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Plasma Deposition of High Temperature Protective Coatings

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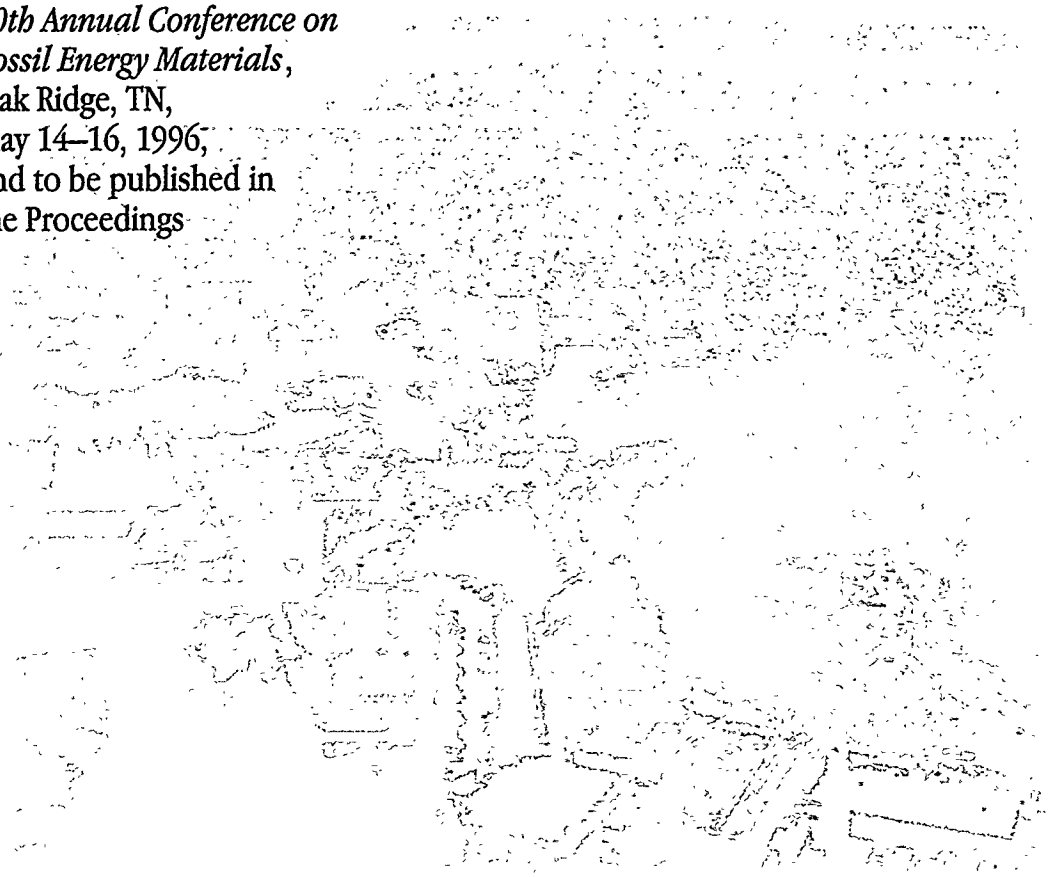

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PLASMA DEPOSITION OF HIGH TEMPERATURE PROTECTIVE COATINGS

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ABSTRACT

Oxide ceramic films can be made using a vacuum arc based technique in which a metal plasma is formed in an oxygen background, with ion energy controlled by the application of a repetitive pulse bias to the substrate throughout the deposition. High ion energy early in the process produces atomic mixing at the film-substrate interface while lower but optimized ion energy later in the deposition can control the structure and morphology, and films that are adherent and dense can be formed in this way. In recent work we have investigated the formation of films of aluminum-silicon oxide on silicon carbide substrates by mixed aluminum and silicon plasmas produced from separate plasma sources. The Al to Si ratio was controlled by the plasma gun parameters, and the guns were pulsed simultaneously so as to mix the plasma streams before deposition; a magnetic multipole plasma homogenizer device was also used to further blend the two plasmas. Films with Al:Si ratios varying from 2:1 to 8:1 were produced in this study (Al:Si is 3:1 for Mullite), and the oxygen content in the films was controlled by varying the oxygen partial pressure during deposition. Additionally, we have compared some of the characteristics of alumina films plasma deposited on iron aluminide substrates with films produced by e-beam evaporation of Al_2O_3 using oxygen ion beam assisted deposition onto unheated or heated substrates. Here we outline the film synthesis techniques and describe the characterization results obtained to-date.

INTRODUCTION

The formation of thin films by plasma deposition is a versatile technique with a wide range of different embodiments. The version that we've developed and used has the important feature that the ion energy can be controlled widely. Highly adherent, dense films of metals, alloys (including non-equilibrium alloys) and other conducting materials, and their oxides and nitrides, can be formed. In the early stages of the process the ion energy is held in the keV range so as to produce atomic mixing at the film-substrate interface, and in the latter stages of deposition the energy is reduced so as to optimize the film structure and morphology.

In this method, which we call Mepiiid (*Me*tal *p*lasma *i*mmersion *i*on *i*mplantation and *d*eposition), the object to be implanted is immersed in a plasma of the desired species and repetitively pulse-biased to a negative voltage[1-6]. A high voltage sheath rapidly forms at the substrate-plasma boundary, and plasma ions are accelerated through the sheath and into the substrate, thereby accomplishing implantation into the substrate of plasma ions at an energy determined by the bias voltage. Because of the surface retention of condensed metal plasma, the plasma immersion process in a metal plasma is quite different from in a gaseous plasma. Ions that are accelerated from the plasma into the substrate during the high voltage bias pulse suffer collisions with previously-deposited neutral metal atoms on the substrate surface and thus also produce recoil implantation. By varying the proportions of the direct and recoil implantation parts of the

cycle (ie, the duty cycle of the pulse biasing) one can tailor the shape of the profile, and the range can be tailored by the amplitude of the applied pulse voltage. The whole operation can be time-varied throughout the processing duration, starting for example with a high energy phase so as to create a deep buried layer of the implanted species and slowly changing to a low energy phase whereby a surface film is built up by plasma deposition. By controlling the plasma ion energy we acquire control over two very important features to the deposition process: the interface width can be tailored, and the film morphology and structure can be controlled. In this way one can synthesize a surface metallic film of precisely controllable thickness having a well-determined and controllable atomically mixed interface with the substrate, and the parameters of the film and the interface can be tailored over a wide range. For purely metallic films, the process is carried out at a vacuum in the 10^{-6} Torr range; oxides and nitrides can be formed by doing the deposition at the appropriate background pressure of oxygen or nitrogen, typically a few tens of microns. The technique has been developed by us and others, and put to a range of laboratory applications.

In prior work we have shown that highly adherent films can be formed in this way. We have produced near-stoichiometric alumina films of thickness typically 0.5 μ on FeAl substrates; the films were amorphous prior to heat treatment and showed an α-alumina phase after heat treating at 1000°C for up to 16 hours[7,8]. The film substrate adhesion was typically greater than ~70 MPa prior to heating, and the adhesion was maintained after repetitive cycling in temperature between ambient and 1000°C.

We report here on the extension of this work in two directions. We have explored the plasma synthesis of aluminum-silicon oxide films on silicon carbide substrates by mixing together separately produced plasmas of aluminum and silicon. The Al to Si ratio was controlled by the plasma gun parameters and the plasmas blended together in a magnetic multipole plasma homogenizer device. Deposition was done onto appropriately positioned SiC substrates. Separately, we have compared the characteristics of alumina films plasma deposited on iron aluminide substrates with films produced by e-beam evaporation of Al_2O_3 using oxygen ion beam assisted deposition onto unheated or heated substrates. We outline here the film synthesis techniques and describe the results obtained to-date.

PLASMA PROCESSING

The vacuum arc is a high current discharge between two electrodes in vacuum in which metal plasma is produced in abundance[9-12]. We have made a number of different embodiments of vacuum arc plasma guns. For the work described here we used a small, repetitively pulsed version operated at a pulse length of 5 ms and repetition rate about 1 Hz, and arc current in the range 100 – 300 A. Along with the metal plasma that is generated, a flux of macroscopic droplets of size in the broad range 0.1 - 10 microns is also produced[9-14]. In general it is desirable to remove the solid particulate contamination and this can be done using a curved 'magnetic duct' which stops line-of-sight transmission of macroparticles while allowing the transmission of plasma[15,16]. The overall plasma deposition system thus consists of the repetitively pulsed plasma gun in conjunction with the 90° bent magnetic filter. Plasma exits the filter and deposits onto the appropriately positioned substrate.

Metal oxides can be formed by carrying out the deposition not in a high vacuum environment but in a somewhat higher pressure ambient of oxygen gas; we have found empirically that a pressure in the range 1 - 100 mTorr is suitable for most purposes. In the present work the oxygen background pressure was 5 to 25 mTorr. The oxygen is both entrained in the plasma stream, ionized, and deposited in the plasma state, as well as reacting at the freshly-deposited metallic surface to form aluminum oxide or silicon dioxide. In either case, for the optimal oxygen pressure a near-stoichiometric film of the metal oxide is formed.

Ion energy of the depositing plasma flux is controlled by repetitively pulse biasing the substrate. Typically the pulse duration might be ~10 μs and the duty cycle ~10 - 50%. Pulsing of the bias voltage is necessary (for all but the lowest bias voltages) because a high-voltage dc bias would cause an electrical discharge between the substrate and the vessel or the plasma gun; the plasma would be grossly perturbed (because the plasma sheath would expand from the substrate to large distances). The solution is to switch off the bias before a discharge can occur (to limit the sheath expansion to modest distances), let the plasma recover, and then repeat the process; ie, to do the biasing in a repetitively pulsed mode. For the early stages of the deposition process the pulse bias is held at a relatively high voltage of 2.2 kV. The mean aluminum ion energy is then 3.75 keV, because the mean ion charge state of the aluminum plasma is 1.7 and $E_i = QV$; for silicon the mean charge state is 1.4 and the mean ion energy 3.1 keV; (the charge state spectra of vacuum arc produced plasmas have been discussed in detail in refs. 17,18). At this energy ions are implanted into the substrate to a depth of up to ~100 Å. The film thus grows on the SiC substrate from a highly mixed interface. When a film thickness of just a few tens of angstroms has accumulated, the pulse bias voltage is reduced, since intermixing with the substrate is no longer a factor and the higher ion energy would sputter away the already-deposited film. Moreover, it is known from a large body of work on ion assisted deposition that a modest ion energy can be highly advantageous for controlling such characteristics as the density, morphology and structure of the film. For the bulk of the plasma deposition process the pulse amplitude is kept at 200 volts.

For synthesis of the mixed aluminum-silicon oxide films we firstly tried a plasma gun cathode that was formed of mixed Al-Si in 3:1 atomic ratio – mullite has composition $3Al_2O_3.2SiO_2$ for an Al:Si atomic ratio of 3:1 – but we found that the deposited film composition ratio did not reflect the cathode composition ratio closely enough. Thus we went to an alternative approach making use of an additional plasma gun/filter for forming a Si plasma that was added to the original Al:Si plasma stream. The two streams are then fed through a special 'plasma homogeneizer', which further mixes and spreads out the plasma stream. The plasma homogenizer is a magnetic multipole plasma confinement geometry, well known in the plasma community and made here using a number of high-field-strength rare-earth permanent magnets. The utility of this kind of arrangement, in particular the novel use of the homogenizer as a tool for blending the two vacuum arc plasmas, has been explored in prior work in our laboratory[19,20]. The precise Al:Si atomic mix can then be tuned via the plasma guns (pulse width or arc current). Films with Al:Si ratios varying from 1:1 to 8:1 were formed. A simplified schematic of the overall system is shown in Figure 1.

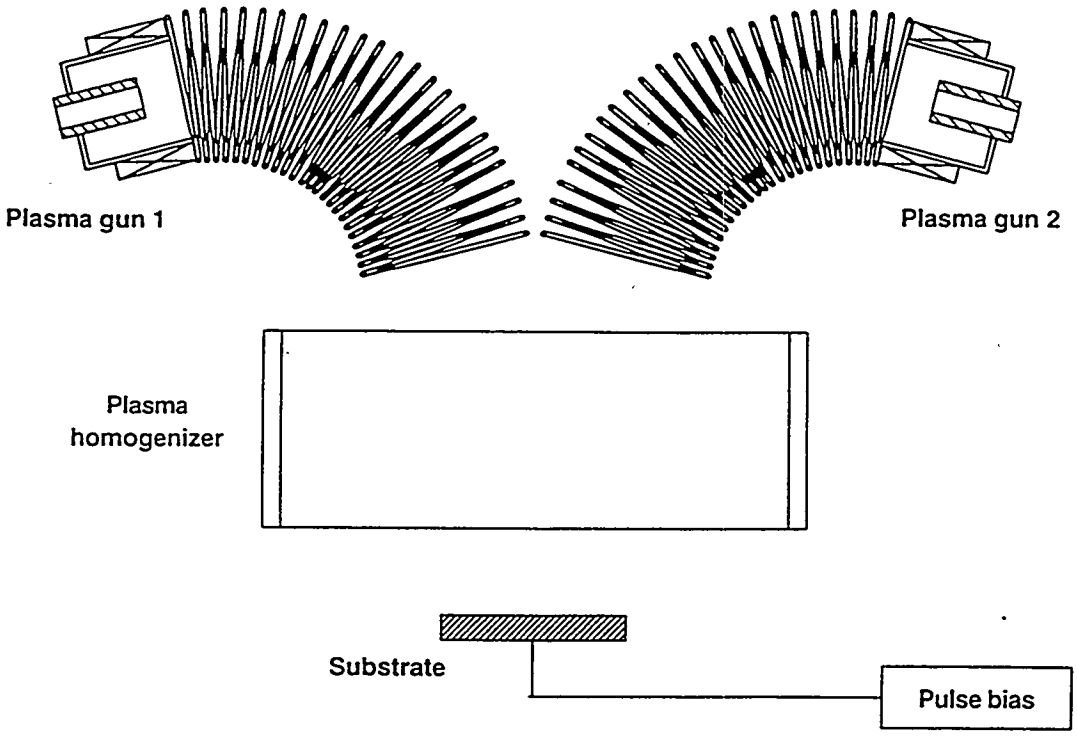

Fig. 1. Schematic of the overall dual-gun deposition system

EXPERIMENTAL RESULTS

Aluminum-Silicon Oxides (Mullite)

Two types of SiC substrate material were used; one was sintered at LBNL from commercially available SiC powders, and the other was a commercial SiC (Hexalloy 5) from Carborundum Industries. The material produced at LBNL contained 94.4% SiC, 3% Al, 2% C, and 0.6% B. The surface was ground and polished to a mirror finish prior to the deposition. Scanning electron microscopy of the polished surface of an uncoated piece of LBNL silicon carbide showed the presence of a second phase (appearing as dark grains).

Control of the stoichiometry of the silicon-aluminum oxide films was achieved by varying the duration of the plasma pulses and the oxygen partial pressure during deposition. The deposition rate of each individual source was determined for all oxygen partial pressures used by measuring the thickness of the deposited films after one thousand pulses of 5 ms duration each. These individual deposition rates were then used to deposit films with desired Al:Si ratios.

An advantage of the Mepiiid process is the high degree of atomic mixing achieved; thus here the duration of the pulses used was always below the time required for the formation of monolayers of silica or alumina, and both sources were triggered simultaneously so as to prevent stratification of alumina or silica. A previous attempt (21) to synthesize mullite coatings by depositing alternate layers of silica and alumina by CVD and subsequent annealing failed due to brittleness resulting from the transformation of silica into crystoballite that takes place during the high temperature annealing required to homogenize the multilayer film. Here we wanted to ensure that complete mixing happened during the deposition.

Table 1 shows the composition of several aluminum-silicon oxides films deposited on SiC under various experimental conditions. The content of each element in the films was determined by Rutherford Backscattering Spectroscopy (RBS) and normalized to the Si content. The accuracy of these measurements is ±15%. Elemental composition of mullite is also included in the table for comparison.

Table 1. Composition of as-deposited films.

Deposition	Al-Si pulse (ms)	Si pulse (ms)	Pressure (mTorr)	Al content	Si content	O content	Thickness (nm)
#1	5	5	25	4.4	1	10.4	240
#2	5	2.5	25	6.7	1	16	230
#3	5	10	25	1.2	1	5.3	240
#4	5	0	25	8	1	19.5	220
#5	5	0	5	7.4	1	13	240
#6	5	5	25	4.2	1	10.4	1250
Mullite				3	1	6.5	

X-ray diffraction indicates that the deposited films were amorphous. No evidence of crystalline structure or phase separation was observed. A feature in the films produced was the high oxygen content when deposition was carried out at 25 mTorr. An analysis of the compositions shown in Table 1 indicates that films produced at this pressure consist of mixtures of Al_2O_{3+x} and SiO_{2+y} with a good match being achieved for $x = 2$ and $y = 0$. Al_2O_5 is not a stable oxide for our deposition conditions, and therefore this composition must be a consequence of oxygen ion bombardment. Such high oxygen content has been found previously in pure alumina films produced by MePiiid at 26 mT (7). At lower pressures, oxygen implantation appears to decrease in importance and the films are basically mixtures of Al_2O_3 and SiO_2.

Annealing of the films at 1100°C in air for 2 hours led to the formation of crystalline mulllite coatings. The alumina-silica binary phase diagram indicates that the only stable phases in the silica-alumina system at the pressures used in this investigation are silica, alumina and mullite. In order to achieve the mullite stoichiometry, the excess oxygen present in the as-deposited films had to be lost to the environment or react with the SiC substrate and form silicon oxide and/or carbon dioxide. X-ray diffraction indicates mullite as the main crystalline phase in the film after annealing (Fig. 2). The low intensity of the diffraction peaks is

mostly due to the small film thickness. Another silicon-aluminum oxide known as sillimanite ($Al_6Si_3O_{15}$) also provides a reasonable fit to the peaks in the diffractogram, but it was discarded because the ratio of the intensities of the diffraction peaks was closer to that expected from mullite. Little difference was observed in the diffraction patterns of the samples #1 through #5, indicating that after annealing at 1100°C, in all cases the main crystalline phase formed was mullite. In the films with very low Al:Si ratio we were expecting that some silicon dioxide would be present as well, however such a phase, if present, did not provide any signature in the diffractograms. In the films with very high Al:Si ratio, peaks from crystalline alumina were expected because the temperature used in the annealing was above the recrystallization temperature of alumina. No evidence of alumina was detected however.

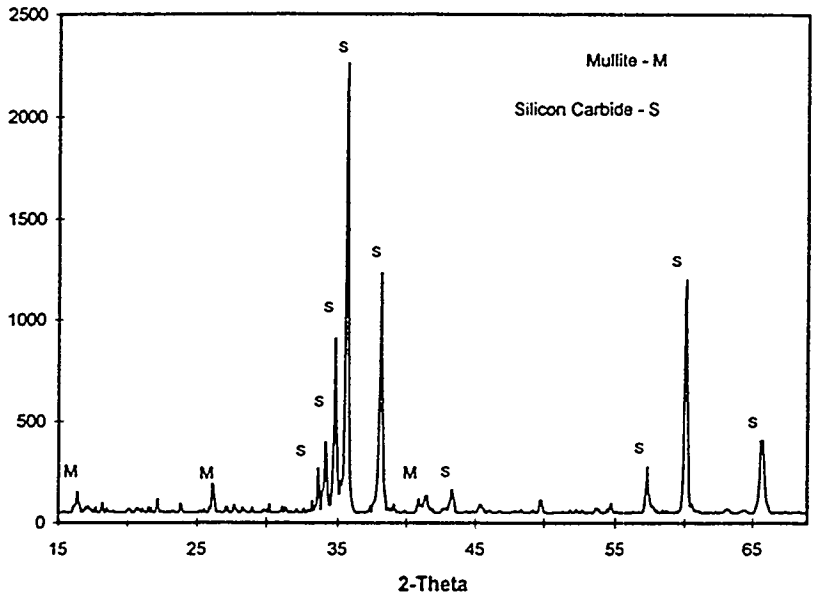

Fig. 2. X-ray diffraction pattern of coated SiC (#1) annealed at 1100°C for 2h in air.

Table 2. Adhesion of as-deposited and annealed films:

Deposition	Adhesion as-deposited (MPa)	Adhesion annealed (MPa)	Al content (pre-anneal)	Si content (pre-anneal)	O content (pre-anneal)	Thickness (nm)
#1	9	70	4.4	1	10.4	240
#2	38	57	6.7	1	16	230
#3	31	63	1.2	1	5.3	240
#4	31	57	8	1	19.5	220
#5	9	65	7.4	1	13	240

Adhesion test results for the as-deposited and the annealed films are shown in Table 2. The value of 70 MPa for sample #1 after annealing is the instrumental limit of our pull-tester before epoxy failure occurs. Thus the film composition closest to that of mullite was most strongly bonded to the SiC substrate.

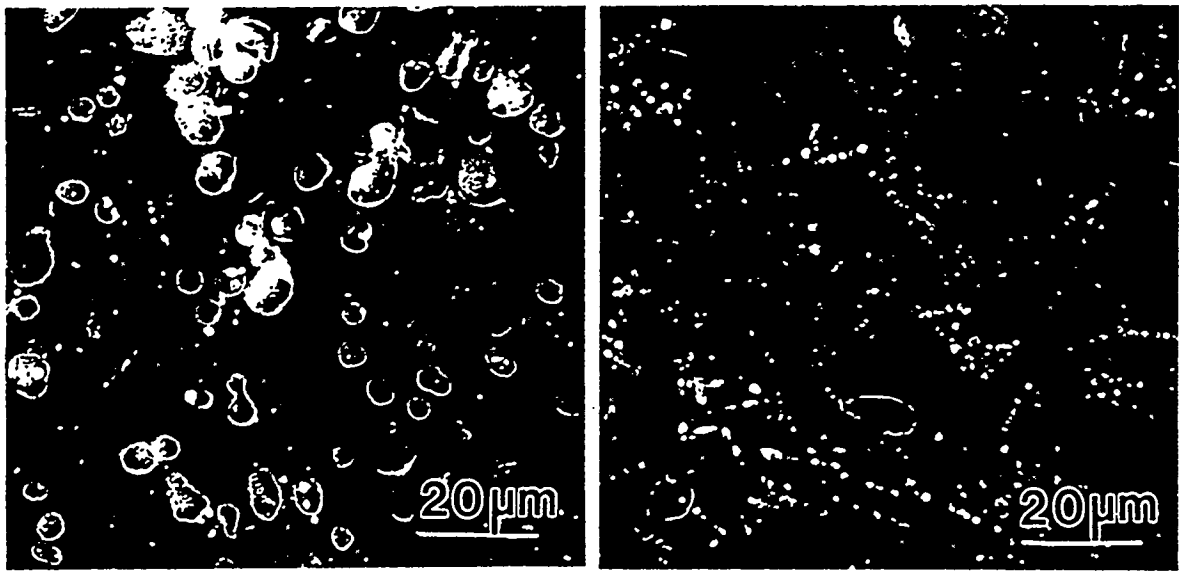

Fig. 3 SEM of a SiC sample with an aluminum-silicon oxide coating after being tested for adhesion. (a) area which had the film removed; (b) area from where the film was still present.

When the aluminum content of the coating was too high, e.g. samples #2, #4 and #5, both the as-deposited and the annealed films had a high concentration of microstructural features that looked like micro-bubbles. The mechanism of formation of these bubbles is not clear to us yet, but their concentration seems to be directly related to the deviation of the Al content from the ideal mullite stoichiometric ratio (i.e. the higher the Al content, the higher the density of voids). These bubbles are clearly seen in the micrographs shown in Fig. 3. Figs 3(a) and 3(b) are SEM images from sample #4 after annealing and after the adhesion test: the former figure is from an area which still has the intact film, and the other from an area without the film. The delamination left the voids exposed, suggesting that these voids were either at the SiC/oxide interface or in the SiC but near to the interface. Upon annealing, very few of these bubbles appear to have broken through the coating, but in the regions where the film delaminates due to the pull-test, these voids have been exposed. Subsurface bubbles (voids) have been observed to form in the silicon carbide used here when subjected to high temperature oxidation.

It is clear from the results presented here that annealing of the deposited aluminum-silicon oxides helps to improve the adhesion of the film, and that proper control of the stoichiometry is necessary in order to maximize the adhesion of the film to the SiC. An extensive analysis of microstructure of the coatings is under way, and should provide an explanation for the observed dependence of the adhesion strength on the aluminum content of the films.

Aluminum Oxides

In this section we describe the initial results of the characterization of the properties of aluminum oxide films deposited on iron aluminides by two different ion beam assisted techniques.

The first technique, Mepiiid, is that as described in the preceding except that instead of two plasma sources only an aluminum source was used, in oxygen. Only depositions at room temperature were conducted. The as-deposited alumina films were amorphous, and with an oxygen content that depended on the oxygen partial pressure. At a pressure of 12 mTorr the film composition was $Al_2O_{3.3}$, whereas at 25 mTorr the composition was Al_2O_5. Such a high oxidation state of aluminum has been observed previously by our group in MePiiid produced films (7). Despite the high oxidation potential of aluminum, the Al_2O_5 produced here is believed to be a direct result of the simultaneous implantation that our plasma method provides. Annealing of the amorphous Al_2O_5 and $Al_2O_{3.3}$ films at 1100°C in air for two hours led to their crystallization into α-Al_2O_3. Adhesion of these films has been reported previously to exceed 70 MPa in the as-deposited amorphous condition as well as after annealing (7).

The second set of aluminum oxide films was prepared using ion-beam assisted deposition according to the following procedure. The substrate was initially ion-milled with 1 kV Ar^+ ions at an ion current of 0.15 mA. Then Al_2O_3 was deposited at a rate of 0.5 nm/s to a total thickness of 500 nm. During the initial 10 - 15 nm, a 1 kV oxygen ion beam (0.25 mA) simultaneously bombard the substrate, and for the remainder of the deposition the energy of the incident oxygen ions was reduced to 0.4 kV. Cooling of the substrate to room temperature after deposition was done in vacuum. Depositions were done at two different temperatures (825 K and 605 K).

The films have been characterized by X-ray diffraction, and the results are presented in Figure 4. Post-deposition heat treatments are presently under way. The e-beam + IBAD films produced at low temperature were predominantly amorphous, while the film deposited at 825K contain microcrystalline aluminum oxides, as evidenced in the X-ray diffractogram. The specific alumina phase present could not be uniquely determined and a mixture of several phases is likely. In addition some amorphous materials may also be present since the deposition temperature was still much lower than the recrystallization temperature, and the diffraction peaks are somewhat diffuse. The film deposited at 825K had a grainy appearance whereas the films produced at 600K looked more homogeneous, similar to the ones deposited by Mepiiid.

The ion energies used in the IBAD and the Mepiiid process are similar. In the early stages of the IBAD process, oxygen ions are accelerated through 1kV, and for the remainder of the deposition the accelerating voltage is reduced to 0.4 kV. In Mepiiid, aluminum ions are responsible for the plasma formation and subsequent ionization of the oxygen. The accelerating voltages used for Mepiiid are 2kV during the first tens of nanometer and 0.2kV for the remainder of the deposition. Characterization and comparison of the two sets of films prepared in the two different but related ways described above is continuing, and we will report on this work as the results become available.

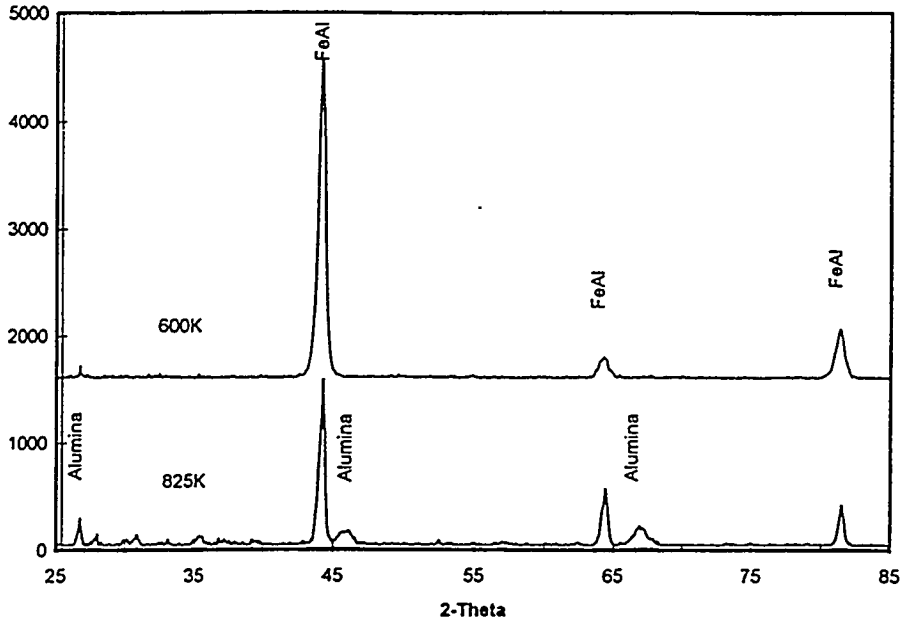

Fig. 4 Diffraction pattern of the as-deposited alumina films produced by e-beam evaporation + ion-beam assisted deposition. The deposition temperatures are indicated.

CONCLUSION

A novel technique for the plasma synthesis of high temperature protective coatings for metallic and ceramic structural materials (*Me*tal *p*lasma *i*mmersion *i*on *i*mplantation and *d*epostion, *Mepiiid*) has been applied to deposit aluminum-silicon oxides on silicon carbide, and aluminum oxide on iron aluminides. Post-annealing adhesion of the aluminum-silicon oxide films was good. Stoichiometry, i.e. the Al:Si ratio, has an effect on the adhesion strength; films with composition closest to mullite have strongest adhesion. When the Al:Si ratio increases, adhesion between the oxide and the SiC is weakened because of the presence of voids at or immediately below the interface. Our plasma method allows convenient and simple control of the "quasi-mullite" film stoichiometry.

Aluminum oxides on iron aluminides were also produced by Mepiiid and by e-beam evaporation + IBAD. Depositions below 300°C resulted in films which are amorphous by both techniques. Deposition at higher temperatures with e-beam + IBAD led to the formation of a mixture of micro-crystalline aluminum oxides. Annealing at 1000°C for 16h in air of the Mepiiid film resulted in a coating that is predominantly α-Al_2O_3. Further work is underway to continue the comparison between the films produced by these two techniques.

ACKNOWLEDGMENTS

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