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ZrB₂ thin films grown by high power impulse magnetron sputtering (HiPIMS) from a compound target

Mattias Samuelsson^{a,b}, Jens Jensen^a, Ulf Helmersson^a, Lars Hultman^a, and Hans Högberg^{a,*}

^a Department of Physics, Chemistry, and Biology (IFM) Linköping University, SE-581 83
Linköping, Sweden.

^b Impact Coatings AB, Westmansgatan 29, SE-582 16 Linköping, Sweden

*Corresponding author. Electronic mail: hans.hogberg@liu.se

Abstract

ZrB₂ thin films were grown on Si by high power impulse magnetron sputtering (HiPIMS) from a compound target in an industrial deposition system. By keeping a constant average power while modifying the HiPIMS pulse repetition frequency, the pulse peak current and thereby the degree of ionisation was varied. The films were characterised using X-ray diffraction techniques, scanning electron microscopy, time-of-flight elastic recoil detection analysis, and four-point probe measurements. It was found that the composition of the films matched closely that of the target material, and the films were low in contamination. The films were crystalline with a strong (000n) preferred orientation, and that the residual stress could be adjusted, from tensile to compressive, by increasing the degree of ionisation. The film morphology appeared dense, with a smooth surface, and the resistivity was found to range from 180 to 250 $\mu\Omega\text{cm}$ with no clear dependence on frequency in the investigated parameter range.

Keywords:

ZrB₂, thin films, HiPIMS, HPPMS, compound target, industrial scale deposition system, crystalline films,

Introduction

Zirconium diboride (ZrB_2) is a refractory material that exhibits interesting properties, such as high hardness, high melting point, and a low electrical resistivity. Owing to its properties, the material has potential for serving as diffusion barrier in microelectronics [1,2]. Ceramic films, e.g., oxides, nitrides, carbides, and borides can be grown by magnetron sputtering techniques from a compound target of the desired composition. Although the potential stability issues of a corresponding reactive process are avoided, sputtering from compound targets inherently introduces complexity to the process owing to differences in physical properties of the two elemental constituents of the target material with respect to, e.g., ejection angle distribution of sputtered material [3], preferential sputtering of one species over the other in the film [4], and gas phase scattering [3].

The complexity of depositing ZrB_2 from compound sources using magnetron sputtering techniques is reported in the literature. For example, composition in the films deviating – at times very much – from that of the source has been reported [4–9]. Moreover, the material system efficiently absorbs, and is strongly influenced by contaminations [1,2,4,7,10,11], which may affect the electrical properties [12]. While the bulk resistivity of ZrB_2 is about $10 \mu\Omega\text{cm}$ [13], thin film ZrB_2 grown by magnetron sputtering techniques thus often shows significantly higher resistivity values than for bulk, ranging from 160 to $440 \mu\Omega\text{cm}$ [1,8,11,12]. Annealing experiments causing recrystallisation has, however, been shown to decrease the film resistivity to $\sim 25 \mu\Omega\text{cm}$ [1,14]. It has been shown that elevated deposition temperatures or intense ion irradiation of the film during growth enhances the crystalline quality and alters the preferred orientation [15,16]. Both contaminations and crystallinity may have implications for the electrical properties of the thin film [1,12] through oxidation or grain boundary scattering. A high ion irradiation during ZrB_2 growth has been shown to improve crystallinity and reduce film porosity [15], and thereby post-deposition

contaminations. Increasing the ion flux towards the growing film could therefore be a strategy for improving the electrical properties of the sputter deposited material.

High power impulse magnetron sputtering (HiPIMS) is a lately developed thin film deposition technique [17–21]. As compared to dc-magnetron sputtering (DCMS), HiPIMS offers increased ion assistance during film growth, which can be beneficial for the resulting film properties. However, the deposition rate using HiPIMS is often reported to be significantly lower when compared to DCMS for the same average power [18]. This reduction in deposition rate has been proposed to be mainly an effect of back-attraction of ionised species to the target [22], and has been seen to correlate to the degree of ionisation and self-sputtering yield of the target material type [18,23]. Up to the present, the HiPIMS technique has mainly been demonstrated for sputtering from a metallic target material and for some reactive sputtering processes of nitride, oxide, and carbide thin films [24–26]. Employing HiPIMS for deposition from a compound source may offer a denser morphology through the intense ion irradiation of the growing film, which can allow less post deposition incorporation of contaminants [26]. However, the introduction of HiPIMS may further increase the process complexity, since the trajectory for ionised sputtered material leaving the target differs for HiPIMS as compared to e.g. DCMS [27,28], and any preferential resputtering effects may be more pronounced owing to the high ion irradiation of the growing film. Reports on non-reactive HiPIMS from compound targets are scarcely found, although examples exist. Alami et al. demonstrated HiPIMS from a Ti_3SiC_2 compound target [29]. However, the authors of the aforementioned study were not successful in attaining the composition or the phase corresponding to the target composition, possibly due to the complexity of the chosen material comprising three elements, and the nano-laminate Ti_3SiC_2 MAX phase structure with hexagonal symmetry being highly anisotropic ($c/a \approx 6$) [30]. Non-reactive HiPIMS from a

binary compound target, where the nominal composition and the intended phases are obtained (or retained, in the case of a target of the desired phase), remains to be demonstrated.

The aim of the present study is to investigate the deposition of a binary compound material using HiPIMS, as well as the influence of degree of ionisation during the process on film morphology and electrical properties. This was realised by depositing films from a ZrB_2 compound target in an industrial deposition system. While keeping the average power constant for different pulse repetition frequencies, the HiPIMS peak power and current, and thereby the degree of ionisation in the deposition flux, were varied for the different experiments.

Experimental details

The depositions were performed in a commercial industrial high vacuum (HV) coating system (CemeCon CC800/9). Prior to depositions the chamber was evacuated to a base pressure below 3×10^{-4} Pa. The Si(100) substrates were cleaned in isopropanol prior to being mounted at a distance of 7 cm in front of a magnetron sputtering source equipped with a ZrB_2 compound target with a specified purity of 99.5%, excluding Hf. The cathode ($50 \times 8.8 \text{ cm}^2$) was operated in unipolar HiPIMS mode at a pulse width of 150 μs , and at pulse repetition frequencies of 300, 500, 700, and 900 Hz. For all frequencies, the average power was kept constant at 3 kW, and the deposition times were 480 s. Owing to depletion of the capacitor bank of the power supply the cathode voltage was significantly diminished during the duration of the pulse. During depositions, the process gas (Ar) pressure was 1.1 Pa. While no substrate rotation or external heating was employed during depositions, a dc bias voltage of -80 V was applied to the substrates. In order to investigate the influence of ion assistance on the film growth, depositions at 900 Hz at a lower process pressure (0.75 Pa) and lower

substrate bias (-60 V dc) were performed. Prior to each deposition the target was sputtered-cleaned in DCMS mode behind a shutter. Before the shutter was opened, the HiPIMS mode was initiated and the power was gradually increased to 3 kW, ensuring a stable operation and reducing the occurrence of arcs. During the processes, the voltage and current waveforms at the cathode, as well as the substrate table, were recorded from an oscilloscope (Tektronix DPO4054).

The crystal structure of the coatings was characterised using X-ray diffraction (XRD) operated in Bragg-Brentano and $\sin^2\psi$ configurations [31], equipped with a Cu K α source. Film morphology and thickness was investigated by cross-sectional scanning electron microscopy (SEM, LEO 1550 Gemini). Images were obtained at an acceleration voltage of 10 kV. Elemental composition (i.e. stoichiometry and contamination occurrence) was determined using time-of-flight elastic recoil detection analysis (ToF-ERDA), using 36 MeV $^{127}\text{I}^{8+}$ with a beam incidence angle of 67.5° to the sample surface normal, and a recoil angle of 45° [32]. The electrical resistivity of the films was calculated from the measured sheath resistivity, obtained from four point probe measurements (Jendel Engineering) and the film thickness.

Results and Discussion

The peak values for cathode current and substrate current, as well as the deposition rate as functions of pulse repetition frequency during the HiPIMS depositions are shown in Figure 1. Since the average power was kept constant for all experiments, decreased peak values are seen as the frequency is increased. As the frequency is increased from 300 to 900 Hz and the cathode peak current decreases from 520 to 120 A, the bias current to the substrate is reduced from 13 to 4 A, and the deposition rate increases from 45 to 80 nm/min (Figure 1). It has previously been suggested that with decreasing cathode peak current values, the degree of

ionisation of the metal in the plasma [33], as well as the ion energy distribution function is shifted towards lower values [34]. With a lower degree of ionisation of sputtered material, the back attraction is reduced, and as a consequence the deposition rate is increased [34–36]. Our observations are thus consistent with the literature, suggesting that also for the present target material, the degree of ionisation, and the number of ions reaching the substrate during each pulse, are decreased as the cathode peak current decreases.

Figure 1. HiPIMS process parameters and deposition rates for the different pulse repetition frequencies used. (Colour online)

The elemental composition of the coatings as determined by ToF-ERDA versus pulse repetition frequency is plotted in Figure 2. It is seen that all the investigated films have equal composition within measurement accuracy. All films are also deficient in B as compared by the nominal composition of the sputtering target (66.7 at.%), giving a B/Zr ratio of the films is ranging from 1.82 to 1.98, which nevertheless is close to stoichiometry. Deviation of film

composition from that of the target when sputtering from ZrB_2 compound sources is often observed, and several explanations for this exist as outlined in the introduction section. In the present case, the overall slightly low B content is possibly due to preferential resputtering of B in the film owing to the ion bombardment, as suggested by Mitterer et al. [4], and generally described by Berg et al. [37]. Considering the very narrow (< 1 at.%) phase region for ZrB_2 [38], it is unlikely that the excess Zr is found in the ZrB_2 lattice, but could rather be precipitating or segregating to the ZrB_2 grain boundaries, similar as described for excess of B in the report on TiB_2 by Mayrhofer et al. [39]. The contamination elements are predominantly O and Ar with concentrations below 2 at.%, respectively. This should be compared to the values reported in the literature [1,4,7,8,10,11], that at times reach levels above 30 at.%. The values presented here should therefore be considered very low – in particular when considering that the growth was carried out in an industrial HV-system, using no external heating. No significant influence on composition or contamination levels from reduced bias voltage (from -80 V to -60 V) or lower process pressure for the 900 Hz films could be observed. For the control DCMS films deposited using the same sputtering source and corresponding experimental conditions, a similar B/Zr ratio of ~ 2.05 was typically found, as well as matching or even lower levels of contaminations.

XRD θ -2 θ diffractograms of the coatings are shown in Figure 3. It is seen that all coatings are crystalline, and all film peaks can be attributed to ZrB_2 . All the coatings exhibit a clear (0001) texturing, which can be attributed to ion irradiation of the films during growth that promotes adatom mobility [16]. Diffraction pattern analysis showed that as the pulse repetition frequency is increased, the maximum intensity of the reflections increase while the peak full width half maximum (FWHM) of the peaks decreases from ~ 0.58 to $\sim 0.22^\circ$. Alami et al. [35] observed a similar trend for the crystallinity of CrN, and through transmission electron microscopy and mass-spectrometer investigations they were able to deduce that the film

Figure 2. Elemental composition, attained from ToF-ERDA measurements, of the investigated films vs. the different pulse repetition frequencies used. The dashed lines represent the nominal B and Zr composition of the sputtering target. Error bars are smaller than the symbol sizes.

structure was altered from polycrystalline to nanocrystalline as the ion content of the deposition flux was increased, i.e. a high degree of ionisation introduces surface defects which promotes renucleation and leads to smaller grain sizes. In the present case, the deposition rate and substrate bias current peak values (Fig. 1) indicate that the degree of ionisation varies with pulse repetition frequency, in that a higher frequency, yielding a lower cathode peak current, also gives a lower degree of ionisation. The maximum peak intensity, and FWHM trends observed in Fig 3 can thus be understood as a consequence of decreased ion irradiation as the frequency is increased. It is also likely that to some extent, the increased XRD intensity and decreased FWHM can be explained by the higher deposition rate (Fig. 1)

yielding thicker films and thereby a larger XRD interaction volume, and larger grains, respectively.

Figure 3. Diffractograms of representative coatings for different pulse repetition frequencies as attained from θ -2 θ XRD scans. The peak at $\sim 32^\circ$ is the Si (200) reflection from the substrate while the peak at $\sim 37^\circ$ seen for the coating deposited at 900 Hz is a detector artifact

It is also seen from Fig. 3, that the ZrB_2 (000n) reflections, except for the 500 Hz film are shifted with respect to the unstrained bulk value [40]. For the lowest frequency (300 Hz), the film peaks exhibit a shift to lower angles, consistent with compressive stress, and as the frequency is increased, the peak position continuously shifts to angles higher than the literature value, indicating tensile stress. Owing to the strong (000n) texture of the films, $\sin^2\psi$ stress measurements showed to be ineffective. However, the trend of the ZrB_2 (000n)

reflection deviations from the unstrained literature value can be understood in terms of intensity of ion irradiation. Firstly, since the ZrB_2 phase permits very little deviations from stoichiometry, we must assume that any deviations in XRD peak positions are derived from residual stress in the films. Stress in thin films grown by magnetron sputtering is commonly accepted to be the sum of two independent components, namely a compressive one due to ion peening (in the presence of energetic ion and neutral bombardment during growth), and a tensile component with its origin in attractive forces over the grain boundaries [41]. The tensile component has also been shown to increase with film thickness due to increasing grain boundary porosity as the grain sizes increase. (There is also a stress component due to discrepancy in the thermal expansion between the substrate and film which is disregarded here since the films were grown without intentional heating.) At low frequencies, with a high density flux of ions towards the substrate (Fig. 1), the renucleation and ion peening are more pronounced, yielding smaller grains (indicated by increasing FWHM and Fig. 4 (a)) and permit little grain boundary porosity. This means that the compressive stress component is promoted and the tensile component suppressed. As the frequency is increased, and the ion ratio of the flux towards the substrate is reduced, the ion peening events are fewer. Moreover, the adatom diffusion distance is slightly lower, also because of the higher deposition rate, and the grain boundaries could be expected to be less dense. A lower ionisation should therefore yield an overall tensile stress. In effect, this means that within certain limits, the residual stress of the ZrB_2 films can be adjusted by the degree of ionisation in the deposition flux (in the present case by altering HiPIMS frequency).

Figure 4. Cross sectional SEM micrographs of films grown using different pulse repetition frequencies used (a-c), as well as for a DCMS films grown under equivalent conditions (d).

Figure 4 shows cross sectional SEM micrographs from the respective HiPIMS coatings (panels (a) to (c)), as well as a DCMS coating (panel d) grown under comparable conditions. It can be seen that all the HiPIMS coatings appear dense and have a smooth surface, an appearance often reported for HiPIMS films [26]. For the highest frequencies used in the present study (700 and 900 Hz), a narrow, and columnar appearance can be discerned. This observation is consistent with comparatively lower ion ratio in the deposition flux and higher deposition rate, effectively reducing adatom mobility compared to the lower frequencies used. Although differences in morphology are seen between the HiPIMS coatings as the frequency increases, these are minor when put in relation to a typical DCMS ZrB_2 coating grown under similar conditions (Fig. 4 (d)), that exhibits a pronounced columnar appearance.

The electrical resistivity values for the as-deposited films, as obtained from four point probe measurements, are plotted versus pulse repetition frequency in Figure 5. No distinct dependence of frequency (i.e. ionisation degree of the deposition flux), process pressure, or bias voltage on the resistivity values can be observed for the films. Albeit the films being

grown under high vacuum conditions and at ambient temperature, all resistivity values are found to be in the range of 180 to 250 $\mu\Omega\text{cm}$. Thus, the resistivity values do not differ much from our control depositions using DCMS (typically ranging from 150 to 170 $\mu\Omega\text{cm}$), or values obtained using other sputtering techniques found in the literature [1,8,12], and show that satisfactory electrical properties can be obtained also under industrial conditions. While the values are more than 20 times larger than the tabulated value for bulk ZrB_2 [11], previous studies have shown that annealing such films to $>1100^\circ\text{C}$ recrystallises the films and resistivity values comparable to bulk ZrB_2 can be obtained [1].

Figure 5. The electrical resistivity of the investigated films for the different pulse repetition frequencies used.

Conclusions

The results show that it is possible to grow near stoichiometric, crystalline ZrB_2 films from a compound target using HiPIMS. The films are strongly (0001) textured, which is consistent with a high adatom mobility during growth. By tuning the HiPIMS process in terms of the degree of ionisation in the deposition flux, the residual stress in the films could be adjusted, ranging from tensile to compressive with increasing degree of ionisation. Contamination levels in the films are low, and the morphological appearance is dense with a smooth surface. The electrical resistivity of the films ranges from 180 to 250 $\mu\Omega\text{cm}$, which is similar to ZrB_2 films grown using other less ionised sputtering techniques.

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References

- [1] J.R. Shappirio, J.J. Finnegan, R.A. Lux, J. Vac. Sci. Technol., B 4 (1986) 1409.
- [2] T.N. Oder, P. Martin, A.V. Adediji, T. Isaacs-Smith, J.R. Williams, J. Electron. Mater. 36 (2007) 805-811.
- [3] J. Neidhardt, S. Mraz, J.M. Schneider, E. Strub, W. Bohne, B. Liedke, W. Möller, C. Mitterer, J. Appl. Phys. 104 (2008) 063304.
- [4] C. Mitterer, J. Komenda-Stallmaier, P. Losbichler, P. Schmölz, W.S.M. Werner, H. Störi, Vacuum 46 (1995) 1281-1294.
- [5] D.J. Li, J. Yang, X.H. Zhang, M. Cao, J. Vac. Sci. Technol., B 25 (2007) L11.
- [6] L. Voss, S.J. Pearton, F. Ren, I.I. Kravchenko, Appl. Surf. Sci. 253 (2006) 1934-1938.
- [7] J. Wright, R. Khanna, K. Ramani, V. Cranciun, R. Singh, D.P. Norton, S.J. Pearton, F. Ren, I.I. Kravchenko, Appl. Surf. Sci. 253 (2006) 2465-2469.
- [8] M. Guziewicz, A. Piotrowska, E. Kaminska, K. Golaszewska, A. Turos, E. Mizera, A. Winiarski, J. Szade, Solid-State Electron. 43 (1999) 1055-1061.
- [9] X. Zou, *Oxidation Behavior of RF Sputter Deposited Zirconium Diboride Thin Films*, master's theses, San Jose State University (1995).
- [10] R. Khanna, K. Ramani, V. Cracium, R. Singh, S.J. Pearton, F. Ren, I.I. Kravchenko, Appl. Surf. Sci. 253 (2006) 2315-2319.
- [11] M.B. Takeyama, A. Noya, Y. Nakadai, S. Kambara, M. Hatanaka, Y. Hayasaka, E. Aoyagi, H. Machida, K. Masu, Appl. Surf. Sci. 256 (2009) 1222-1226.

- [12] U.K. Chakrabarti, H. Barz, W.C. Dautremont-Smith, J.W. Lee, T.Y. Kometani, J. Vac. Sci. Technol., A 5 (1987) 196.
- [13] M. Rahman, C.C. Wang, W. Chen, S.A. Akbar, C. Mroz, J. Am. Ceram. Soc. 78 (1995) 1380-82.
- [14] R. Khanna, S. Pearton, F. Ren, I.I. Kravchenko, Appl. Surf. Sci. 253 (2006) 2340-2344.
- [15] E. Brandstetter, C. Mitterer, R. Ebner, Thin Solid Films 201 (1991) 123-135.
- [16] E. Kelesoglu, C. Mitterer, M.K. Kazmanli, M. Ürgen, Surf. Coat. Technol. 116-119 (1999) 133-140.
- [17] V. Kouznetsov, K. Macak, J.M. Schneider, U. Helmersson, I. Petrov, Surf. Coat. Technol. 122 (1999) 290-293.
- [18] U. Helmersson, M. Lattemann, J. Bohlmark, A. Ehasarian, J. Gudmundsson, Thin Solid Films 513 (2006) 1-24. [19]
- [19] K. Sarakinos, J. Alami, S. Konstantinidis, Surf. Coat. Technol. 204 (2010) 1661-1684.
- [20] J.T. Gudmundsson, N. Brenning, D. Lundin, U. Helmersson, J. Vac. Sci. Technol., A 30 (2012) 030801-1.
- [21] D. Lundin, K. Sarakinos, J. Mater. Res. 27 (2012) 780-792.
- [22] D.J. Christie, J. Vac. Sci. Technol., A 23 (2005) 330.
- [23] M. Samuelsson, D. Lundin, J. Jensen, M.A. Raadu, J.T. Gudmundsson, U. Helmersson, Surf. Coat. Technol. 205 (2010) 591-596.

- [24] A. Ehasarian, W.-D. Münz, L. Hultman, U. Helmersson, I. Petrov, *Surf. Coat. Technol.* 163-164 (2003) 267-272.
- [25] E. Wallin, T.I. Selinder, M. Elfving, U. Helmersson, *Europhys. Lett.* 82 (2008) 36002.
- [26] M. Samuelsson, K. Sarakinos, H. Högberg, E. Lewin, U. Jansson, B. Wälivaara, H. Ljungcrantz, U. Helmersson, *Surf. Coat. Technol.* 206 (2012) 2396-2402.
- [27] D. Lundin, P. Larsson, E. Wallin, M. Lattemann, N. Brenning, U. Helmersson, *Plasma Sources Sci. Technol.* 17 (2008) 035021.
- [28] W.P. Leroy, S. Konstantinidis, S. Mahieu, R. Snyders, D. Depla, *J. Phys. D: Appl. Phys.* 44 (2011) 115201.
- [29] J. Alami, P. Eklund, J. Emmerlich, O. Wilhelmsson, U. Jansson, H. Högberg, L. Hultman, U. Helmersson, *Thin Solid Films* 515 (2006) 1731-1736.
- [30] P. Eklund, M. Beckers, U. Jansson, H. Högberg, L. Hultman, *Thin Solid Films* 518 (2010) 1851-1878.
- [31] M. Birkholz, *Thin Film Analysis by X-ray Scattering*, Wiley-WCH, Weinheim, Germany (2006).
- [32] J. Jensen, D. Martin, A. Surpi, T. Kubart, *Nucl. Instrum. Methods Phys. Res., Sect. B* 268 (2010) 1893-1898.
- [33] J. Bohlmark, J. Alami, C. Christou, A. P. Ehasarian, U. Helmersson, *J. Vac. Sci. Technol., A* 23 (2005) 18

- [34] M. Aiempanakit, U. Helmersson, A. Aijaz, P. Larsson, R. Magnusson, J. Jensen, T. Kubart, *Surf. Coat. Technol.* 205 (2011) 4828-4831.
- [35] J. Alami, K. Sarakinos, F. Uslu, M. Wuttig, *J. Phys. D: Appl. Phys.* 42 (2009) 015304.
- [36] M. Samuelsson, D. Lundin, K. Sarakinos, F. Björefors, B. Wälivaara, H. Ljungcrantz, U. Helmersson, *J. Vac. Sci. Technol., A* 30 (2012) 1-5.
- [37] S. Berg, I.V. Katardjiev, *J. Vac. Sci. Technol. A* 17 (1999) 1916.
- [38] T.B. Massalski, J.L. Murray, L.H. Bennett, H. Baker, *Binary Alloy Phase Diagrams*, American Society for Metals, Metals Park, Ohio (1986).
- [39] P.H. Mayrhofer, C. Mitterer, J.G. Wen, J.E. Greene, I. Petrov, *Appl. Phys. Lett.* 86 (2005) 131909.
- [40] PDF Number: 00-034-0423, International Centre for Diffraction Data.
- [41] G.C.A.M. Janssen, *Thin Solid Films* 515 (2007) 6654-6664.