

§7. Progress in MHD Coatings Fabrication by RF Sputtering Method

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Liquid blanket system is attractive blanket concept for DEMO (demonstration) fusion reactor, because liquid tritium breeding material can be continuously replaced for tritium recovery, and it has no radiation damage, larger TBR (tritium breeding ratio) and large heat transferability. Liquid lithium is considered to be one of the best candidate tritium breeding materials for a self-cooled liquid blanket system, in which the liquid lithium is also used as a coolant. Thus, the liquid lithium blanket concept has possibility to provide an economic blanket system with simple structure. One of the critical issues for this blanket concept is so-called MHD (Magneto-Hydro-Dynamics) pressure drop. An intolerably large pumping power for the metallic coolant in the magnetic field will be required due to the pressure drop induced by electric current between the coolant and its pipe wall. In order to reduce this problem, it is proposed to fabricate electrically insulating ceramic coatings (MHD coating) on an inner surface of the pipe wall. These coatings should have high electrical resistivity, high corrosion resistance, high thermomechanical integrity and small irradiation damage. Aluminum nitride (AlN), yttrium oxide (Y_2O_3) and erbium oxide (Er_2O_3) have been chosen as candidate materials for MHD coating by previous investigations on the compatibility with liquid lithium. By the investigations on bulk materials, it is considered that high dense and crystallized coatings are important to maintain high corrosion resistance of candidate materials. RF (radio frequency) sputtering method is a major method to fabricate adhesive and crystalline coatings. In this study, the coatings were fabricated by the sputtering method and immersed in liquid lithium at 573-773 K for 100 h.

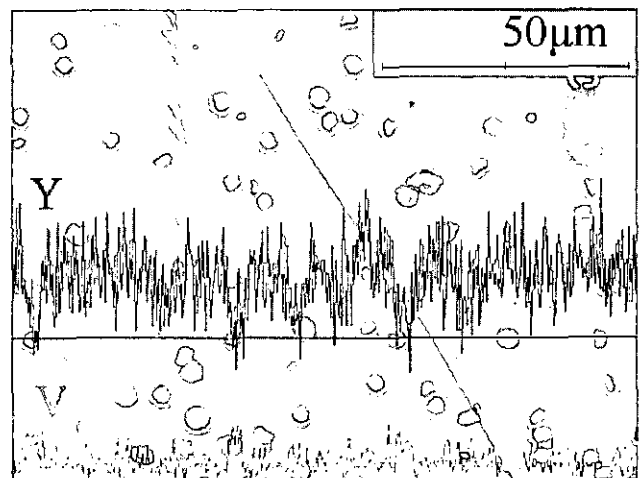
In the sputtering device, a bulk material of AlN, Y_2O_3 or Er_2O_3 whose purity was 99.9 % and diameter was $\phi 60 \times 5$ mm was set as a target material. Substrates which were 10 mm * 10 mm * 0.5 to 1.0 mm size of V-4Cr-4Ti (NIFS-HEAT-II) were cleaned in an ultrasonic bath. The substrates were put into a vacuum chamber and the chamber was evacuated. After exhausted, N_2 gas of 99.995% in purity for AlN coating or Ar gas for Y_2O_3 or Er_2O_3 coating was introduced into the chamber at 0.17 to 0.80 Pa. The substrates were heated up to 423-673K. For the most of the fabrication, the substrates were heated and controlled to 573 K or 673 K. RF electric power to generate plasma was 100-400 W. Distance between the target and the substrates were fixed at 40 mm.

After fabrication, all the coatings had interference fringes, and the coating thickness was 0.3-7.0 μm . Observed by SEM-EDS, the coatings appeared to be smooth and uniform. By XPS (X-ray Photoelectron Spectroscopy) analysis, AlN coatings had almost no impurity except oxygen. The ratio of Al and N was about 1:1, and there was about 3-10 at% of

oxygen in the coatings. In case of Y_2O_3 or Er_2O_3 coatings, there was almost no impurity, and the ratio of Y (or Er) and O was about 2:3. The coatings had crystalline phase observed by XRD (X-ray Diffraction) analysis by Cu-K α . The coatings, however, show a smaller number of peaks than the bulk materials. This indicates the crystal grains grow in specific directions. It was also observed that crystallinity of the coatings was affected by the fabrication temperature. In case of AlN coating, several peaks can be seen in the pattern of the coatings fabricated at 423 K. But, almost no other peak except the peak at 33° degree (100) can be seen in the pattern of the coatings fabricated at 673 K. This suggests that crystal grains grow in several directions at low substrate temperature, and when the temperature becomes high, most grains grow to specific directions.

The resistivities of the coatings were measured at room temperature by the two-probe method with three kinds of electrodes, a copper tape, a silver paste and a platinum layer fabricated by sputtering. Their apparent electrical resistivities were about 10^{13} - 10^{14} Ωm with the copper tape electrode, 10^{10} - 10^{12} Ωm with the silver paste, and 10^3 - 10^7 Ωm with the platinum layer. These differences were due to the difference in electrode configuration: silver paste or platinum particle got into the pits and formed short circuit, while copper tape could not get through or reach to the substrate. Thus the first resistivity value may be the most essential one, and the other two values may become close to the first value as the density or coverage of the coating increased. The apparent resistivities were higher than the required value (10^2 - 10^4 Ωm) in the fusion reactor.

After fabrication, some of the coated specimens were exposed to Li. After exposure, the specimens were removed out from the liquid lithium, and were cleaned in water or vacuum chamber to remove residual Li on the surface. In case of Y_2O_3 and Er_2O_3 coatings, some part of the specimens cleaned in water were peeled off and revealed the substrates, while the specimens cleaned in vacuum chamber were only had cracks without peeling off as shown in figure. This indicates that Y_2O_3 and Er_2O_3 coatings were stable in liquid lithium and they were peeled off by reaction of lithium and water in the cleaning process after exposure test.



Surface of Y_2O_3 coatings after exposure to liquid lithium observed by SEM-EDS