Rinsing Effects on the Structural and Morphological Evolution of BiOI Thin Films Prepared from BiI_3

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Abstract: Bismuth oxyiodide (BiOI) is a layered semiconductor with visible-light absorption and potential for photocatalytic, photovoltaic, and optoelectronic applications. In this study, the effect of rinsing treatment on BiOI thin films prepared from BiI_3 by thermal oxidation was investigated. BiOI was deposited on cleaned glass/FTO substrates by BiI_3 heating at 360 ºC for 30 min. The resulting films were subjected to three treatment conditions: no rinsing, rinsing with an ethanol/water mixture, and rinsing with water, followed by reheating at 350 ºC for 15 min. X-ray diffraction (XRD) identified tetragonal BiOI as the dominant crystalline phase, with the (001) and (102) reflections being prominent. Rinsing was accompanied by the appearance of additional reflections and a narrowing of the (001) peak. The FWHM decreased from 0.871º for the non-rinsed film to 0.489º and 0.471º after ethanol/water and water rinsing, respectively. Raman spectroscopy provided complementary evidence of BiOI through the characteristic Bi-I stretching feature near 149.6 cm^{-1}. UV-Vis analysis of the prepared films gave an optical band gap of 1.58 eV. FESEM observations showed a more pronounced vertically oriented flaky morphology after rinsing. Overall, the results indicate that rinsing is an important processing parameter that is associated with changes in the crystallographic and morphological development of BiOI films prepared by thermal oxidation of BiI_3.

Keywords: BiOI; Thin film; BiI_3; Thermal oxidation; Rinsing treatment; Structural; Morphology.

1. INTRODUCTION

Bismuth oxyiodide (BiOI) is a layered bismuth oxyhalide semiconductor that has attracted considerable research interest. This semiconductor has a narrow band gap and good response in the visible spectral region. Actually, the crystal structure of BiOI consists of arrangement of positively charged namely $[Bi_2O_2]^{2+}$ slabs separated by iodide anions [1,2]. This structural feature is relevant to the optical and electronic behavior of BiOI that has good contribution as visible-light photocatalysts and photovoltaic absorbers [3–5]. BiOX (X = Cl, Br, I) family also shows systematic changes in optical band gap with the changing of halide anions and BiOI located in the visible-light region [6,7].

BiOI has been reported with diverse morphologies, including microspherical, layered, and flower-like structures. These differences can be achieved by modification through different dosage of solvent and pH [8]. Moreover. BiOI flakes-like and nanosheets have been reported to promote the photocatalytic removal of organic contaminants under simulated sun light exposure [9]. Beyond photocatalysis, BiOI thin films have also been investigated for photovoltaic applications. Hoye *et al*. reported air-stable BiOI thin films and demonstrated BiOI-based all-inorganic solar cells. The same study reported a bulk carrier lifetime of 2.7 ns [10]. Those reports support continued interest in controlling the structure and morphology of BiOI thin films.

Several routes have been developed to obtain BiOI and its derivative films, including successive ionic layer adsorption and reaction (SILAR) [11], spin coating [12], and doctor-blade deposition [13], while hydrothermal [14] and sol-gel approaches [15] are commonly used for BiOI powders. The present study explores a different preparation concept: direct annealing of BiI_3 in air like in the previous work [16]. The process is represented by the oxidation reaction $BiI_3 + 1/2O_2 \rightarrow BiOI + I_2$.

Previous studies have demonstrated the preparation of BiOI thin films through the thermal oxidation of BiI_3 and have examined the effects of thermal-processing conditions on their structural and functional properties [17]. In contrast, the present study focuses on the influence of an intermediate rinsing

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treatment under a fixed thermal-processing configuration. Three conditions, i.e no rinsing, ethanol/water rinsing, and water rinsing are compared to examine the associated changes in X-ray diffraction features and surface morphology, supported by Raman and UV-Vis characterization. The objective is to establish whether this simple processing step is associated with measurable differences in the structural and morphological development of BiOI thin films, without assuming a specific chemical or crystallization mechanism.

2. EXPERIMENTAL SECTION

2.1. Film Preparation

Cleaned glass/FTO substrates were used as the deposition substrates. 0.1 g of BiI_3 (purchased from Nacalai Tesque Inc. Japan) was placed in an aluminum-foil-covered Petri dish, while the substrate was positioned above the BiI_3 source. The system was heated at 360 ^oC for 30 min in air, resulting in the formation of an initial BiOI film on the substrate. The deposited films were subjected to one of three simple treatments: (i) no rinsing, (ii) rinsing with ethanol/water 50:50%, or (iii) rinsing with water. Rinsing treatment was conducted by dipping the prepared films for 60 second. The rinsed and non-rinsed films were subsequently reheated at 350 ^oC for 15 min in a hot plate at ambient situation. The illustration of film preparation is shown in Fig. (**1**). This work was adapted from Mabuti, *et al*. [18] .

2.2. Characterization

The structural properties of the films were examined by X-ray diffraction (XRD, Rigaku RINT-2100 diffractometer), while Raman spectroscopy (JASCO NRS 2100) was used to identify characteristic vibrational features. Optical absorption was investigated using UV-Vis spectroscopy (JASCO 670 UV), and the optical band gap was estimated from a Tauc plot. Surface morphology was examined by field-emission scanning electron microscopy (FESEM, JEOL JSM-7100F).

3. RESULTS AND DISCUSSION

Figure (**2**) presents the XRD patterns of BiOI films prepared under the different rinsing treatments. The diffraction patterns demonstrate that BiOI can be obtained from BiI_3 by annealing in air. The dominant reflections are assigned to the (001) and (102) crystal planes, consistent with the tetragonal layered matlockite-type BiOI structure. The crystal planes that refer to (001) and (102) indicates the crystal structure of BiOI that matches to reference pattern (JCPDS card No. 73-2062) [19]. The presence of these characteristic reflections confirms that the thermal treatment produced the BiOI phase under the conditions employed in this study.

The rinsing treatment produces visible changes in the diffraction profiles. In particular, the appearance or relative intensity of additional reflections of (110) planes

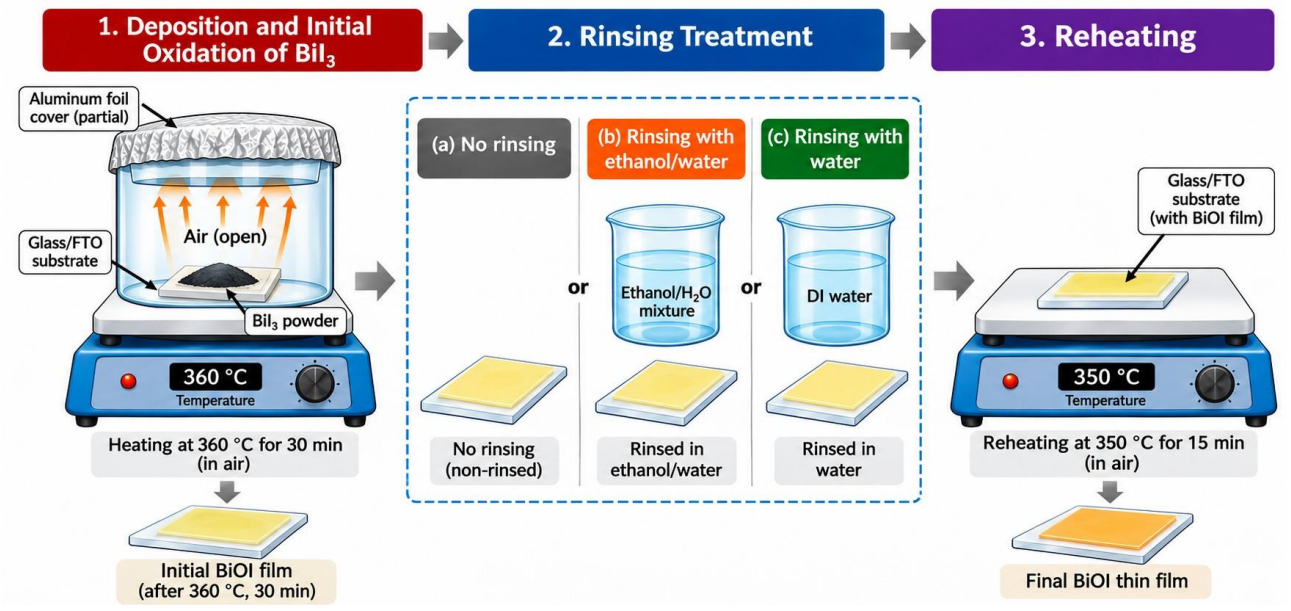


Figure 1: Schematic representation film preparation by oxidation/annealing of BiI_3.

becomes apparent after rinsing. Other reflections, including (002), (200), and related planes, are also still observed under the rinsing conditions. These observations indicate that the rinsing treatment influences the crystallographic development of the BiOI film.

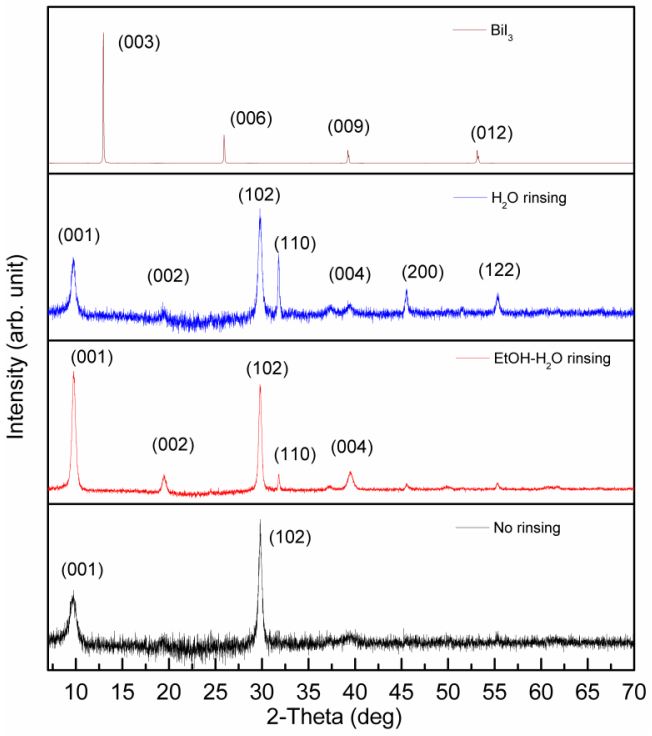


Figure 2: XRD patterns of BiOI films prepared under different rinsing conditions.

A more pronounced difference was observed in the width of the (001) diffraction peak. As summarized in Table **1**, the FWHM of the (001) reflection decreased from 0.871° for the non-rinsed film to 0.489° and 0.471° for the ethanol/water and water rinsed films, respectively. The narrower diffraction peak after rinsing indicates a reduction in X-ray diffraction peak broadening and is consistent with changes in the degree of structural ordering within the BiOI films. The slightly smaller FWHM obtained for the water-rinsed film compared with the ethanol/water-rinsed film suggests that the two rinsing conditions may not influence the subsequent crystallographic development in exactly the same manner.

Table 1: FWHM of the (001) Reflection

Treatment	FWHM (°)
No rinsing	0.871
Rinsing in EtOH/water	0.489
Rinsing in water	0.471

The observed changes in the diffraction patterns can reasonably be associated with the condition of the BiI_3-derived film prior to the final thermal treatment. Rinsing may modify the surface condition and/or the distribution of residual precursor species on the deposited film, which could subsequently influence the formation and development of BiOI crystallites during reheating. Overall, the XRD results demonstrate that rinsing is associated with measurable changes in the crystallographic development of the BiOI films, particularly in the appearance of additional reflections and the narrowing of the (001) diffraction peak. These observations provide structural evidence that the rinsing step can influence the evolution of BiOI thin films during thermal conversion of the BiI_3 precursor.

3.2. Raman spectroscopic confirmation of BiOI

Raman spectroscopy provides complementary evidence for the formation of BiOI and allows the vibrational response of the films to be examined under rinsing and no-rinsing conditions. As shown in Fig. (**3**), the films prepared with ethanol/water rinsing and without rinsing exhibit a characteristic Raman feature at approximately 149.6 cm^{-1}. This band is assigned to the E_g vibrational mode associated with Bi-I stretching. The experimental position is consistent with values reported for BiOI-based materials. Park *et al*. reported a Bi-I stretching feature near 149.6 cm^{-1} [20].

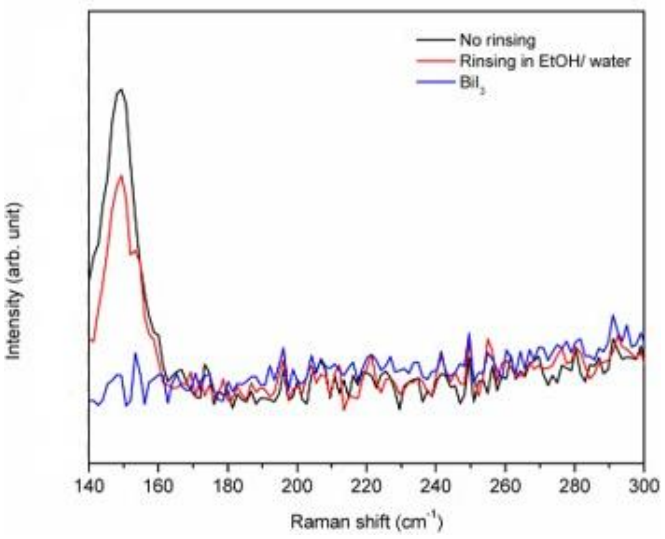


Figure 3: Raman spectra of BiI_3/BiOI films prepared under different rinsing conditions.

Importantly, the characteristic Bi-I-related with Raman feature is retained under the water/ethanol rinsing processing and no-rinsing conditions. This observation is consistent with the XRD results, which

identify tetragonal BiOI as the dominant crystalline phase. Thus, within the resolution of the present measurements, rinsing does not appear to prevent the formation of BiOI. Instead, the main effect observed after rinsing is related to the development of the diffraction profile and surface morphology rather than an evident loss of the BiOI vibrational signature. In addition, the Raman results serve as complementary phase evidence, while the XRD data provide the more direct information regarding changes in crystallographic development.

3.3. Optical properties

The UV-Vis spectra in Fig. (**4**) show the optical response of the films prepared under the different rinsing conditions. The absorption occurs predominantly in the visible spectral region, consistent with the narrow band gap nature of BiOI. The Tauc plots in Fig. (**5**) were used to estimate the optical band gap, and an experimental value of 1.58 eV was obtained. This value lies within the low-energy range associated with visible-light absorption by BiOI and is lower than the approximately 1.8 eV band gap reported for BiOI thin films prepared by aerosol-assisted chemical vapour deposition [6]. Differences in film morphology, thickness, crystallinity, and preparation route can contribute to variations in experimentally determined optical band gaps.

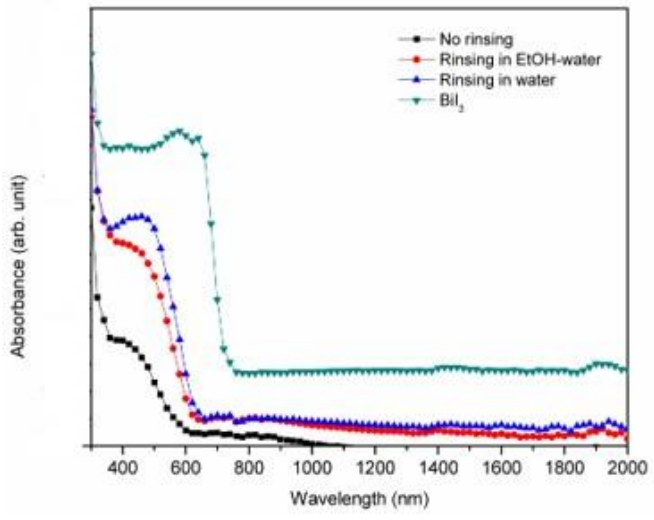


Figure 4: UV–Vis absorption spectra of BiOI films prepared under different rinsing conditions.

3.4. Surface Morphology

Figure (**6**) compares the FESEM images of the BiI_3 precursor film and the BiOI films obtained after the different treatments. The precursor film exhibits a relatively compact surface, whereas the converted BiOI films display the flaky morphology characteristic of layered BiOI. The non-rinsed sample shows a comparatively less vertically developed flaky texture, while the rinsed samples exhibit a more pronounced population of vertically oriented flakes. Therefore, the rinsing process can be a factor that influences the morphology of the synthesized BiOI film.

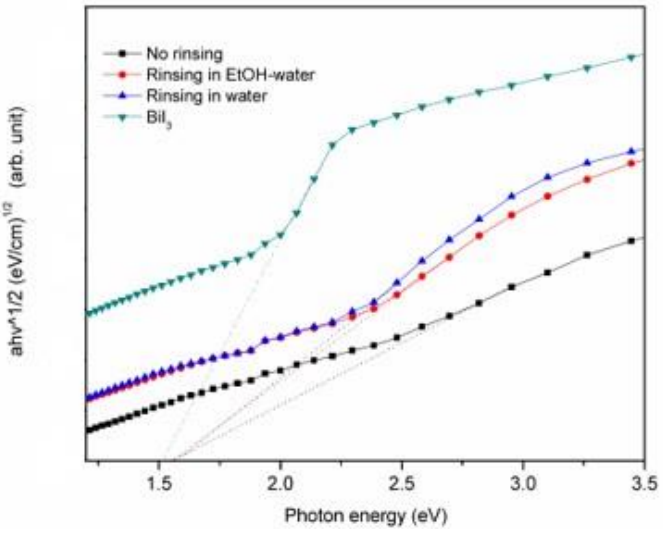


Figure 5: Tauc plots used for estimation of the optical band gap of the prepared films.

The morphological changes can be considered together with the XRD results. Rinsing was accompanied by narrower (001) diffraction peaks, while FESEM showed a more pronounced flaky morphology. These observations suggest that the rinsing step is associated with changes in the structural and morphological development of the BiOI film during the subsequent heat treatment. The observed changes may be related to modification of the surface condition or distribution of precursor-derived species before the final thermal treatment. Such changes could influence the subsequent development of BiOI crystallites during reheating. Therefore, this result is important because a simple post-deposition treatment can modify several film properties without introducing a new deposition technique. The direct annealing approach also offers a conceptually simple route from a BiI_3 precursor to BiOI.

Overall, the results obtained from XRD, Raman spectroscopy, UV-Vis spectroscopy, and FESEM can be considered together to understand the role of rinsing in the development of the BiOI films. The XRD results show that all investigated films exhibit the characteristic tetragonal BiOI structure, while rinsing is accompanied

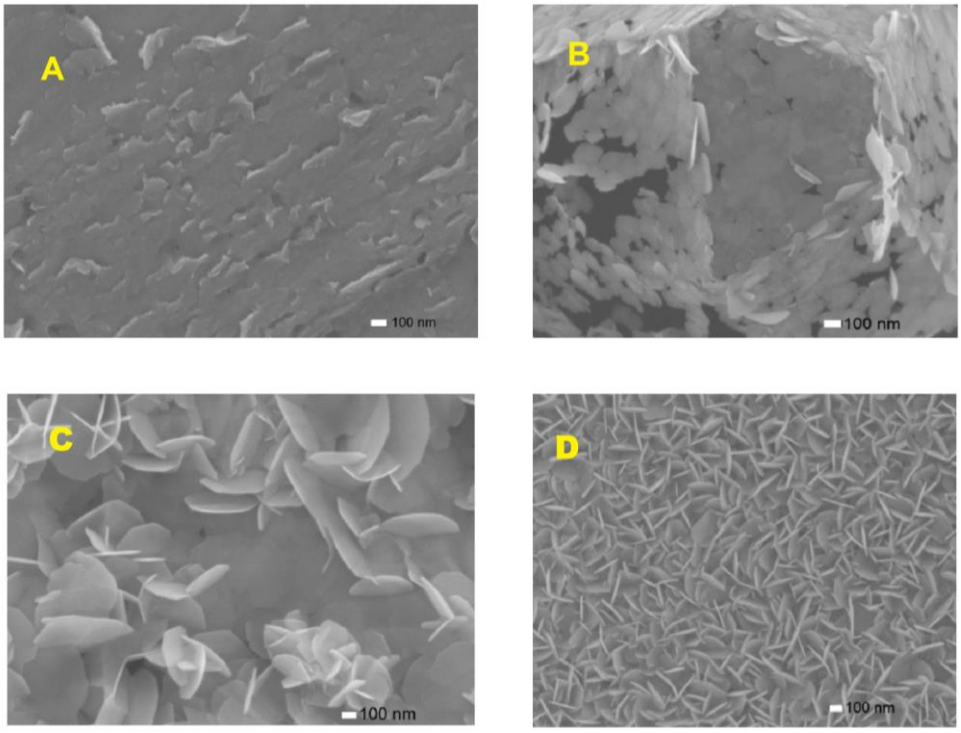


Figure 6: FESEM images of BiI_3 film (A), BiOI film: without rinsing (B), rinsing in ethanol/ water (C), rinsing in water (D).

by changes in the relative appearance of diffraction reflections and a substantial narrowing of the (001) peak. The FWHM decreases from 0.871° for the non-rinsed film to 0.489° and 0.471° for the ethanol/water and water rinsed films, respectively. These changes indicate that the rinsing treatment is associated with differences in the crystallographic development of the films during the subsequent thermal treatment. The FESEM observations provide complementary morphological evidence. Compared with the non-rinsed film, the rinsed films exhibit a more pronounced vertically oriented flaky morphology. Because BiOI possesses a layered crystal structure, the development of flake-like features is consistent with the morphological characteristics commonly observed for this material.

A possible explanation about that circumstance is the rinsing step modifies the surface condition or distribution of residual precursor-derived species before the final reheating at 350 °C. Such changes could affect the local conditions under which the BiOI film undergoes further structural development during reheating. The subsequent changes in diffraction peak width and surface morphology are consistent with this possibility.

From a thin-film processing perspective, the present results show that rinsing can be considered as an additional processing variable in the thermal conversion of BiI_3-derived BiOI films. The treatment does not require a change in the deposition method or the introduction of a new film-forming technique, yet the resulting films exhibit measurable differences in their diffraction profiles and surface morphology. This observation is relevant because relatively simple post-deposition treatments may influence the subsequent development of functional thin films. Further studies involving controlled rinsing conditions and complementary compositional or cross-sectional characterization would be useful to clarify the origin of the observed structural and morphological changes. That studies would be useful for clarifying how the rinsing step affects BiOI film development.

4. CONCLUSIONS

Bismuth oxyiodide (BiOI) thin films were fabricated via the direct annealing of BiI_3 in air. It hihlighted the effect of an intermediate rinsing treatment on the subsequent development of BiOI films. While both rinsed and non-rinsed samples confirmed the successful synthesis of BiOI through XRD and Raman characterization. From this work, the rinsing step noticeably altered the XRD profiles and FWHM of (001) plane. Raman spectra revealed the expected Bi-I stretching mode at approximately 149.6 cm^{-1} and UV-Vis Tauc plots indicated an optical band gap of 1.58 eV.

Furthermore, FESEM imaging showed that rinsing modified the film's morphology by inducing more vertically oriented flakes. The more flakes were generated due to the rinsing treatment. These findings indicate that rinsing is an important processing parameter associated with the crystallographic and morphological development of BiOI films. Further studies are needed to study more quantitatively the relationship between the processing conditions, film structure, morphology, and functional properties.

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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