Fabrication of CaO Insulator Coatings by MOCVD for Application in Fusion Reactor Blankets*

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ABSTRACT

The liquid lithium blanket for fusion reactors requires an electrically insulating coating on the duct to minimize the magnetohydrodynamic pressure drop that occurs during the flow of liquid metal in a magnetic field. Calcium oxide (CaO) is a good candidate for the coating material because it is an excellent electrical insulator and it is stable in a liquid lithium environment. In this paper, details are presented on metalorganic chemical-vapor deposition method that was used to fabricate the CaO coating. Composition and phase analyses of the coating were performed by energy dispersive X-ray analysis and X-ray diffraction. Scanning electron microscopy images show that the coating did not crack after several thermal cycles from room temperature to 715°C. The resistance of the coating is high enough for an insulating coating on the liquid lithium blanket of fusion reactors.

INTRODUCTION

In fusion reactor designs, liquid metals are used as a coolant in fusion reactor blankets. The main challenge in the design of self-cooled blankets is to accommodate the strong influence of the magnetic field on the liquid-metal flow. The magnetic field can cause a large electrical current if the duct wall is a conductor. An electrical current flowing perpendicular to a magnetic field results in a mechanical force that leads to a magnetohydrodynamic (MHD) pressure drop. This pressure drop could be as high as 8.6 MPa in a poloidal duct of an inboard blanket segment [1]. Therefore, insulator coatings on the duct wall are needed to reduce the MHD pressure drop.

Lithium-containing liquid metals are attractive materials in fusion reactor blankets. The insulator coatings should be compatible with liquid lithium, which is a very strong reduce reagent. CaO is a good candidate as a coating material since it is a good insulator and its free energy (-128 kcal/mol at 700°C) is lower than that of Li2O (-114 kcal/mol at 700°C). Experiments show that CaO is stable in liquid lithium [2,3].

Metalloragnic chemical vapor deposition (MOCVD) provides a promising method to prepare a CaO coating on a complex channel geometry for the fusion reactor blanket. In this paper, we report the preparation of CaO coatings by the MOCVD method.

EXPERIMENTAL

The CaO coatings were fabricated in a low pressure, cold wall MOCVD system. The MOCVD apparatus used in this study is shown in Fig. 1. Calcium bis(2,2,6,6-tetramethyl-3,5-heptanedionato) [Ca(TMHD)2] is used as a precursor for the CaO coating. The vapor pressure of this chemical is low. To achieve a precursor pressure high enough for the MOCVD coating, the chemical has to be heated over 200°C. However, this precursor starts to decompose at 240°C.
Since the temperature window is so narrow, the vaporizer has to be assembled in the quartz reactor, so that the distance between the vaporizer and the sample heater is kept as short as possible. In that way, the temperature can be easily controlled to avoid the precursor condensing or decomposing.

V-4Cr-4Ti specimens (2 x 1 x 0.2 cm) were heated to 650°C. Ca precursor was carried out with 99.999% Ar and a flow rate of 40 sccm (standard cubic centimeters per minute). The oxygen flow rate was 760 sccm. The pressure in the chamber was 1.7 torr. Specimens were cooled to room temperature at a controlled rate of 5°C/min in an oxygen flow.

Scanning electron microscopy (SEM) and energy dispersive X-ray (EDX) analysis were performed with a JSM-6400 scanning electron microscope. Resistances were measured by a two-probe method in an Ar gas flow from room temperature to 715°C.

RESULTS

Effect of vaporizer temperature on the sublimation rate of precursor and the growth rate of CaO coatings

The vaporizer in our MOCVD equipment is removable. Therefore, the mass of the sublimated precursor could be acquired by measuring the mass loss of the vaporizer. The sublimation rate of the Ca(TMHD)₂ precursor at 225°C is much higher than that at 205°C (Fig. 2). The partial pressure of the precursor should increase with higher vaporizer temperature, which would lead to a higher CaO-coating growth rate (Fig. 3).
Figure 2. Ca(TMHD)$_2$ precursor sublimation rate for Ar carrier gas with flow rate of 40 sccm/min and pressure of 1.6 torr.

Figure 3. CaO coating growth rate for Ar carrier gas with flow rate of 40 sccm/min and pressure 1.6 torr.
Effect of substrate temperature on CaO coatings

When the substrate temperature is 750°C, vanadium alloy is easily oxidized. A vanadium oxide layer was observed between the vanadium alloy and CaO coating. In the liquid lithium environment, vanadium oxide can be reduced by Li, which causes the CaO coating to split. A CaCO₃ impurity formed when the substrate temperature was 550°C. Therefore, 650°C is a good temperature for forming a CaO coating on the substrate of vanadium alloy.

Figure 4 shows a SEM photomicrograph of CaO coated V-alloy specimens. The CaO layer was fairly compact and uniform. No crack was found after 5 thermal cycles from 25 to 715°C. Therefore, thermal expansion did not cause a problem for the CaO coating fabricated by MOCVD. EDX analysis showed almost no vanadium in the CaO coating layer (Fig. 5). This condition is desirable since some vanadium oxides such as V₂O₄ are good conductors and decrease the resistance of the coating if they are in the CaO layer.

The major phase in X-ray the diffraction pattern is CaO (Fig. 6). Sharp diffraction peaks indicate a well crystalline coating. The SEM image (Fig. 4) also shows the cubic shape of the particles, with average grain size ~2 µm. No vanadium oxide was detected by X-ray diffraction. EDX analysis also indicated no vanadium in the film. Therefore, the coating is pure CaO.

The resistance of the coating is too high to measure at room temperature. The minimum value of the product of resistance and the coating area (RxA) is 100 Ω•cm² to obtain an acceptable pressure drop for the fusion blanket. The RxA for a 20-µm CaO coating forming by MOCVD is 2x10⁷ Ω•cm² at 715°C. This value is much higher than the requirement. Figure 7 shows RxA increases with decreasing temperature from 720 to 450°C.
Figure 5. EDX depth profiles for Ca and V of CaO coating.

Figure 6. X-ray diffraction of CaO coating.
CONCLUSIONS

CaO coatings as thick as 40 µm on vanadium alloy are prepared by MOCVD. This provides a possibility to coat CaO on a complex channel geometry for the fusion reactor blanket. X-ray diffraction shows the coatings to be pure CaO. No vanadium impurity was found in the CaO layer from EDX analysis. The CaO layers were fairly compact. No crack was found after 5 thermal cycles from 25 to 715°C. Resistances of the coatings were high enough for the insulator application to reduce the MHD pressure for a fusion reactor.

ACKNOWLEDGEMENTS


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