Growth and characterization of transitional metalsnitrides and carbides thin films

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The transitional metals carbides and nitrides are a special class of materials that combine excellent mechanical, optical, and electrical properties with high thermochemical stability. Thin films of ZrC, TiC, ZrN, TiN have been used in challenging application in nuclear industry [1-5], space exploration [6-7], and microelectronics [8].
Exactly the same properties that recommended ZrC, TiC, TiN or ZrN for these special applications, such as high melting temperature, low sputtering rate, and good thermal conductivity, coupled with the high oxygen affinity of Zr and Ti atoms are those that prevented researchers to easily obtain high quality films.

PLD is probably the best method to grow films with various stoichiometries and degrees of structural perfection, from amorphous to high quality single crystal. Such films are ideal for performing accurate lattice parameter measurements, atomic diffusion or irradiation effects studies to elucidate the mechanisms of defects formation, diffusion and clustering, as well as their influence on the mechanical and thermochemical properties.

Our group from National Institute for Laser, Plasma and Radiation Physics, in collaboration with the group lead by Prof Taylor from the University of Florida, Gainesville, USA have studied the Pulsed laser Deposition of ZrC, ZrN, and TiN, TiCand their properties for several years. The set up for PLD synthesis consists of an all metal-stainless steel chamber equipped with turbomolecular, Ti sublimation, and ion pumps backed by a dry pump. The ultimate pressure is lower than 10-6 Pa. The deposition chamber is also fitted with an Residual Gas Analyser, a liquid nitrogen trap and a ion gun, which could be used for in situ substrate cleaning or low energy ion bombardment of the growing film. The chamber has a target carrousel with six positions for multilayer deposition and a substrate that could be heated up to 1000 ^{o}C under different atmospheres. For the ablation we used a KrFexcimer laser with wavelengthλ=248 nm, pulse duration, τ= 25 ns and a beam ajustable energy in 50-750 mJ range.

The results obtained so far, published in ISI indexed journals [9-13] and presented at several international conferences showed that PLD technique allows for the deposition of very high quality films.

Recently,the scientific interest for carbonitrides and nitrocarbides materials with special properties has greatly increased. To grow these materials by the PLD technique, we deposited transition metals carbides under a N2atmosphere and transition metal nitridesunder a CH4atmosphere and expect to obtain compounds such as TixNyCz or ZrxNyCz. We used XPS and AES to investigate the elemental and chemical composition, but the results depend on matrix effects: the sensitivity factors for C, for example, are different for C in graphite, ZrC or a ZrCN compound.

A different method, where such problems do not exist, will be very helpful.

Being very difficult to estimate the chemical composition of a complex compose by other technics we need to use the RBS technic.

By XPS investigations we determined that C atoms were incorporated as in a carbide type compound and not as graphite. Auger electron spectroscopy helped us find relative concentrations of N and C. The structure of the films has beenanalysed by XRD. Regardless of the C and N content, the films exhibited the typical fcc structure for all Ti or Zr compounds.

To understand the effect of the chemical composition on the mechanical properties it is very important to accurately determine the C, N, O concentrations as well as that of the transition metal.

RBS is the best method to achieve this.

Rutherford backscattering spectrometry with light ions, typically 1–2 MeV ^{1}H or ^{4}He ions, is an often used technique for depth profiling of elements concentrations.

In the extensive use of elastic backscattering for materials characterization purposes, ^{4}He particles up to several MeVhave been for long considered as most convenient projectile. This often gives sufficient mass and depth resolution. However, the case of more complex film structures, with compound materials, have put higher demands on both the mass and the depth resolution in the analysis. It will be rather challenging to accurately determine C, O, and N, since all these elements have very close Z numbers.

The RBS technique has also its limitations. Mass resolution for heavy elements and sensitivity for light elements are poor, and, except for the surface, mass determination is not unambiguously possible. The analysis of light elements in a heavier matrix is often impossible, because of the energy overlap of the beam ions scattered by light surface atoms and by heavier bulk atoms deeper in the sample. Furthermore, small amounts of light elements are difficult to analyze, because of the Z^2dependence of the Rutherford cross section.

A severe disadvantage of conventional RBS is low sensitivity for light elements. The Rutherford scattering cross section is proportional to the square of the nuclear charge of the target nucleus. Therefore, the scattering peaks from light elements such as C, N and O are superimposed on a relatively high background due to backscattering from heavy elements in the sample. In recent years, high energy ^{1}H and ^{4}H backscattering has been utilized to overcome this difficulty and to quantify the stoichiometry or to profile the light elements in the heavy bulk samples. In the high energy backscattering experiments, ^{1}H and ^{4}He ions of *3–9 MeV* (or even more) are used as incident projectiles. The elastic scattering cross section for light elements becomes a nuclear rather than a Rutherford interaction, called non–Rutherford backscattering or nuclear resonance elastic scattering. The non–Rutherford backscattering can be used to enhance the sensitivity for light elements. For example, at ^{4}He energies of *3.045*, *4.265* and *3.72 MeV* the elastic backscattering cross sections for O, C and N are 25, 150 and 6 times larger than their corresponding Rutherford cross sections, respectively.

We intend to use RBS, NRA and ERDA techniques to characterize the thin layers of the above mentioned material. Both RBS and NRBS using ^{4}He will be used. The measurements will be performed at the 3 MV Tandetron accelerator and at the 9 MV Tandem accelerator using dedicated target chambers. The experiments will be performed at our Tandem using a standard backscattering setup. The energy of the ^{4}He beam used for measuremens will be calibrated. The method adopted for calibration of the analysing magnet fields of the Bucharest FN tandem accelerator consists simply of comparing the energies of alpha particles from a radioactive source with the energies of ^{4}He projectiles back-scattered into an silicon detector by thin carbon and gold layers.

The ions scattered at 165^0 will be detected by a Si detector having 17 keVresolution.

We need 3 days (9 shifts) at the 3 MV Tandetron and 5 days (15 shifts) at the 9 MV Tandem.

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