
A novel thin film phase of oriented MgO grown from a liquid solution

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Abstract

Close lattice matching with a MgAl₂O₄ (001) substrate has been used to synthesize thin films of a rare spinel-like polymorph of MgO (β-MgO) that is unstable in bulk form. The film is produced by spin casting an acetate-substituted Mg-ethoxide precursor, which forms a crystalline magnesium acetate phase at room temperature. Pyrolysis of most organic constituents occurs during continuous heating from room temperature to 400 °C and is followed by formation of phase-pure β-MgO at T < 700 °C. Four-circle X-ray diffraction indicates that the film is highly oriented with the substrate lattice, with <0.05° tilt or twist between the (001) planes of the film grains and the (001) planes of the substrate. Kinetic and/or energetic constraints arising from the presence of a substrate are responsible for stabilizing β-MgO: bulk powders formed from the same precursor yield only the usual rock salt MgO structure and, although the formation of some β-MgO occurs on widely different substrate types, its volume fraction crystallographic orientation can be tuned by using closely lattice-matched substrate crystals. © 2001 Published by Elsevier Science B.V.

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1. Introduction

Magnesium oxide is one of several materials used as thin insulating layers in electronic devices [1–6]. It has the advantage of remaining chemically stable at the elevated temperatures at which subsequent thin layers may be grown. Its lattice parameter (4.21 Å) is close to that of several important ferroelectric oxides, making it possible to template the textured growth of such oxides for device applications. MgO has the added advantages of a wide band gap, low optical loss, and a relatively low refractive index (~1.7) which can permit confined optical modes in many ferroelectric materials. To date, techniques such as pulsed laser deposition [7], RF sputtering [8], ion beam-assisted deposition [9], metalorganic chemical vapor deposition (MOCVD) [10], spray pyrolysis [11], and sol–gel spin casting [12,13] all have been used to deposit textured films of MgO on GaAs and Si at low temperature (<400°C).
MgO is a highly ionic solid with an electronegativity difference of 2.13. Moreover, its cation/anion radius ratio of 0.50 implies that it is stable only in the common rock salt (NaCl) crystal structure. The stability and prevalence of this structure is reflected in the facts that (1) all natural deposits of MgO (periclase) have the rock salt structure, and (2) virtually all synthetic processes for producing MgO from MgCO₃ [14,15], from Mg(OH)₂ [14,16–18], or from oxidation of Mg [19–21], result in this crystal form. Not surprisingly, in every previous investigation of the growth and properties of MgO thin films, only the rock salt structure has been observed at all processing temperatures [7–12].

Despite its nearly universal occurrence in the rock salt structure—hereafter called α-MgO—MgO can also exist as a rare polymorph with a spinel-like cubic structure and roughly twice the lattice parameter (8.12 Å) of α-MgO. So uncommon is this polymorph, to which we will refer as β-MgO, that we are aware of it being observed only twice [13,22]. Freund [22] reported the appearance of β-MgO as a transient phase mixed with α-MgO during slow, in vacuo dehydration of Mg(OH)₂.

More recently, we have used sol–gel deposition on Si(111)/SiOₓ substrates to synthesize polycrystalline, 100-nm films of MgO that are composed of both α and β forms [13]. Significantly, powders synthesized by the same sol–gel method were devoid of β-MgO, which demonstrates that either kinetic or thermodynamic constraints imposed by the substrate on the phase evolution of the film are responsible for stabilizing β-MgO.

Because it often involves chemical evolution along complex paths, the low-temperature synthesis of solids from liquid precursors is peculiar in its ability to promote the formation of metastable phases. Furthermore, as earlier work has demonstrated [13], there is clear evidence that thin films can sustain otherwise unstable phases because of the structure-directing influence of the substrate upon which they are deposited. Therefore, it seems desirable to investigate the possibility of intentionally selecting, using liquid solution routes, specific metastable polymorphs through templated growth on substrates that have close lattice matching with the target structure. In this paper, we use such methods to stabilize and isolate phase-pure β-MgO at 200°C on polished (001)-oriented MgAl₂O₄ single crystals, the lattice parameter of which differs by only 0.5% from that of β-MgO. The phase purity, structure and orientation of the films are characterized by four-circle X-ray diffraction (XRD) and transmission electron microscopy (TEM), and the chemical composition is monitored by X-ray photoelectron spectroscopy (XPS).

2. Experimental procedure

2.1. Gel deposition and pyrolysis

Details of the sol–gel synthesis and chemical evolution of MgO thin films on Si(1 1 1) substrates is described in detail elsewhere [13]. The same procedures are used in these experiments to deposit MgO thin films on MgAl₂O₄ (0 0 1) substrates, so we give only a cursory description here. Magnesium ethoxide,¹ Mg(OEt)₂ was suspended in absolute ethanol, and was refluxed by adding acetic acid (27:3:1 molar ratio of EtOH:AcOH:Mg(OEt)₂). Water was then added, in a 5:1 molar ratio with Mg(OEt)₂. This precursor solution was spin-cast at 5000 rpm onto a cleaned and polished MgAl₂O₄ (0 0 1) (see footnote 1) substrate.

Thermogravimetric analysis of this precursor indicates that mass loss occurs over two temperature ranges [13]. The first mass loss event, from room temperature to 80°C, is associated with the evaporation of the residual solvent (EtOH). The second mass loss event occurs from 150°C to 200°C and is caused by pyrolysis of an intermediate triclinic magnesium acetate (β-Mg(OAc)₂) phase. To accommodate these mass loss events without undue damage to the structure, films in this study were heated in two stages: (1) 1°C/min to 80°C, followed by a 10-h anneal at that temperature, and (2) 1°C/min from 80°C to 1000°C.

¹ Alfa Æsar, Ward Hill, MA.
200°C, 400°C, 700°C, or 900°C, followed by a 2-h anneal.

X-ray photoelectron spectroscopy (XPS)\(^2\) was used to perform chemical analyses of the films. Ar-ion milling was used to remove adsorbed species and expose the interior of the film. Based on milling rates estimated from independent experiments, the top 60±20 nm (50±15%) of the film was removed prior to analysis.

2.2. Structural and phase analyses

Since the lattice parameter of the crystallized film is nearly equal to that of the substrate, the X-ray diffraction peaks for corresponding lattice planes in the film and the substrate were in close proximity, and special care was taken to distinguish between those diffraction peaks contributed by the substrate—including twins and/or compositional heterogeneities—and those contributed by the film.

The first diffractometer was used to separate and identify diffraction peaks from (001)-type lattice planes of the film and substrate, and to determine their lattice parameters. MoK\(_\alpha\) X-rays\(^3\) were passed through a Ge incident-beam monochromator (0.01° resolution) and then through a slit to expose a 50 µm strip in the center. Both \(\omega\) (rocking curve) and \(\omega−2\theta\) (powder) diffraction scans of the film and substrate were made using this diffractometer.

The second diffractometer was used to investigate diffraction peaks from lattice planes other than (001), and to examine the orientation of the film on its substrate. MoK\(_\alpha\) X-rays\(^4\) were passed through a graphite incident-beam monochromator (0.05° resolution), and then were passed through two orthogonal slits to expose a 50 µm square in the center of the film. The specimen was mounted on a four-circle goniometer to obtain independent diffraction scans on the film and substrate through the three rotational degrees of freedom. Fig. 1 schematically illustrates the four-circle goniometer geometry and the symbol used for each independent rotation axis of the specimen and detector.

For either diffraction setup, the specimen was aligned by maximizing the intensity of the (004) substrate peak. Because of the close proximity of the (004) peaks diffracted from the film and substrate, alignment of the substrate about its (004) peak was used as a reference position about which to scan for the peak generated by (004) film planes. The displacement of the substrate and film (004) peaks indicates a misorientation of the film and substrate. Higher peak intensities from the film were therefore obtained by making slight adjustments in the \(\chi\) and \(\omega\) circles, thereby rendering the incident X-ray beam perpendicular to the (004) film planes. Using this alignment of the film, the \(\chi\) and \(\phi\) angles were systematically scanned for additional film peaks.

For each peak, the following types of diffraction scans were made on the film and substrate: \(\omega\) (rocking curves), \(\omega−2\theta\) (powder scans), \(\phi\) and \(\chi\) (see Fig. 1). For larger values of \(\chi\) (grazing incidence) the X-ray source power and/or the collection time were increased to obtain sufficiently intense signals from the film.

Cross-sectional TEM specimens were prepared by a small-angle cleavage technique. This technique eliminates artifacts and damage—such as surface amorphization, surface mixing and preferential etching—that are induced by conventional ion milling methods. TEM observations were made on a CM-12 microscope, operating at an accelerating voltage of 120 kV.

\(^2\)Physical Electronics PHI 5400 X-ray Photoelectron Spectrometer, Eden Prairie, MN.
\(^3\)RU 200, Rigaku, Japan.
\(^4\)Elliott GX-21, Marconi Elliott Avionics.
3. Results

3.1. Chemical Analysis

Fig. 2 shows X-ray photoelectron spectra of films annealed at 200°C and 400°C. The only signals recorded are those identified with Mg, O and C. The carbon content decreases from ≈14 at% at 200°C to ≈4 at% at 400°C, as residual carbon from the gel is oxidized and removed. Within the detection limit of the instrument (≈1 at%), no other impurities are observed in the films.

A quantitative analysis of the films was made from the integrated peak intensities for each element and the tabulated elemental sensitivity factors. Because the baseline for each peak is not precisely defined, there are random uncertainties in these kinds of calculations which likely amount to <5 at% for a given element, depending on its sensitivity factor. However, even taking these uncertainties into account, the O:Mg ratio—2.58 at 200°C and 1.67 at 400°C—is significantly higher than one would expect from stoichiometry. Ion milling of metal oxides and nitrides can preferentially etch the anionic component, but such an effect cannot explain these results. Instead, it is likely that the residual carbon detected in the films reacts to form MgCO$_3$, which is thermodynamically stable relative to MgO at these low temperatures. If one assumes that (i) all the carbon remaining at 400°C is in the form of MgCO$_3$, and that (ii) the remainder of the film is stoichiometric MgO, then 4 ± 2 at% carbon corresponds to 0.1 ± 0.07 mole fraction of MgCO$_3$ and a ratio O:Mg of 1.18 ± 0.11. Therefore, even a relatively small amount of C in this form can yield fairly large O:Mg ratios. Furthermore, simple linear extrapolation of the XPS data to 0% carbon would imply O:Mg = 1.3, which is more consistent with values reported by other investigators in the synthesis of bulk MgO powders using similar procedures [23], especially considering the measurement uncertainties already described.

3.2. Structural Analysis

Fig. 3(A) shows the XRD powder scan for the film (heated to 200°C) at $\phi = 0^\circ$ and $\chi = 0^\circ$. The 20 value for this peak coincides with that for the published value [25] of (0 0 4) peak of $\beta$-MgO and, since the film consists solely of Mg, O and C, it is therefore indexed as such. (It will be shown later the other peaks of $\beta$-MgO were also observed at the expected 20, $\omega$, $\phi$, and $\chi$ values.) The rocking curve for the (0 0 4) film peak (Fig. 3(B)) shows a full-width at half-maximum (FWHM) of only 0.03°. This low value implies a (0 0 1) texture in the films that is sharper, by over an order of magnitude, than any MgO film textures reported in the literature to date, regardless of the substrate or deposition method used [4,5,12,13]. However, the significance of this result should be tempered by the fact that a closely lattice-matched substrate was intentionally used here, whereas previous studies used either Si or GaAs substrates, each of which has considerably greater misfit with $\alpha$-MgO.

Five $\beta$-MgO X-ray peaks were sought and found by systematic scans over different regions of reciprocal space. The results are compiled in Table 1. Fig. 4 shows a $\chi$ scan around the $\beta$-MgO (4 4 4) peak. The substrate and the film peaks are separated by ≈1°. Furthermore, as expected for cubic crystals, the (4 4 4) peak for the film...
is 54.7° away from the (004) peak for \( \chi = -90° \) (see Table 1).

Fig. 5(A) shows a \( \phi \) scan for the (444) peak of \( \beta \)-MgO. The 0.2° FWHM of the \( \phi \) scan indicates excellent in-plane texture. The texture is further confirmed by the sharp rocking curve (see Fig. 5(B)) about the (444) peak. The (444) peak, which in a single crystal should be displaced by 90° from the (444) peak in the \( \phi \) circle, was indeed observed at this position when all the other angles were held constant (see Fig. 5(C)).

Fig. 6 shows a reciprocal space map of the specimen in the vicinity of the (044) peaks of the film and substrate. By using the MgAl\(_2\)O\(_4\) unit cell as reference and aligning the beam to the substrate, the maximum intensity for the (044) film peak appears at a value of \( h = 0.02 \pm 0.02, k = 3.99 \pm 0.02 \) and \( l = 3.90 \pm 0.02 \). Again, the 2\( \theta \) value for the film peak coincides with that for the (044) peak of \( \beta \)-MgO [25]. Furthermore, the (044) peak appears \( \approx 45° \) away from the (004) peak—as expected for a cubic single crystal.

Powder scans and rocking curves performed on films heated to 400°C produced results that are identical to those just described for films annealed at 200°C. Therefore, we do not reproduce the data here. However, taken together the XRD data collected on these MgO films are persuasive evidence that the films are single-phase \( \beta \)-MgO.

### Table 1

<table>
<thead>
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<th>((h k l))</th>
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<th>(\phi) (deg)</th>
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<td>20.47</td>
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Fig. 3. (A) \(\omega-2\theta\) scan and (B) \(\omega\) scan (rocking curve) for (004) peak of the \( \beta \)-MgO film.

Fig. 4. \( \chi \) scan for the (444) peak. Film and the substrate are misaligned by \( \approx 1° \), and the (444) peak for the film is displaced by 54.7° in the \( \chi \) circle from the (004) peak.
with individual grains that are highly aligned both with the substrate and with each other.

### 3.3. Microstructure

Fig. 7 shows a contact mode AFM image of the film surface annealed at 400°C. The film is approximately 100–150 nm thick, and three pores of ≈2 μm diameter are visible in the randomly selected 50 μm × 50 μm area. Otherwise the film appears to be fairly dense and uniform. The larger pores were probably formed during the removal of organics; an even more carefully optimized heating schedule may minimize the pore formation. Furthermore, no grain boundary features are visible in the 50 μm × 50 μm area, as they are for polycrystalline MgO films deposited on Si by other methods [10,12,24]. Taken together with the XRD evidence, the AFM data indicate that the film is composed of highly oriented grains separated by
low-angle, low-energy grain boundaries. The average grain size cannot be determined from these figures, although a previous study by the authors of MgO synthesized on Si/SiO$_2$ by the same methods suggests that the average grain diameter can be as low as 50–100 nm [13].

A plan-view, bright-field TEM image of a $\beta$-MgO (001) film grown at 200°C is shown in Fig. 8(A), along with a $\langle 001 \rangle$ selected-area electron diffraction (SAED) pattern obtained from the region. The dark gray contours are due to bending of thin regions of the TEM specimen. No grain boundaries were observed in either of two
TEM specimens, which is consistent with the AFM image in Fig. 7. A region of bare substrate, presumably coincident with one of the pinhole defects indicated in Fig. 7, was found during TEM observation (Fig. 8(B)). This region produces an \( \langle 001 \rangle \) SAED pattern that is similar to that of the film, which indicates that the film domains and the substrate have essentially the same crystallographic orientation. Fig. 8(C) and (D) are two X-ray energy dispersive spectra (EDS) obtained from the regions shown in Fig. 8(A) and (B), respectively. The substrate contributes a small Al signal in Fig. 8(C), and carbon is again indicated as previously detected by XPS. It should be mentioned that \( \langle 001 \rangle \) SAED patterns obtained from the regions covering both film and substrate do not show spot splitting due to their slight difference in lattice parameters, in agreement with XRD results.

4. Discussion

The XRD and TEM data of the previous section show that the \( \beta \)-MgO films formed on MgAl\(_2\)O\(_4\) (0 0 1) substrates by this solution processing method are phase-pure \( \beta \)-MgO, and that the film exhibits domains that are well aligned both with each other and with the substrate. Of the eight diffraction peaks previously reported for \( \beta \)-MgO [25], five were sought—three of which were noncoplanar—and were found at precisely the film orientations in 3D space that would be predicted for a single crystal of that structure. Furthermore, we may be relatively certain that the film crystallinity and phase purity extend over at least a 3 mm \( \times \) 3 mm area, because measurements made on separate diffractometers, for which the beam position likely varied within this range, are selfconsistent. No attempts were made to locate the other three peaks catalogued in Ref. [25].

Freund [22] hypothesized from XRD measurements on bulk powders and from IR spectra that the crystal structure of \( \beta \)-MgO is spinel like with Mg\(^{2+}\) cations in low-coordinated states. Other than this hypothesis, little is known or has been speculated about the crystal structure of this phase. The availability of diffraction signals from three non-coplanar lattice planes in these \( \beta \)-MgO films offer the opportunity to determine its crystal system and to calculate the lattice parameters and interaxial angles for the \( \beta \)-MgO unit cell. Because the bulk lattice parameter and point group of MgAl\(_2\)O\(_3\) is well known, and because independent rocking curves from substrate and film indicate that the film has no measurable tilt or twist with respect to the substrate, we may readily use the apparent position of the \( \beta \)-MgO (0 4 4) peak in Fig. 6 to show that \( a = b = 8.08 \pm 0.01 \) Å, and \( c = 8.29 \pm 0.01 \) Å. The angular displacements measured between the (0 0 4), (0 4 4), and (4 4 4) and (4 4 4) indicate that the interaxial angles for the \( \beta \)-MgO structure are all 90° \( \pm \) 0.1°. Furthermore, because the bulk lattice parameter for MgAl\(_2\)O\(_4\) is 8.08 Å, these measurements on the \( \beta \)-MgO unit cell confirm that the film is essentially an epitaxially oriented single crystal with \( \beta \)-MgO\([100] \parallel \)MgAl\(_2\)O\(_4\) [1 0 0] and \( \beta \)-MgO\([110] \parallel \)MgAl\(_2\)O\(_4\)[0 1 0].

XRD measurements on \( \beta \)-MgO powders have indicated that its bulk lattice parameter is 8.12 Å, (unit cell volume = 535.4 Å\(^3\)) [22]. The in-plane lattice parameters (\( a \) and \( b \)) measured from the films are smaller, and the \( c \) parameter larger, than this reported value. However, the volume of the unit cell in the films is 541.2 \( \pm \) 2 Å\(^3\), which exceeds the published value by only 1%. Some of the volume difference, especially at low temperatures, is probably caused by interstitial carbon. The film therefore appears to be under a uniform 0.5% biaxial compression due to lattice mismatch in the (0 0 1) planes. Furthermore, judging from the similarity in unit cell volumes,\(^5\) the film is essentially fully relaxed in the [0 0 1] direction.

The question of the precise mechanism by which \( \beta \)-MgO is selected in these films has not been answered unequivocally. However, we now may be certain about several facts concerning the mechanism(s). First, the spinel-like structure is not formed as a consequence of an impurity, like Al or Fe, that can cause formation of an actual spinel compound. A previously reported spinel-like structure of MgO [26], formed from dehydration of Mg(OH)\(_2\), was actually later found to be

\(^5\) Some of the volume difference, especially at low temperatures, is probably caused by interstitial carbon.
MgFe$_2$O$_4$ formed by accidental introduction of iron impurities during the process. However, Figs. 2 and 8(C) indicate that no such impurities are present in these MgO films. And, although the films contain as much as 14 at% carbon at 200°C, carbon is also present in roughly the same amount in MgO particles that form $\alpha$-MgO from the same solution at the same temperature.

The formation of $\beta$-MgO requires a structure-directing constraint imposed by the underlying substrate during pyrolysis of the gel precursor: powders formed from the same solution by exactly the same procedure yield phase-pure $\alpha$-MgO after pyrolysis at 200°C. In a previous paper, we have described in some detail, the chemical and structural evolution of the liquid precursor during evaporation and pyrolysis [13]. In that paper, it is shown that, on Si/SiO$_x$ substrates, deposition of the same solution yields a highly oriented $\langle 110 \rangle$ $\beta$-Mg(OAc)$_2$ phase at room temperature. We speculated that this acetate phase may template the subsequent crystallization of $\beta$-MgO [13]. However, on these MgAl$_2$O$_4$ (001) substrates, low-angle XRD data do not indicate strong texturing of the $\beta$-Mg(OAc)$_2$, even for films heated to 150°C. Therefore, formation and growth of $\beta$-MgO is not predicated by strong orientation of $\beta$-Mg(OAc)$_2$ prior to pyrolysis.

Conditions for forming $\beta$-MgO appear to be relatively insensitive to the type of substrate on which it is grown (Si(111)/SiO$_x$ or MgAl$_2$O$_4$ (001)), although employing a closely lattice-matched substrate does improve the volume fraction of $\beta$-MgO observed. Therefore, the substrate may play both a thermodynamic and a kinetic role in the formation of $\beta$-MgO. That is, it may give the interface between the substrate and $\beta$-MgO a lower free energy density than that for the substrate/$\alpha$-MgO interface, as is almost certainly the case for the epitaxial interface between MgAl$_2$O$_4$ (001) and $\beta$-MgO. But the substrate also may kinetically inhibit the large structural rearrangement necessary to crystallize $\alpha$-MgO from the relatively open crystal structure of the organic film, especially since $\alpha$-MgO is probably denser than $\beta$-MgO. Such a kinetic constraint is probably a stabilizing influence for $\beta$-MgO on Si(111)/SiO$_x$ substrates [13]. Further clarification of all these issues, as well as a determination of the point group and space group of $\beta$-MgO, are expected from additional plan-view and cross-sectional TEM investigations that are currently under way.

We have observed that the crystal quality of the film is strongly tied to that of the substrate. Note that in Fig. 8(B) the MgAl$_2$O$_4$ substrate appears to have a fairly dense dislocation network, yet the film is well-formed and textured. However, it is difficult to grow and polish pure spinel substrates; the one represented in Fig. 8(B) had the most crystalline and defect-free surface of any we observed. Not surprisingly, films grown on these latter substrates under the same processing conditions formed nanocrystalline $\beta$-MgO domains with little or no texture. The untexured films were not successfully analyzed by XRD because of their much lower diffracted intensities.

$\beta$-MgO appears to remain stable at temperatures up to 1000°C. In a previous study [13], we demonstrated that $\beta$-MgO formed as a film on Si/SiO$_x$ was retained at 1000°C, despite the fact that, on that substrate, $\beta$-MgO coexisted with an approximately equal amount of $\alpha$-MgO. Although we did not specifically study the high-temperature behavior of $\beta$-MgO on spinel substrates, we may reasonably speculate that it is likely to be at least as stable as that on Si/SiO$_x$ substrates because of the close lattice matching and the fact that no $\alpha$-MgO is available to seed the $\beta \rightarrow \alpha$ conversion.

5. Conclusions

$\beta$-MgO has been successfully isolated in highly textured thin films grown on MgAl$_2$O$_4$ (001) substrates. The film is composed of grains that are all oriented with respect to the substrate and with each other. Structure selection was achieved by using a close film/substrate lattice matching and exploiting the constraints imposed by the substrate during the formation and growth of both an intermediate phase of magnesium acetate phase and, after pyrolysis, of the oxide phase.

The ability to tailor not only the microstructure and orientation, but also, at least in some situations, the crystal structure of a thin film could
have significant implications for creating novel devices. It is possible that β-MgO thin films could exhibit unique electrical, optical, or thermal properties, although we have not yet made such measurements. But even in the absence of such novel properties, the larger lattice parameter of β-MgO may make it suitable as a buffer layer for subsequent textured growth of other oxide phases. Finally, we have no reason to expect that the phase selection behavior found in this paper is unique to MgO. It may well be possible in other thin film systems to select otherwise unstable phases using the same ideas presented here.

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References

[25] Joint Committee on Powder Diffraction Standards, Files 30-794 (spinel-like MgO) and 21-1152 (MgAl2O4) 1996.