SYNTHESIS AND STUDY OF ALMGB COMPOSITE FILMS

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Synthesis and Study of AlMgB Composite Films
鋁鎂硼複合膜的沉積與研究

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ABSTRACT OF DISSERTATION

Along with the development and commercialization of many novel and innovated products, demand for the use of superhard materials continuously increases. Especially diamond and cubic boron nitride (cBN) have received considerable attention. The synthetic diamond and cBN prepared by high temperature-high pressure (HTHP), in forms powders with different sizes of crystalline grains, are widely commercialized as abrasives and in production of cemented tools. Synthetic HTHP diamond is used also in jewelry. Diamond and cBN have also been prepared in forms of thin films by low pressure chemical vapor deposition (CVD), but only diamond films are industrially exploited, since the technologies of cBN films are far more challenging than those of diamond. Cubic BN is unique material that surpasses some properties of diamond and it cannot be substituted by diamond in some applications. Therefore it is logical to search for new materials that can substitute cBN. For instance, in mechanical applications, alternative materials have to have superhard property, high thermal conductivity, and they have to be chemically stable and resistant to oxidation at high temperatures. As a result multi-element solids emerge as a new group of super-hard materials. In 1970, AlMgB$_{14}$ with outstanding mechanical properties have been prepared. Despite this fact, only little attention has been given to this excellent material. Cook et al predict that a new class of ultra-hard materials based on AlMgB$_{14}$ structure and report that their
intermetallic AlMgB$_{14}$ composites with density of 2.59 g/cm$^3$, high chemical inertness and thermal stabilities. The AlMgB hardness can approach the microhardness of 35 GPa, which can further be enhanced by adding other elements such as Si and TiB$_2$ into the AlMgB$_{14}$ structure.

The presented work therefore focuses on the development of unique films based on the ternary AlMgB and quaternary AlMgBTi structures and investigation of their properties. In this work, considerable attention is given to the structural analysis and mechanical properties of these films. Electrical and electronic properties of the boron rich AlMgB film are investigated using ultraviolet photoelectron spectroscopy (UPS) and Hall measurements. Since electronic analyses point at relatively low work function of some AlMgB films, the field emission properties of the boron rich AlMgB film are also studied. The high film hardness certainly justifies investigations of tribological properties of thick AlMgB and AlMgBTi films in this study.

Hard films with AlMgB ternary matrices have been prepared by sputter deposition on (100) silicon wafer. The film composition was varied by controlling the electric power density applied to an Al-Mg target. As a result, two types, boron and metal rich, AlMgB films have been prepared at lower and higher power densities applied to the Al-Mg target, respectively. Both the boron and metal rich films reach the hardness of about 30 GPa. The boron rich films, with composition closed to stoichiometric AlMgB$_{14}$,
are not well crystalline and have AlMgB\textsubscript{14} nanoparticles dispersed in an amorphous structure. The nanohardness is just below the boundary of the superhard films. Although the higher deposition temperature promotes the development of B\textsubscript{12} nanocrystalline icosahedra in boron rich films, the substrate temperatures above 200 °C up investigated 600 °C cannot significantly improve the forming of nanocrystalline AlMgB\textsubscript{14} structure. The annealing of boron rich films performed at various temperatures indicate that the film hardness drop due to the constituent agglomeration in the films and relaxation of intrinsic stress. However, the deposition at temperature higher than 800 °C notably contributes to the improvement of the nanocrystalline AlMgB\textsubscript{14} structure.

Hard quaternary AlMgBTi composite films have also been prepared by sputter deposition with an additional TiB\textsubscript{2} target. Comprehensive structural analysis indicates the features of metastable AlMgTiB amorphous structures with embedded TiB\textsubscript{2} nanoparticles which contrast the AlMgB\textsubscript{14} nanocrystallites confined in ternary amorphous AlMgB films. Raising the Ti content in the film leads to the increase of the film hardness, which is mainly attributed to the dispersion of TiB\textsubscript{2} nanoparticles in AlMgBTi matrices. The hardness can reach the value around 40.7 GPa, which is close to the hardness of cBN. The elastic modulus of the films is lower than that of diamond and cBN films. The higher deposition temperature and/or high annealing temperature at 1200 °C cannot notably promote the formation of AlMgB\textsubscript{14} and TiB\textsubscript{2} nanoparticles in
the AlMgBTi matrices. The trials of corrosion tests show that the Ti additives reduce the corrosion resistance of the films, primarily owing to the enlargement of the surface interface.

Boron rich Al$_{0.06}$Mg$_{0.02}$B films have been prepared on both silicon wafer and quartz for detailed studies of electronic and electrical properties. The Al$_{0.06}$Mg$_{0.02}$B films exhibit semiconducting behavior with a narrow optical bandgap (~0.5eV). They show low resistivity (18.1 mΩ.cm.) and p-type charge carrier mobility (15.5 cm$^2$/Vs) at charge carrier concentration of 2.23×10$^{19}$ cm$^{-3}$. Since the electrical charge transport is facilitated by holes (provided by boron elements), the excited electron can easily be seized in the trap states. The electronic study suggests that the films have low work function (3.93 eV), which is suitable for electron field emission. The electron field emission measurements indicate the turn-on field of the films is 5.2 V/μm at a field emission current density of 10 μA/cm$^2$, while the current density of 10mA/cm$^2$ is achieved when the electrical field is 8.3 V/μm.

The tribological study of the thick AlMgB and AlMgBTi films suggests that the friction coefficients of AlMgB and AlMgBTi films are 0.55 and 0.59, respectively. However, when a lower force is applied in tribological measurements, both the film types show very low friction coefficients (~0.13). This low value coefficient is mainly attributed to the surface boron oxide layers that sustain at low applied forces. The
boron oxide layer is inherently characteristic with low friction coefficient.

In summary, the investigated AlMgB films show hardness close to 30 GPa. The hardness can be further improved to above 40 GPa by introducing TiB$_2$ additives. Accordingly, AlMgB and AlMgBTi films can be cataloged to be hard (>30GPa) and super hard (>40GPa) coatings. The boron rich films show nearly intrinsic semiconductor behavior with p-type carrier mobility and good electron field emission properties indicating that these films could potentially be used in some electronic device applications.
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<tr>
<td>$\theta$</td>
<td>The angle of degree</td>
<td>[$^\circ$]</td>
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<tr>
<td>ER</td>
<td>elastic recovery</td>
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<tr>
<td>$D_{\text{max}}$</td>
<td>displacement at the maximum load</td>
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</tr>
<tr>
<td>$D_{\text{res}}$</td>
<td>residual displacement after unloading</td>
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<td>$\alpha$</td>
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**Abbreviations**

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<td>HTHP</td>
<td>high temperature-high pressure</td>
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<tr>
<td>CVD</td>
<td>chemical vapor deposition</td>
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<tr>
<td>cBN</td>
<td>cubic boron nitride</td>
</tr>
<tr>
<td>ANSI</td>
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</tr>
<tr>
<td>PLD</td>
<td>pulse laser deposition</td>
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<td>DLC</td>
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