Effects of Process Parameter Variations on the Removal Rate in Chemical Mechanical Polishing of 4H-SiC

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The effects of temperature, slurry pH, applied pressure, and polishing rotation rate on the material removal rate during chemical mechanical polishing (CMP) of 4H-silicon carbide wafers using colloidal silica slurry and polyurethane/polyester fiber polishing pads have been studied. Measured removal rates varied from around 100 Å/hr to nearly 2500 Å/hr depending on the values of the various parameters. The amount of material removed was determined by measuring the wafer mass before and after polishing. Variations in temperature and slurry pH did not produce significant changes in the measured removal rates. Higher polishing pressures resulted in increased material removal rates from 200 to 500 Å/hr but also produced excessive polishing pad damage. Variations in pad rotational speeds produced the largest changes in material removal rates, from around 200 to around 2000 Å/hr for rotational speeds between 60 and 180 rpm, but the variations were non-linear and somewhat inconsistent. This CMP formula is shown to consistently produce damage free surfaces but the optimum removal rate is slow.

Key words: Chemical mechanical polishing, silicon carbide, colloidal silica

INTRODUCTION

Silicon carbide (SiC)[1] is a wide bandgap semiconductor material that is resilient to chemical attack, radio frequency interference and radiation damage. Because of its large bandgap and high thermal conductivity, SiC devices have shown the ability to operate at temperatures as high as 650°C. In addition, its lattice constant makes it a good choice as a substrate material for gallium nitride based devices. Silicon carbide also has excellent mechanical and wear properties which create a challenge when attempting to apply conventional polishing techniques to obtain a damage free surface. Indeed, commercially available SiC wafers show considerable surface scratching and subsurface damage[2] which make the growth of high quality epitaxial films difficult. Figure 1 shows an atomic force microscopy (AFM) image of the surface of an as-received vicinal 4H-SiC where the scratches are clearly visible. The scratches here are as wide as 430 nm and as deep as 4.6 nm.

Chemical mechanical polishing (CMP) is widely used in the semiconductor industry to produce mirror-like surfaces with no measurable subsurface damage. This technique is usually used after mechanical polishing with abrasive slurries of progressively finer grit size. Due to the hardness of SiC, diamond grit is the only option for mechanical polishing.

Whereas diamond based polishing solutions depend entirely on mechanical removal of the semiconductor material by plastic deformation, CMP slurries are effective by using both chemical and mechanical removal mechanisms and one would expect that this technique would be applicable to SiC. Unfortunately, at present, very little research has been published on surface preparation of SiC. Zhou et al.[3] reported a colloidal silica-based CMP for SiC. Other studies of SiC surface preparation include chromium III oxide abrasive polishing solutions,[4] hydrogen etching,[5] tribochemical polishing in oxidant solutions,[6] and surface roughness measurements of chemically mechanically polished 3C-SiC wafers.[7] Unfortunately, to the authors' knowledge, only Zhou et al. have reported removal rate data for SiC CMP. In Fig. 2 we

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show the AFM image of the same wafer as the one in Fig. 1 after a colloidal silica-based CMP. No scratches are visible and the CMP process is clearly effective at removing residual damage from SiC wafers. However, the polishing time required to achieve "scratch-free" surfaces with this polish usually ranges between one and three hours. A shorter time is desirable for a commercial CMP process and studies of optimization of the removal rate are necessary. We report here an investigation of the effects of temperature, pH, pressure, and rotation rate on the removal rate in 8° off axis 4H-SiC wafers by colloidal silica CMP.

EXPERIMENTAL SETUP AND PROCEDURES

The wafers used during this study were 1 3/8 inch, 8° off axis 4H-SiC wafers. The wafers were numbered consecutively by the manufacturer and are believed to be adjacent wafers from the same boule. The micropipe density was reported by the manufacturer to be 50 cm^{-2}. Prior to any polishing, the wafers were thoroughly examined and photographed using a Zeiss Axiotron II microscope, a Hitachi HV-C2O camera, and supporting Zeiss *Image* 3.0 software. This microscope was capable of 1000x magnification and had the capacity of Nomarski differential contrast. In addition to optical microscopy, AFM amplitude and height images were obtained using a Dimension 3000 large sample microscope system in tapping mode with a Digital Instruments NanoScope IIIa microscope controller. Following initial mass measurements, the wafers were given a CMP polish using Logitech SF1 colloidal silica polishing solution and Rodel regular politex polishing pads which are composed of polyurethane/polyester fibers. This is similar to the polishing solution used by Zhou et al.[3] An attempt was made to maintain a thin layer of polishing slurry on the pad at all times by dripping the slurry directly on the pad surface. However, during high pressure and high speed studies this was not always possible. The wafers were polished for 30 or 60 min periods using a Strasbaugh Precision Polishmaster with a random motion polishing armature. The wafer was then cleaned and the material removal rate was calculated from wafer mass measurements which are described below. In addition, the wafer surface was re-examined using optical microscopy and digital photographs were obtained.

The temperature of the wafer was monitored at all times during the polishing process by passing a type-K thermocouple through a pre-drilled hole in the wafer attaching mount. Thus, the thermocouple was in direct contact with the back, unpolished side of the SiC wafer. During temperature studies, the temperature of the wafer was increased by passing hot air over the surface of the wafer mount with a heat gun. During pH studies, the slurry pH was increased by adding small amounts of 1.25 M NaOH solution. The polishing pressure was adjusted by adding/removing lead weights from the wafer mount shaft. In an effort to reduce pad effects on removal rate, a new pad was used for each set of test parameters and the life of the pad is displayed on the following figures as the plot abscissa.

A key aspect of this study was to identify a method of determining the removal rate that would provide accurate and repeatable results. Several methods currently exist that can be used for this purpose. Perhaps the simplest of such methods is to measure the thickness of the wafer before and after each polishing period. Devices are commercially available

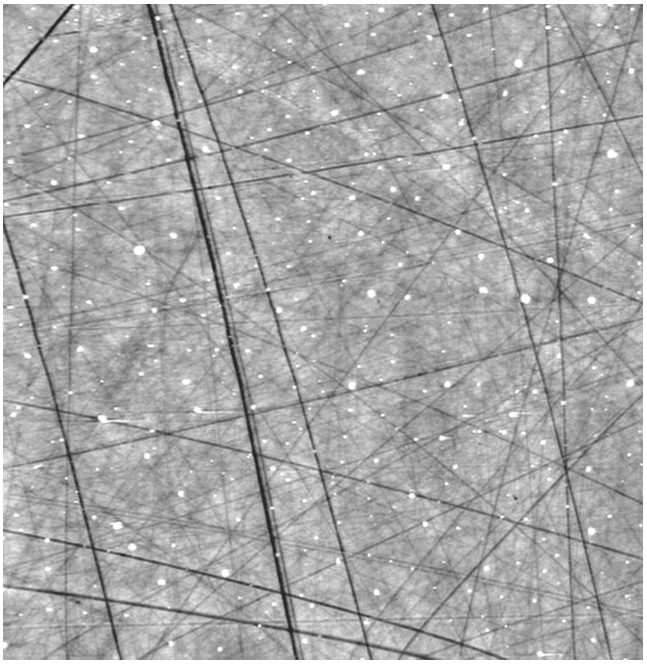

Fig. 1. 50 × 50 mm AFM amplitude image of as-received 4H SiC wafer.

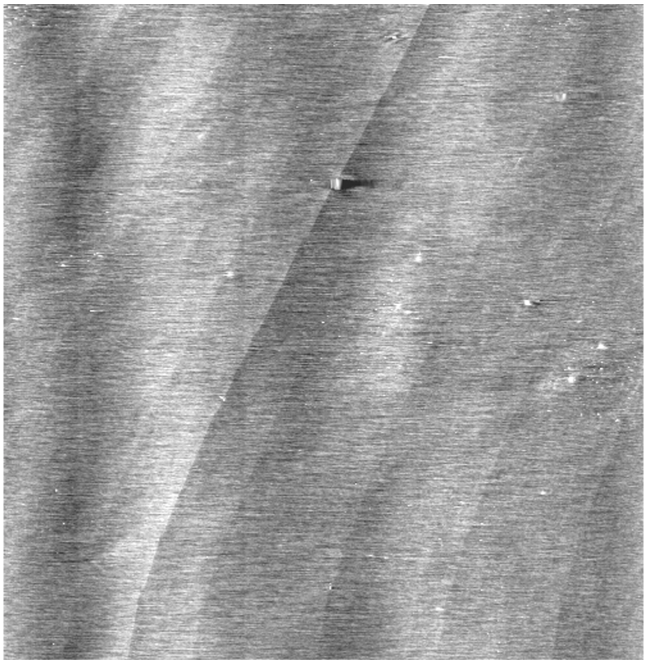

Fig. 2. 50 × 50 mm AFM amplitude image of the 4H SiC wafer after 1 h of chemical mechanical polishing at 23°C, 9.9 slurry pH, 5lb/in^2, and 200 rpm.

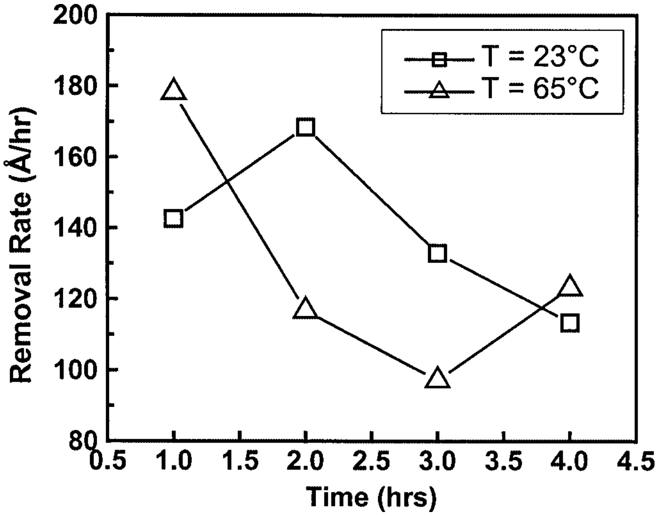

Fig. 3. Effect of wafer temperature and polishing time on the material removal rate. 60 rpm, 5lb/in^2, and pH ~ 9.9. A new polishing pad was used for each temperature study.

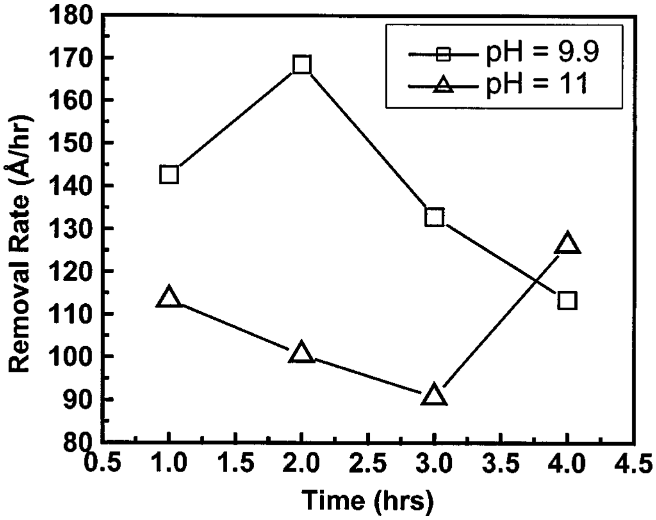

Fig. 4. Effect of slurry pH on removal rate. Slurry pH for rotation rate of 60 rpm, applied pressure of 5lb/in^2, and temperature of 23°C. A new polishing pad was used for each slurry pH study.

that have this capability and provide up to 0.01 micrometer resolution. Another technique involves placing an indentation of known geometry on the wafer surface and measuring the diameter of the indentation before and after each polishing period using optical microscopy. It is believed that this method of removal rate determination is less effective at providing accurate and repeatable results. Since polishing often results in rounding of surface edges, error is introduced into this type of measurement during the polishing process. Additionally, it is extremely difficult to determine the precise location of an edge using optical microscopy after edge rounding has occurred.

A third method of removal rate determination was examined during this study. The method involved reactive ion etching a series of parallel trenches in the wafer surface of about 2 mm deep, 75 mm wide, and 4 mm long. Dektak profile measurements of the trenches were taken before and after each polishing period. It was thought that the trenches would be decreased in depth by the amount of material removed from the wafer surface. It was, however, quickly discovered that this method of removal rate determination would be ineffective for two main reasons. First, the depth of the trench varied by as much as several thousand angstroms along the length of the trench. Although identifying features on the surface helped locate the Dektak stylus to the same general area, variability of hundreds of angstroms occurred even with the aid of surface features. Secondly, a damage layer at the trench bottom was created during the reactive ion etch process. The damage layer was of unknown thickness and was incrementally removed from the trench bottom during the polishing period. The damage layer removal introduced additional error into removal rate calculations using Dektak measurements since the removal of the damage layer proceeded at a highly accelerated rate compared to the removal of bulk material from the wafer surface.

The final method of material removal rate that was examined and ultimately used for this investigation involved taking wafer mass measurements before and after each polishing period. For this study a Mettler AT20 scale was used which has a resolution of the nearest even micro-gram. Assuming a 1 3/8 inch SiC wafer with major and minor flats, a mass of 31 micro-grams is associated with an evenly distributed removal of approximately 100 Å from the wafer surface. This was found to be the most repeatable method of measuring mass removal. Wafers were inspected for chipped edges after each polish and were removed from study if a new chip was found.

RESULTS

Polishing at wafer temperatures of 23°C and 65°C were performed to investigate the effects of increased temperatures on the material removal rate. After polishing the wafer with a new pad for four hours at 23°C, scratches were re-introduced onto the wafer surface using a 3 mm diamond grit polish and the old pad was replaced with a new pad for the 65°C study. Figure 3 shows the effects of both temperature and the life of a polishing pad on the removal rate. Although some removal rate variability occurred, it is evident from the figure that increased temperatures do not produce significant improvements in removal rate.

The effects of slurry pH were studied at rotational speeds of 60 and 90 rpm. Figure 4 is a plot of data obtained during the 60 rpm study. The average removal rate over the four-hour period using a 9.9 pH polishing slurry was 139 Å/hour while the average using an 11 pH polishing slurry was only 108 Å/hour. Thus, for this particular type of polishing slurry, wafer material removal rates did not increase with increasing levels of slurry pH. It is believed the decrease in the observed removal rate occurred due to the decrease in silica particle percent content in the

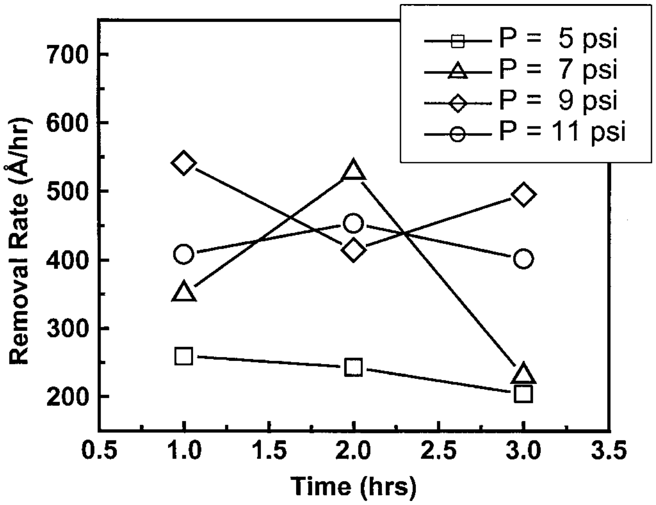

Fig. 5. Removal rate versus polishing time for different applied pressures at 90 rpm, 23°C, and pH ~ 9.9. A new polishing pad was used for each pressure study.

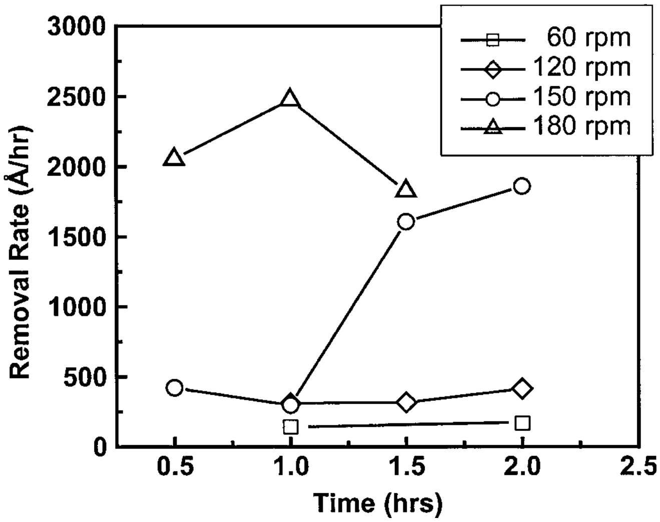

Fig. 6. Effect of pad rotational speed on the removal rate for 5lb/in^2 pressure, 23°C temperature, and pH ~ 9.9. A new polishing pad was used for each rotational speed study.

polishing slurry as a result of adding 1.25 M NaOH solution to increase the slurry pH level. The formation of a silica precipitate upon introduction of the NaOH solution into the polishing solution may have also contributed to the decreased material removal rates. During the 90 rpm study, slurry pH values of 9.9, 11, and 12 were examined. The effects of increasing the polishing slurry pH levels for the 90 rpm study were similar to the 60 rpm experiment.

The effect of pressure was also studied for four different pressures. The SiC wafers were polished for three 60 minute intervals at pressures of 34.5, 48.3, 62.1, and 75.8 kPa (5, 7, 9, and 11 psi) and the results are shown in Fig. 5. As expected, removal rate increases with increasing pressure. As the applied pressure increases, the wafers are pressed deeper into the polishing pad fibers and interact with more silica particles. However, the three highest pressures also resulted in noticeable pad damage seen as black fibers floating on the surface of the white polishing slurry. Since higher pressures resulted in unwanted polishing pad damage, it was decided to study the effects of polishing pad rotation rate at a pressure of 34.5 kPa (5 lb/in^2) where no noticeable pad damage occurred.

Polishing pad rotational speed had, by far, the largest effect on material removal rate of all the parameters studied. During this study, pad rotational speeds of 60, 120, 150, and 180 rpm were examined. During the studies at 120, 150, and 180 rpm, wafer mass measurements were obtained after each 30 min polishing period as opposed to the 60 min intervals used during the 60 rpm experiment. Polishing intervals were decreased to 30 min for the higher speed studies due to the large amounts of slurry used in an attempt to maintain a thin film of polishing slurry on the pad at all times. Figure 6 displays the material removal rate as a function of pad life for each of the four rotational speeds. A comparison of the data reveals that material removal rates are quite stable at lower polishing pad rotational speeds. However, as the polishing speed is raised, both material removal rate and variability increase significantly. In particular, the removal rate variability during the 150 rpm study is quite high and ranges from 298 Å/hour observed during the second 30 min polish to 1860 Å/hour during the last 30 min polishing interval. Surprisingly, the removal rate variability during the study at 180 rpm stabilizes in comparison to the 150 rpm data. It is believed that the high variability is due to the inability to maintain a constant amount of polishing solution on the pad at all times during the polishing interval. Although it was uncertain that a constant amount of polishing slurry was maintained on the pad at all times, it is apparent that increasing the polishing pad rotational speed has more effect on material removal rates than temperature, slurry pH, or pressure.

DISCUSSION

Several theories are available that describe the CMP process. Following the work of Pietsch et al.[8] on Si and Trogolo and Rajan[9] on SiO_2, Zhou et al.[3] suggest for SiC that OH^- ions in an alkaline solution bond with the single dangling bonds of the surface silicon atoms and form SiO_2 on the surface which is significantly softer than SiC. The SiO_2 is then, according to the model of Zhou et al., removed by mechanical wear between the silica particles in the polishing solution and the wafer surface. Trogolo and Rajan, however, suggest an alternative process in which silica is dissolved in the slurry after the formation of silanol ($Si(OH)_4$), which is soluble in high pH slurries. Thus, by either model, one parameter that should play a major role in the polishing process is the chemical composition of the slurry. Compositions that maximize the reaction rate between semiconductor and slurry atoms should, in principle, result in higher material removal rates and reduced polishing times for CMP of SiC. Further, one would also expect that optimizing polishing parameters which result in accelerated chemical reaction rates between atoms should result in an associated increase in removal rate. Based on their study of the effects of increased pH levels and polishing temperature on the removal rate during colloidal silica based CMP of SiC, Zhou et al. reported that increased temperatures and pH values resulted in increases in material removal rate. They reported a maximum material removal rate of approximately 2000 Å/hour at a polishing temperature of 55°C and a slurry pH of 11. In another report on silicon CMP, Pietsch et al. reported slurry pH has perhaps the greatest effect on material removal rate during the CMP process.[11] Both of these studies found that increasing the slurry pH levels resulted in increased material removal rates. However, Pietsch et al. claim that the beneficial effects of increasing slurry pH plateaus at a slurry pH of about 11.5 and the material removal rate decreases as further increases in slurry pH are made. Several reports are available on the effects of polishing pressure and rotation rate on the material removal rate for CMP of silicon wafers. In particular, Tseng et al.[12,13] compared experimental and numerically derived removal rates for CMP of silicon. However, there is no available information on the effects of varying polishing pressure and rotation rate for CMP of SiC and comparisons with silicon must be made with care. The removal rates quoted by Pietsch et al. for Si CMP are about a factor of a thousand higher than the SiC CMP removal rates we have measured with very similar polishing slurries and equipment.

Our results appear to contradict the standard theories on CMP discussed above in that we did not see a strong temperature or pH dependence with our CMP process, but did see effects for pressure and rotation rate. These last two parameters in themselves do not affect chemical reactions but can have secondary effects. Li et al.[14] have studied the mechanics of CMP of SiO_2 and report that temperature affects the pad fibers by decreasing the fiber dynamic shear modulus with increasing temperatures. Decreasing the dynamic shear modulus allows the wafer to sink deeper into the pad and hence contact more silica particles. Since the dynamic shear modulus of the pad fibers used in our study vary little over the temperatures investigated in our study we would not expect to see this effect. Therefore the absence of a temperature dependence in our experiments suggests that the chemical reaction rate is low enough that other effects dominate the removal of material. We have measured a reduction in the pH of the waste slurry and have found that it is lower. This suggests that a chemical reaction is indeed taking place and that the process is not simply mechanical. Due to the major effect of mechanical process parameters we have observed, differences between our results and those of Zhou et al. might be explainable in that different polishing pads were used as well as different suppliers for the siton slurry. Different polishing machines were also used.

CONCLUSION

The effects of variations of wafer/slurry temperature, slurry pH, applied pressure, and pad rotational speed on SiC removal rates during colloidal silica CMP were investigated. Variations in wafer temperature and pH did not significantly affect material removal rate. The largest effects were found for rotation speed and pressure. Our results can be explained by assuming, according to Li et al., that polishing temperature affects the dynamic shear modulus of the pad fibers and it is through this mechanism that variations in removal rate are experienced. Higher slurry pH levels do not necessarily result in accelerated material removal rates. It is believed that the effects of slurry pH are dependent on the type of polishing slurry being used. As expected, increasing applied pressure results in accelerated removal of wafer material. However, excessive pressure results in polishing pad damage and increased wafer preparation costs. Polishing pad rotational speeds had the greatest impact on wafer material removal rates with an associated increase in removal rate variability. Additional rotational speed studies would be useful in an attempt to decrease variability while maximizing material removal rate.

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REFERENCES

1. G.L. Harris, *Properties of Silicon Carbide*, *INSPEC* (Piscataway, NJ: IEEE, 1995).
2. W. Qian, M. Skowronski, G. Augustine, R.C. Glass, H.McD. Hobgood, and R.H. Hopkins, *J. Electrochem. Soc.* 142, 4290 (1995).
3. Z. Zhou, V. Audurier, P. Pirouz, and J.A. Powell, *J. Electrochem. Soc.* 144, L161 (1997).
4. M. Kikuchi, Y. Takahashi, T. Suga, S. Suzuki, and Y. Bando, *J. Amer. Ceram. Soc.* 75, 189 (1992).
5. V. Ramachandran, M.F. Brady, A.R. Smith, R.M. Feenstra, and D.W. Greve, *J. Electron. Mater.* 27, 308 (1998).
6. Z. Zhu, V. Muratov, and T.E. Fischer, *Wear* 225/229, 848 (1999).
7. A.A. Yasseen, C.A. Zorman, and M. Mehregany, *J. Electrochem. Soc.* 146, 327 (1999).
8. G.J. Pietsch, G.S. Higashi, and Y.J. Chabal, *Appl. Phys. Lett.* 64, 3115 (1994).
9. J.A. Trogolo and K. Rajan, *J. Mater. Sci.* 29, 4554 (1994).
10. D.V. Ragone, *Thermodynamics of Materials Volume II* (New York: John Wiley & Sons, Inc., 1995), p. 205.
11. G.J. Pietsch, Y.J. Chabal, and G.S. Higashi, *J. Appl. Phys.* 78, 1650 (1995).
12. W.T. Tseng and Y.L. Wang, *J. Electrochem. Soc.* 144, L15 (1997).
13. W.T. Tseng, J.H. Chinn, and L C. Kang, *J. Electrochem. Soc.* 146, 1952 (1999).
14. W. Li, D.W. Shin, M. Tomozawa, and S.P. Murarka, *Thin Solid Films* 270, 601 (1995).