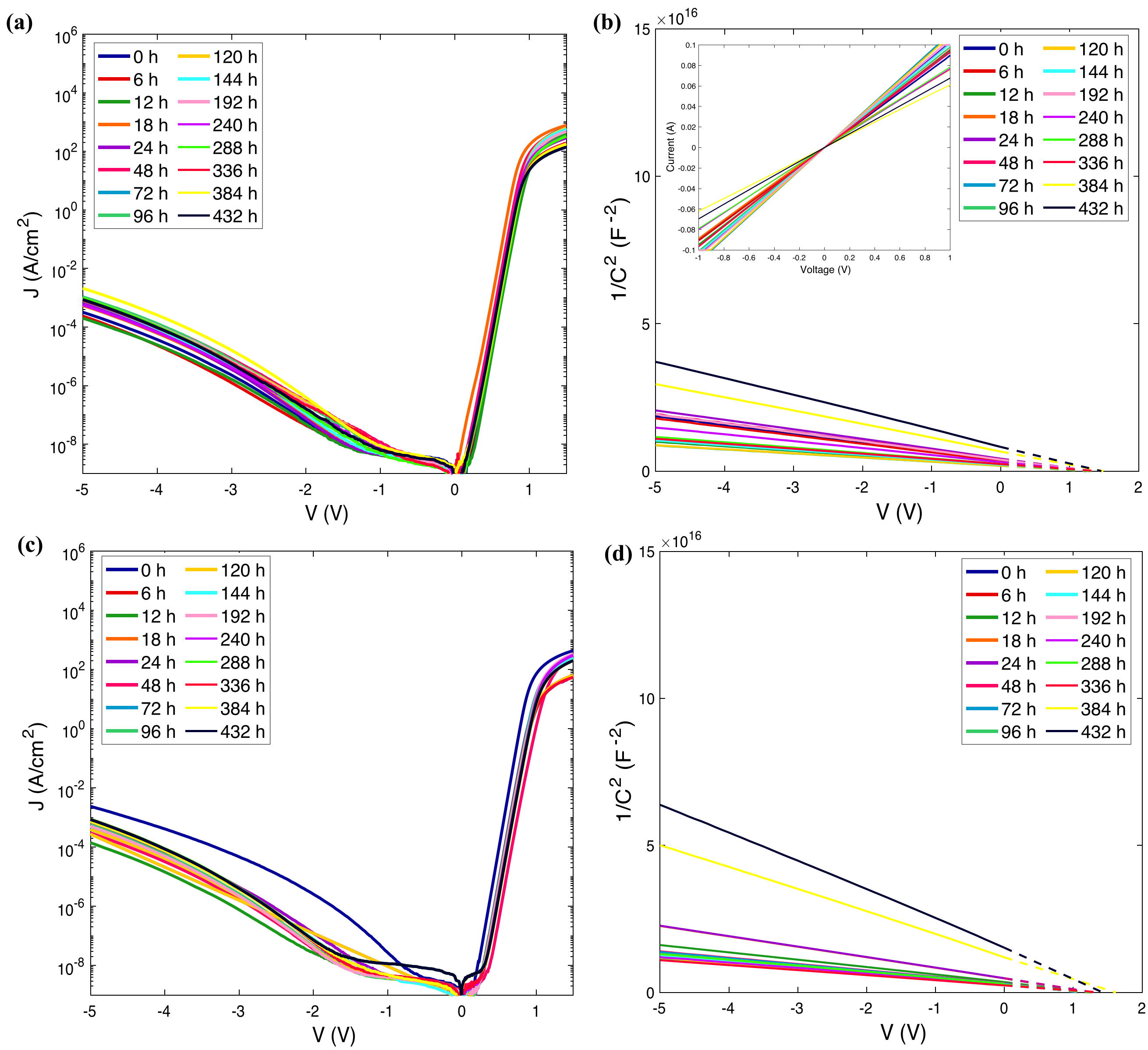

Fig. 2 Representative *J–V* and *C–V* characteristics for different annealing times at 300°C: (a, b) Co/Au contact and (c, d) Ni/Au contact (contact diameter = 125 μm); *inset* in (b) shows *I–V* curves for the backside ohmic contacts after each annealing step.

$$I = I_s \exp\left[\frac{q(V - IR_s)}{nkT} - 1\right] \quad (1)$$

with

$$I_s = AA^{**}T^2 \exp\left(-\frac{q\Phi_B}{kT}\right) \quad (2)$$

where I_s is the saturation current, A is the area of the diode, A^{**} is the Richardson constant, q is the electronic charge, k is the Boltzmann constant, T is temperature, R_s is the series resistance, n is the ideality factor (determines the departure from the ideal diode characteristic), and Φ_B is the SBH. The Richardson constant for β-Ga_2O_3 has been calculated to be 33.65 A/cm^2 K^2.[2] The SBHs (Φ_B^{I-V}) were calculated from the I–V measurements using the method of Cheung and Cheung,[12] as described in our previous study.[3]

The SBHs (Φ_B^{CV}) were also calculated from the C–V measurements using the method outlined by Sze.[13] The depletion-layer capacitance in a Schottky diode is given by:

$$\frac{1}{C^2} = \frac{2\left(q\Phi_B^{CV} - kT\ln\left(\frac{N_c}{N_d}\right) - qV - kT\right)}{A^2q^2\varepsilon_s N_D}. \quad (3)$$

where ε_s is the semiconductor permittivity and N_c is the conduction band density of states:

$$N_C = 2\left(\frac{2\pi m^* kT}{h^2}\right)^{3/2}. \quad (4)$$

where $m^* = 0.28m_o$ is the electron effective mass in β-Ga_2O_3 and m_o is the free electron mass. For β-Ga_2O_3 at room temperature, the conduction band density of states was calculated to be $N_c = 2.67 \times 10^{18}$ cm^{-3}.[2] By plotting $1/C^2$ versus V, the net donor density N_D can be calculated from the slope and Φ_B^{CV} can be calculated from the x-intercept.[14] Plots of $1/C^2$ versus V for the 300°C annealing series for Co/Au and Ni/Au contacts are shown in Fig. 2b and d, respectively. These plots remain linear, implying a constant distribution of N_D through the depth of the sample after each annealing cycle. The slopes of these curves tended to increase with annealing time, indicating a slight decrease in the doping concentration from the initial value of 1.66×10^{18} cm^{-3} to a final value of 3.67×10^{17} cm^{-3}.

Figure 2a and c shows representative current–density (J) versus V characteristics of Co/Au and Ni/Au SBDs on β-Ga_2O_3 after different annealing times at 300°C. For forward bias, Co/Au and Ni/Au display linearity over several decades of current before being limited by series resistance. Relatively high reverse leakage currents are not surprising due to the high doping level in the Ga_2O_3 substrate. For the purposes of this study, changes in the electrical characteristics as a result of annealing are more important than the absolute values of characteristics like the reverse-bias current. No significant changes were observed in the J–V characteristics of the Co/Au diodes throughout the 300°C annealing series for a total of 432 h. The J–V characteristics of the Ni/Au contacts displayed a little more variability between the contacts, but also did not show significant signs of electrical degradation during the 300°C annealing series. The annealing experiments at 300°C were terminated after 432 h due to a furnace controller malfunction.

Figure 3a shows the barrier heights calculated from the I–V and C–V measurements plotted as a function of annealing time at 300°C. It should be noted that the ideality factors were 1.17 eV for both Co/Au and Ni/Au. These moderately high ideality factors are again attributed to the high doping level in the Ga_2O_3 substrate, and suggest that the Φ_B^{I-V}

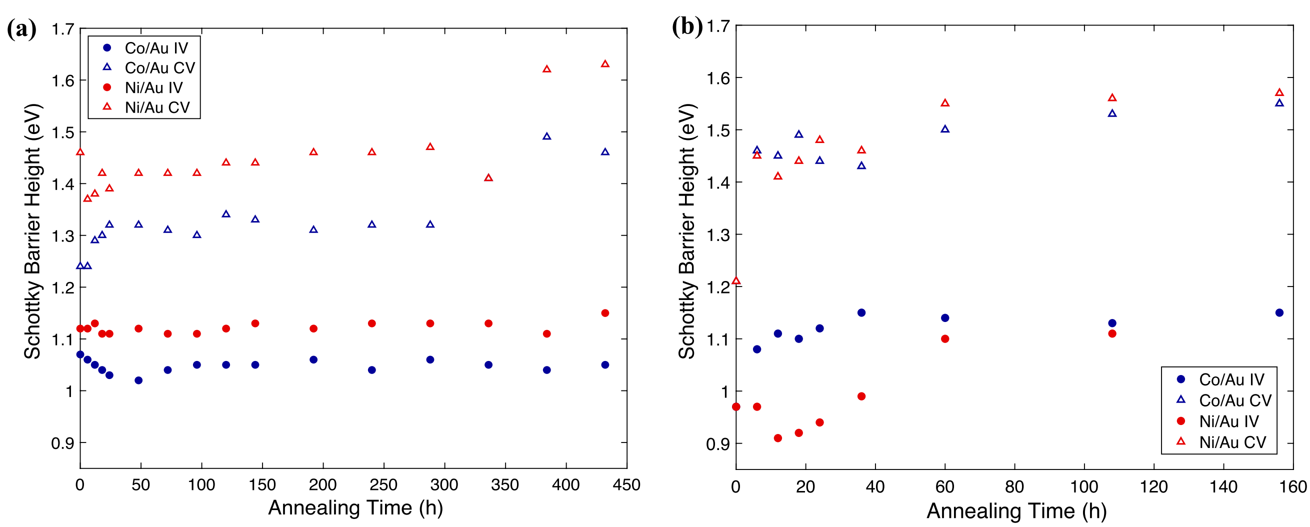

Fig. 3 Average Schottky barrier heights calculated from I–V and C–V measurements versus annealing time at (a) 300°C and (b) 500°C.

values are underestimated. Again, for the purposes of this research study, the changes in the electrical properties as a result of annealing are considered more important than the absolute values. The Φ_B^{I-V} values remained essentially constant throughout the annealing series, with average values of Φ_B^{I-V} (Co/Au) = 1.05 eV and Φ_B^{I-V} (Ni/Au) = 1.13 eV. The Φ_B^{C-V} values also remained relatively constant for at least 300 h, with average values of Φ_B^{C-V} (Co/Au) = 1.33 eV and Φ_B^{C-V} (Ni/Au) = 1.45 eV. It is noted that the Φ_B^{C-V} values are higher (~ 0.3 eV) than the Φ_B^{I-V} values. The Φ_B^{C-V} values are generally expected to be less affected by the high doping in the Ga_2O_3 substrate. Spatially inhomogeneous Schottky barriers at the M–S interface could also account for part of the difference in the Φ_B^{I-V} and Φ_B^{C-V} values.[13] Beyond 350 h, the Φ_B^{C-V} values increased to 1.46 eV and 1.62 eV, respectively. It is unknown why the Φ_B^{C-V} values for both contacts increased for anneal times of 384 h, while the Φ_B^{I-V} values remained essentially unchanged. Overall, the electrical characteristics for the Co/Au and Ni/Au Schottky contacts on β-Ga_2O_3 remained largely stable through 300°C annealing for up to ~ 350–450 h.

After completing the annealing series at 300°C, a separate sample with Co/Au and Ni/Au contacts was characterized electrically, and then used for an annealing study at 500°C.

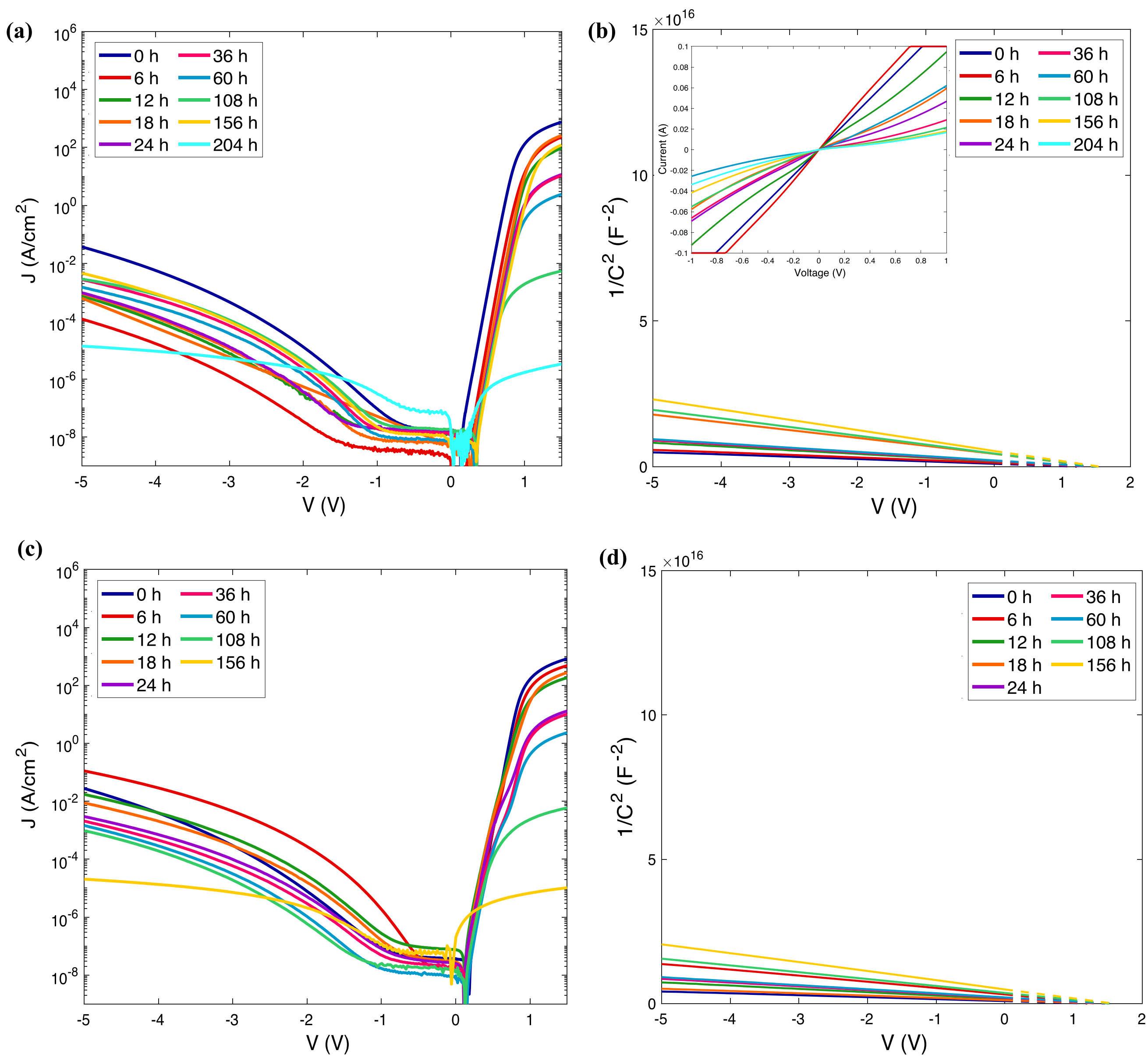

Fig. 4 Representative *J–V* and *C–V* characteristics for different annealing times at 500°C: (a, b) Co/Au contact and (c, d) Ni/Au contact; contact diameter = 125 mm; *inset* in (b) shows *I–V* curves for the backside ohmic contacts after each annealing step.

In contrast to the results for the 300°C annealing (Fig. 3a), significant degradation in the electrical characteristics was observed during the 500°C annealing series (Figs. 3b, 4). The Ni/Au contacts had degraded to the extent that they could not be measured after 156 h, and the Co/Au could not be measured after 204 h. After the first (6 h) anneal at 500°C, the $\Phi_B{}^{I-V}$ and $\Phi_B{}^{C-V}$ values for both contacts tended to show gradual increases through ~ 40–60 h of annealing time (Fig. 3b). Increases in barrier height have been previously reported in Ni Schottky diodes on *n*-type epitaxial layers on conducting bulk Ga_2O_3 substrates at temperatures up to 200°C, attributed to interfacial reactions post-annealing.[5]

It should also be noted that the electrical behavior of the ohmic contacts was monitored throughout the 300°C and 500°C annealing series by placing one probe on each of the two backside ohmic contacts. Modest increases in resistance were observed with increasing annealing time (see insets in Figs. 2b and 4b).

To investigate whether the β-Ga_2O_3 diode performance degradation may be associated with changes in the interfacial chemistry and/or morphology, we characterized samples before and after annealing at 500°C using STEM, EDX, and SEM. As points of reference, cross-section bright field STEM images of the as-deposited Co/Au and Ni/Au contacts (Fig. 5a and c) display relatively sharp interfaces and comprise continuous layers having uniform thicknesses. A difference in image contrast can be observed between the top and bottom layers of the bilayer film, with the Co (or Ni) layer having a brighter contrast than the Au layer, indicating no detectable interdiffusion has occurred between Au and Co or Ni before annealing. There is a noticeable delineation between the Au and Co (or Ni) layers.

In contrast, after annealing at 500°C for 204 h (Co/Au) or 156 h (Ni/Au), the TEM cross-sections show a dramatic difference in morphology, and a redistribution of the metals is observed throughout the contacts, such that separate Au, Co and Ni layers no longer exist (Fig. 5b and d). The films became semi-continuous with distinct Co-rich, Ni-rich, and Au-rich regions.

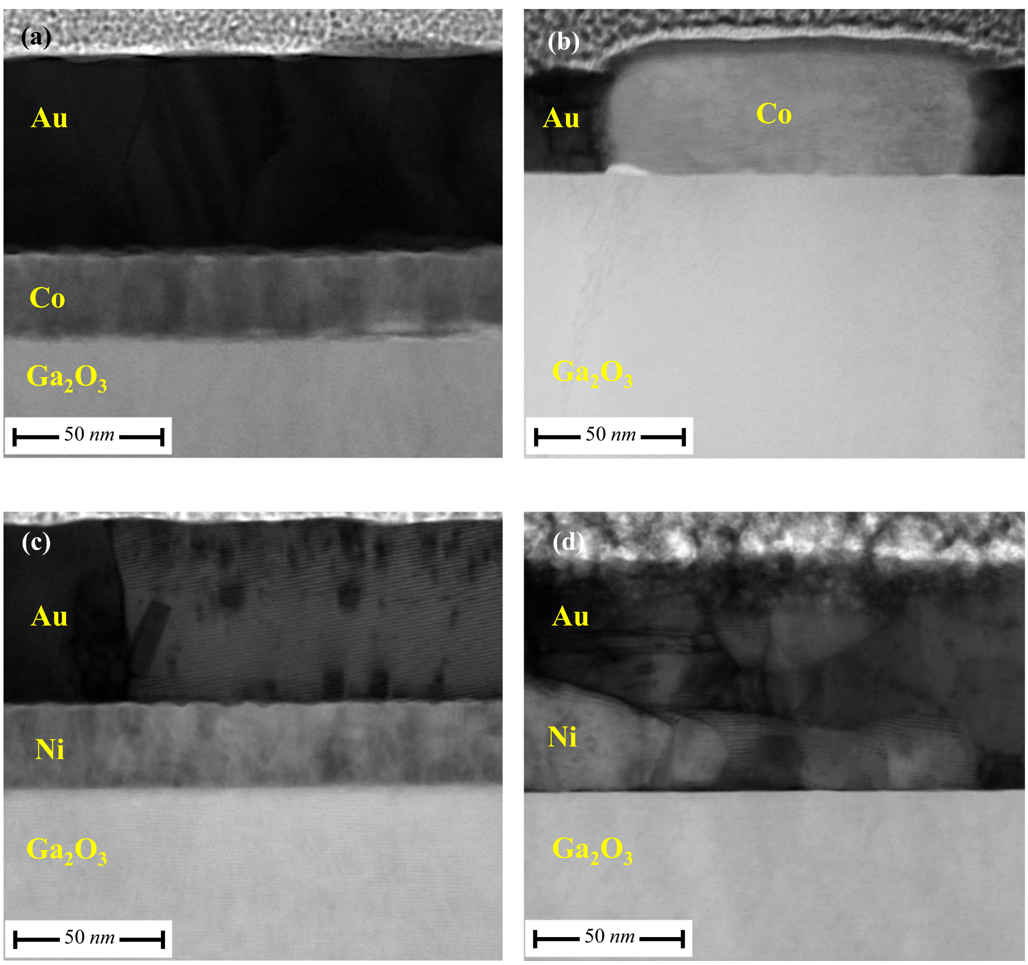

Fig. 5 Bright-field STEM images of the Au/Co/β-Ga_2O_3 interface: (a) pre-anneal, (b) after 204 h at 500°C; bright field STEM images of the Au/Ni/β-Ga_2O_3 interface: (c) pre-anneal, (d) after 156 h at 500°C; [001] zone axis.

HAADF images and EDX elemental maps of the as-deposited Co/Au and Ni/Au contacts, shown in Figs. 6a and 7a, respectively, indicate sharp and abrupt interfaces, in agreement with the STEM images in Fig. 5. No detectable interdiffusion has occurred between or within the layers. There is a well-defined indication of where each metal layer starts and stops. The EDX results confirm that the as-deposited samples do not display any interdiffusion of Co or Ni with the surrounding layers (Ga_2O_3 and Au).

However, EDX maps of samples annealed at 500°C reveal substantial changes in film morphology and chemical distribution. Au and Co (or Ni) formed segregated Au-rich and Co-rich (or Ni-rich) regions across the interface (Figs. 6b and 7b, respectively). The observed phase segregation agrees with the phase diagrams, which indicate that Co and Au do not form solid solutions or compounds with each other; this is also true for Ni and Au at the temperatures of interest here.

Solid films are usually metastable or unstable in the as-deposited state, and can agglomerate to form islands when heated to sufficiently high temperatures. This is driven by surface energy minimization and can occur via surface diffusion well below a film's melting temperature, especially when the film is very thin.[15] The Co/Au contact shows the presence of a large void at the interface with Ga_2O_3 (Fig. 6b), which could be a precursor for agglomeration, otherwise known as solid-state dewetting.[16] Dewetting proceeds in several stages, including void initiation, void growth. and void coalescence.[17] Once a hole in the film has formed, it can grow subsequently by continuous diffusion, exposing the surface of the underlying substrate.

SEM was used as a complementary tool to characterize the top surfaces of the contacts. Before annealing, the SEM images (not shown) showed smooth, uniform surfaces of the contacts. Figure 8 shows representative secondary electron and backscattered electron images of Co/Au contacts annealed at 500°C for 204 h and Ni/Au contacts annealed at 500°C for 156 h. All the images show clear evidence of phase segregation, with Co-rich or Ni-rich regions embedded in a matrix of polycrystalline Au. EDX spectra (not

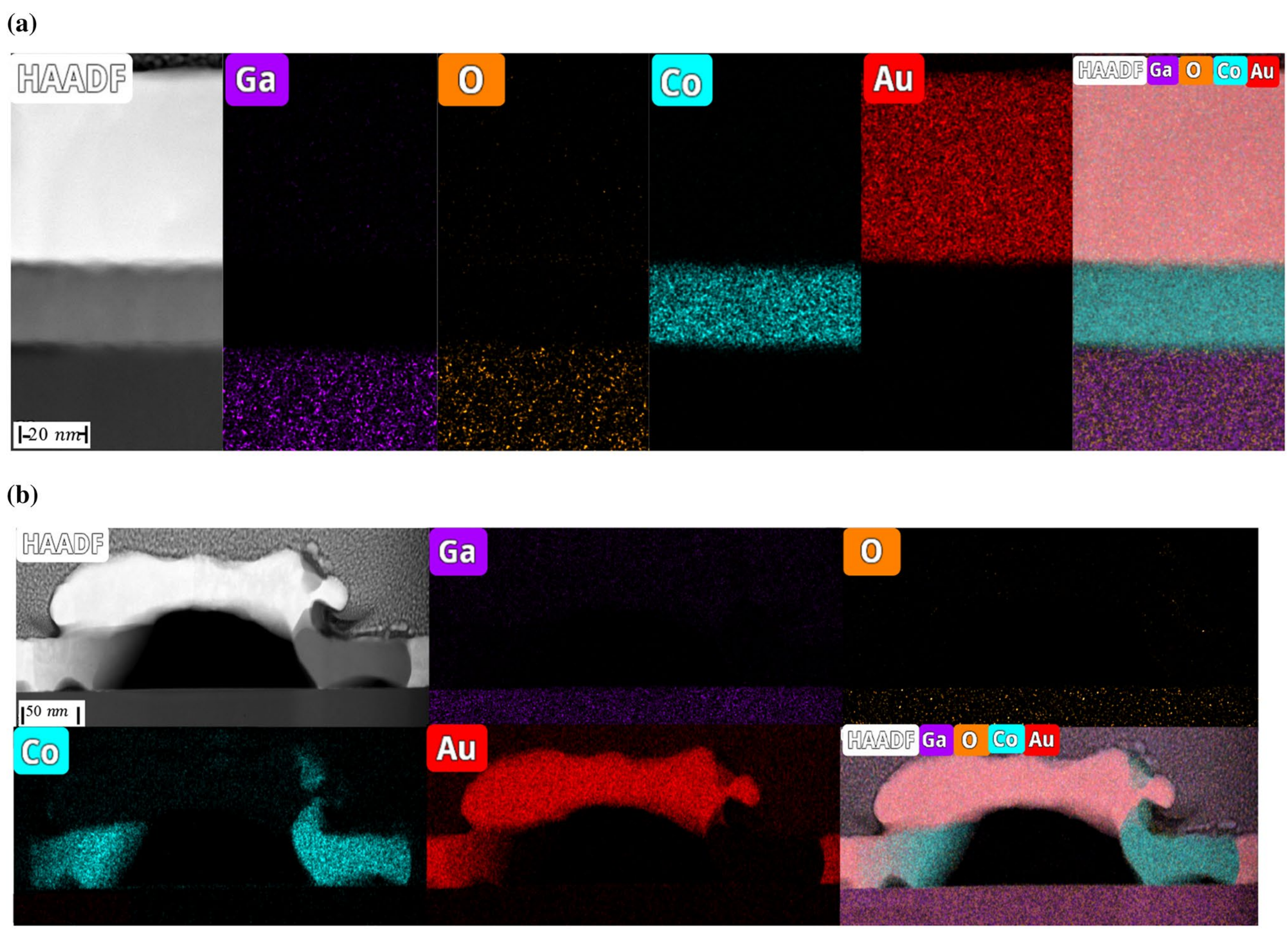

Fig. 6 HAADF and EDX maps of the Au/Co/ β-Ga_2O_3: (a) pre-anneal, (b) after 204 h at 500°C; the elemental distribution counts are shown as purple (Ga), orange (O), blue (Co), and red (Au) (Color figure online).

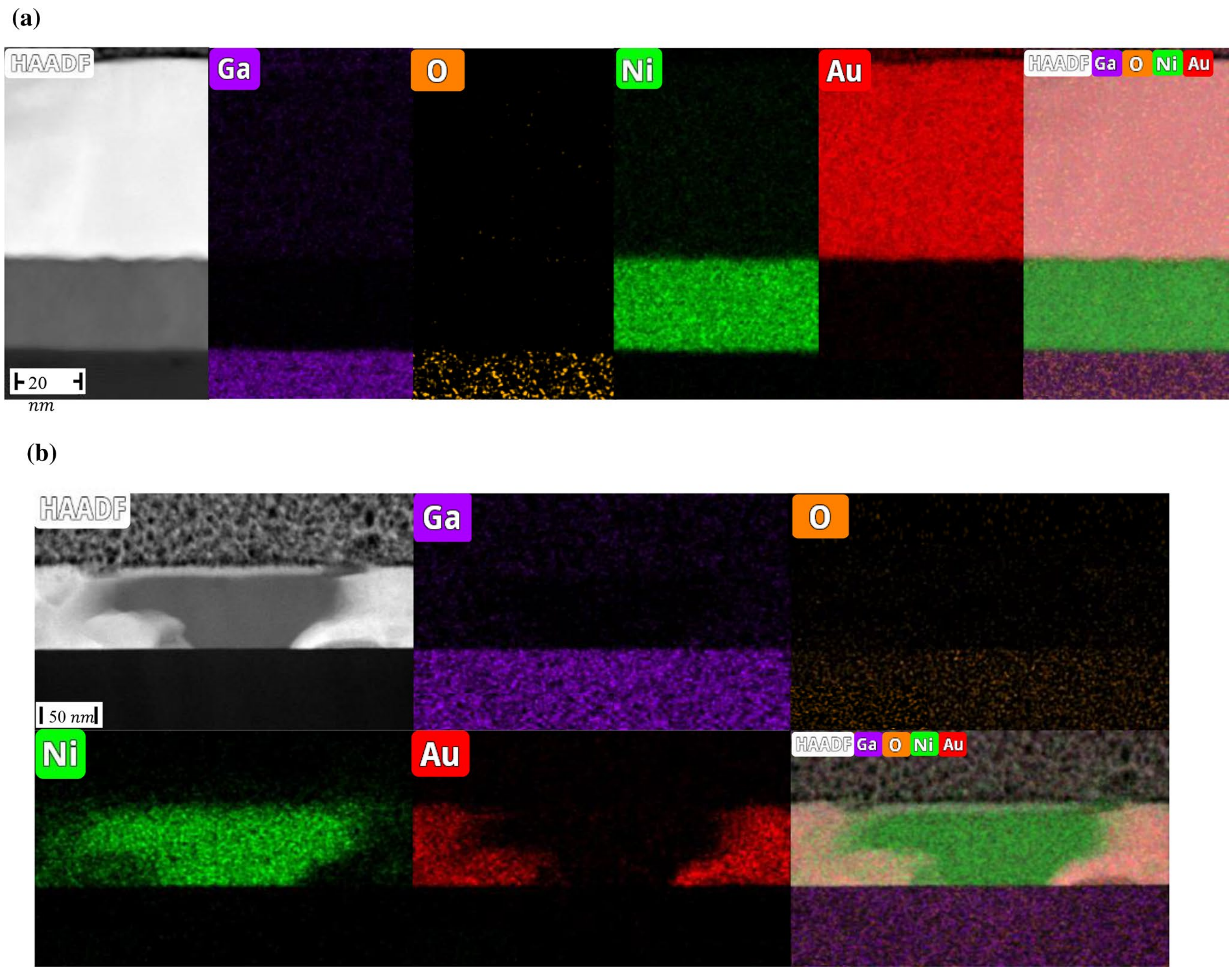

Fig. 7 HAADF and EDX maps of the Au/Ni/β-Ga_2O_3: (a) pre-anneal, (b) after 156 h at 500°C; The elemental distribution counts are shown as purple (Ga), orange (O), green (Ni), and red (Au) (Color figure online).

shown) confirmed that the regions that appear light gray are the Au-rich areas, whereas the dark areas correspond with Co-rich or Ni- rich phases.

Conclusions

We have investigated changes in the electrical characteristics of Ni/Au and Co/Au SBDs on $(\bar{2}01)$ β-Ga_2O_3 substrates as a function of annealing time at 300°C and 500°C in vacuum. The *I–V* and *C–V* characteristics remained largely stable throughout the annealing series at 300°C for > 400 h. However, for 500°C anneals, continual changes were observed, with significant degradation evident in the *I–V* characteristics after 156 h for the Ni/Au contacts and after 204 h for the Co/Au contacts. Characterization of the samples annealed at 500°C using STEM, EDX, and SEM revealed substantial interdiffusion and phase segregation within the contacts. The results point to the potential use of these pure-metal (Co/Au or Ni/Au) contacts in Ga_2O_3 Schottky diodes at temperatures below, or possibly up to, 300°C, whereas alternative contact metallization schemes, possibly incorporating metal oxides,[4] will be necessary for Ga_2O_3-based device operation at temperatures at or above 300°C.

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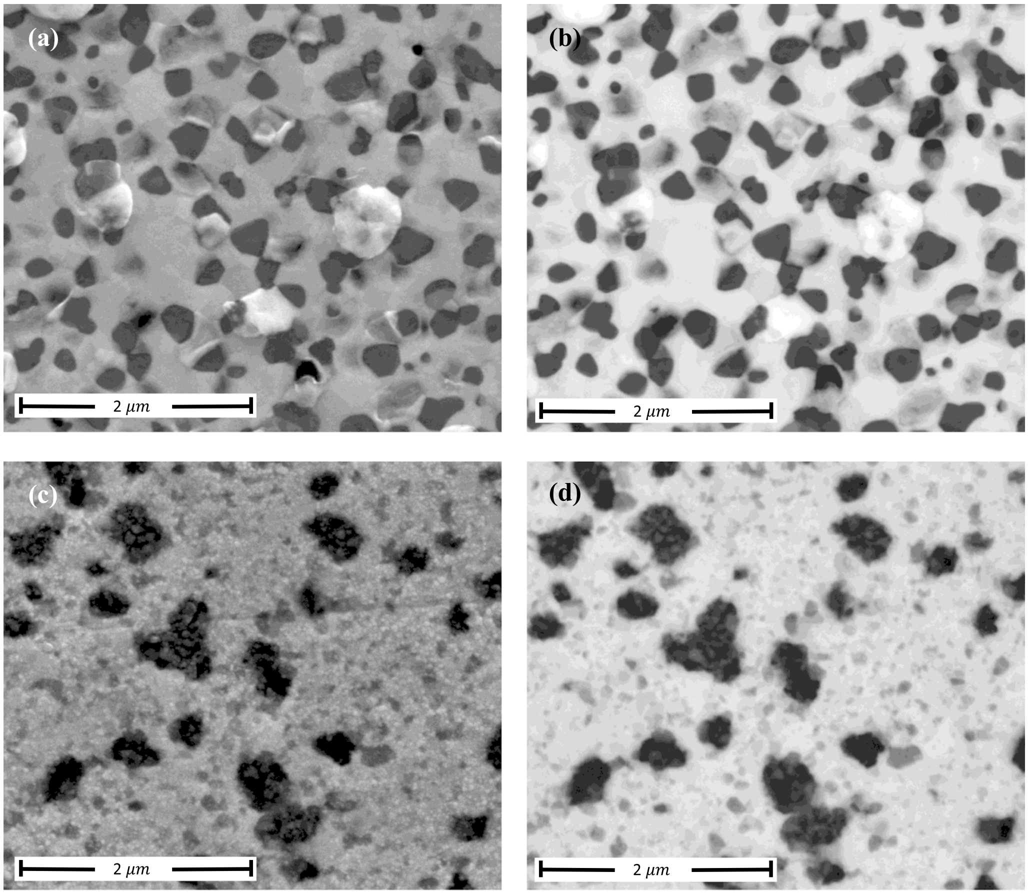

Fig. 8 SEM micrographs of samples annealed at 500°C: (a, b) Co/Au after 204 h, (c, d) Ni/Au after 156 h; secondary electron images are shown in (a) and (c); backscattered electron images are shown in (b) and (d).

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Conflict of interest The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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