Radiation effects on properties of transition metal carbides thin films

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ZrC, HfC and TiC are a refractory materials that are considered for many special applications, including nuclear fuel encapsulation applications in advanced reactors.

For such applications, the effect of radiation on structure, stoichiometry and properties of these materials must be investigated. Our group has developed the synthesis of high quality thin transition metal carbide films using the pulsed laser deposition techniques. The advantage of this technique is the good control of structure (control of grain size), stoichiometry (control of Metal/C values) and surface morphology. Such films are ideal samples to investigate the effect of radiation on their properties. We have employed X-ray reflectivity measurements to extract information about density, thickness and surface roughness, grazing and symmetrical incidence X-ray diffraction and transmission electron microscopy to characterize the structure and nanoindentation to obtain the mechanical properties of the deposited films.

To simulate the effect of neutron irradiation, thin carbide films will be irradiated with 1-2 MeV Au ions at fluences from 1×10^{13} ions/cm^2 up to 1×10^{16} ions/cm^2. Changes in the irradiated material density will be investigated using both X-ray reflectivity and X-ray diffraction techniques. Changes in stoichiometry will be investigated using Rutherford backscattering spectrometry and X-ray photoelectron spectroscopy (XPS).

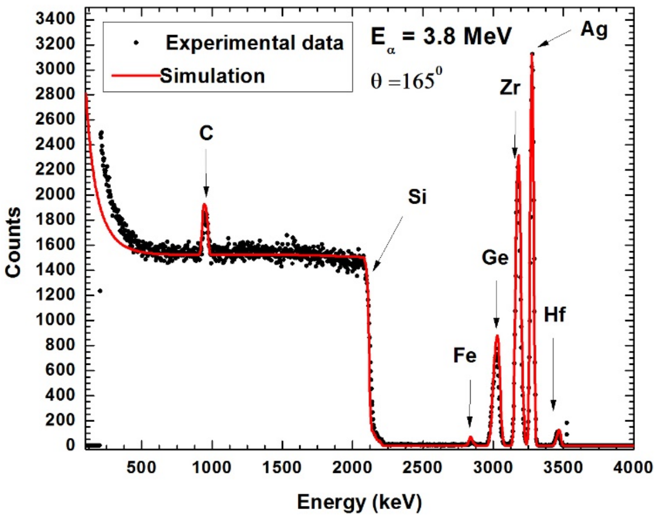

Fig. 1. RBS analysis of a thin ZrC film (200 nm) deposited on a SiGe/Si substrate; the SiGe layer is ~ 200 nm thick).

A major 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}He 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. An example of this type of non-Rutherford scattering is presented in Fig. 1 for a thin ZrC film deposited on a SiGe/Si sample.

We intend to use the 4.265 MeV resonance to characterize C. Also, we intend to perform an energy calibration of the 3 MV Tandetron accelerator. We intend to use NRBS techniques to characterize the thin layers of the above mentioned materials. The measurements will be performed at the 3 MV Tandetron using a dedicated target chamber. The energy of the ^{4}He beam used for measuremens will be calibrated. The method adopted for calibration consists simply of comparing the energies of alpha particles from a radioactive source (^{239}Pu+^{241}Am) 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 16 keV resolution.

The RBS results will be compared to XPS results. We have used XPS investigations to determine the composition and chemical bonding in these carbides, including a special installation that is using high energy X-rays to sample much thicker regions without the need of Ar ion sputtering. In addition, by RBS analysis one can get the thickness of the films. Taking into account that our group could measure the mass density of the deposited films by using X-Ray Reflectivity, it results that one can get an accurate thickness measurement as well.

Another important result that was obtained recently is the lateral chemical composition that could be obtained since the RBS machine has a good x-y resolution. Based on these results we were able to identify deposition conditions that resulted in 1 to 1 ratio between Si and C or Zr and C. Such samples are extremely important for the investigation of radiation induced defects, since the C overstoichiometry will result in graphite formation, that could degrade the mechanical properties.

We need 14 days (42 shifts) at the 3 MV Tandetron accelerator.