## TIN COATINGS ON FUEL CLADDING TUBE

A Senior Scholars Thesis

by

## ZHICHAO YU

Submitted to Honors and Undergraduate Research Texas A&M University in partial fulfillment of the requirements for the designation as

## UNDERGRADUATE RESEARCH SCHOLAR

May 2012

Major: Electrical Engineering

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Approved by:

Research Advisor: Associate Director, Honors and Undergraduate Research: Haiyan Wang Duncan MacKenzie

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## ABSTRACT

TiN Coatings on Fuel Cladding Tube. (May 2012)

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In this study, TiN coatings were deposited on HT-9 and MA957 stainless-steel tubes and polished bars. In order to deposit TiN coatings on those certain length tubes, we need to modify our stage in old chamber. We designed a stage which can move along with XYZ axis's direction and also can do rotation with the speed at 10 circles per minute. The flange we designed would be mounted to the 12 inch chamber obtaining ultra-high vacuum. After designing and setting up the instruments, we would demonstrate this new design and deposite TiN on the tubes. A systematic physical property study including surface characterization (SEM), mechanical testing (hardness and scratch test), thermal cycle test and thermal conductivity measurements, was conducted to explore the effects of the coatings on the overall mechanical, thermal stability and thermal conductivity of the fuel cladding materials. A preliminary diffusion barrier study of Ce in HT-9 with and without the coating was also conducted. The results would be fulfilled our expectation.

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Computer Engineering, Texas A&M University.

The introduction and information was provided by Fauzia Khatkhatay from Department

of Electrical and Computer Engineering, Texas A&M University.

# NOMENCLATURE

TiN Titanium Nitride

PLD

Pulse Laser Deposition

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# CHAPTER I INTRODUCTION

Fuel cladding tube development has been one of the key efforts for future advanced nuclear reactor development, including fast burner reactors, high temperature reactors, molten salt reactors, supercritical water reactors, and fusion reactors [1]. Martensitic stainless steels, such as a HT-9 (12Cr-1MoVW) and oxide dispersion strengthened alloys like MA957 (14Cr-0.3Mo-0.27Y2O3), are currently used as fuel claddings for fast burner reactors [1]. They are also considered to be potential candidates for blanket and first wall structures of fusion reactors [2][3]. HT-9 is used in Pb-Bi cooled fast-spectrum reactors at a temperature of  $500 \,^{\circ}$ C [4]. The increased Cr content in HT-9 provides good resistance to atmospheric corrosion and resistance to degradation in many organic media, and molybdenum (Mo) enhances the localized corrosion resistance in environments containing deleterious species by preventing the breakdown of protective oxide films. MA957, mechanically alloyed ferrite steel, has enhanced creep resistance due to the addition of yttria [5], in addition to the benefits of Cr and Mo.

However, the fuel-cladding interaction and interdiffusion under elevated temperatures have been a long term problem that significantly reduces the life time of the fuels and

This thesis follows the style of Applied Physics Letters.

causes safety concerns, especially for high burn up fuels [6][7]. There are several proposed methods for prevention of fuel-cladding interactions. One way is to implement a metal liner inside the cladding tube using a co-drawing process [8].

Another approach is to develop a multifunctional ceramic coating that serves as effective diffusion barrier to prevent the inter-diffusion between the nuclear fuel and the cladding material [7] and therefore, to dramatically enhance the lifetime of the fuels and the reliability of the fuel claddings in advanced nuclear reactors. This thin coating serves many purposes, as a diffusion barrier, a sacrificial layer, as well as a thermal conducting layer. Ultimately, different diffusion barriers can be used for different fuel types while keeping the cladding materials the same. If implemented, this thin layer could dramatically improve the performance (i.e. life time and reliability) of the fuel structure and drastically reduce the cost of service and exchange of the fuel structures. Titanium nitride (TiN) coatings have found numerous applications owning to their excellent corrosion and erosion resistance, relative inertness, high sublimation temperature, high hardness and desirable optical and electronic properties. For instance, hardness and elastic modulus of TiN film had been reported as up to 33.58 GPa and 407 GPa, respectively [9]. In addition, ion irradiation effects in nanocrystalline TiN coatings as a function of grain size was reported previously and it showed that TiN has excellent radiation tolerance properties, and the damage accumulation reduces as the grain size reduces in nanocrystalline TiN films [10]. In addition, the thermal expansion coefficient and thermal conductivity of HT-9 are 12.5×10-6 K-1 and 28 Wm-1K-1, respectively

[11]. TiN, has a thermal expansion coefficient of 9.36×10-6 K-1 and thermal conductivity of 30 Wm-1K-1 [12][13][14]. Based on this, the adhesion between TiN and HT-9 is expected to be excellent.

## **CHAPTER II**

# **METHODS**

### Instrumental

In order to deposit TiN on fuel cladding tube, setting up a PLD( pulse laser deposit FIG.1) is a simple way to focus laser striking on a target forming plasma(FIG.2) plume and then growing on the target, which is also called substrate.



FIG.1. Experimental Setup



FIG. 2. Plasma



FIG. 3. PLD System

The FIG. 3 is the PLD system we used in our research. There are two parts in this system. First, the laser generator in using is Lambda Physik COMPex Pro 205 High-Pulse Energy Exciter Laser (KrF) with maximum pulse energy of 700mJat rate of 50Hz, and average power is 30W.

## Design

The second part in this research is the main chamber where the deposition occurs. The chamber, tube holder and, top flange need to be modified to meet our research . The FIG. 4 is my re-designed top main flange of the chamber:



FIG. 4. Self-designed Main Flange

I made 30 holes on the main top flange and the chamber has 20 holes to be matched. Hence, we can achieve 6 degree difference at each time rotation. The differential degree is stable and small so we could enhance our accurate of deposition.

The draft position picture FIG. 5 is about the location of the holder in the chamber. The picture shows the plane cutting through the center of the chamber. The holder is 1.8 inch off center which plot all the possible location of the holder in a half circle with 1.8 inch radius. The tube we deposited has a range of radius from 0.25 inch to 0.6 inch. In order to avoid block the laser path through the center of the chamber. We only arrange 27 positions for 0.25 inch tubes and 13 positions for 0.6 inch tubes.



FIG. 5. Holder Position Layout

The draft whole design FIG. 6 is listed below:



FIG. 6. 3D Draft Design

The rotation flange holder (FIG. 7) was designed as U shape which can easily insert between rotation flange and flexible below. This design could reduce the volume of whole design and achieve ultra-high vacuum. There are three through holes on this holder which could secure at any height with setting screw and doing linear moving along with the deposition accordingly.



FIG. 7. U-shaped Top Flange Holder

The key of this design is to achieve ultra-high vacuum while the holder is doing 3D motion according to the deposition parameters. The linear motion will be secured by a flexible below and the rotation part is built by instrument with AC motor.

# **CHAPTER III**

# RESULTS



FIG. 8. Assembly Instrumental

The FIG. 8 showed that all the parts are connected as we expected. The following pictures would show each part and how they could obtain ultra-high vacuum while doing the 3D motion with small adjustments on the main flange.



FIG. 9. Self-Designed Part

The FIG. 9 in the picture is flexible and perfectly sealed for ultra-high vacuum. There are 3 rail rack for the U-shaped holder going through, which are mounted to the main self-designed flange. The top flange was secured by the setting screws which ensure the instrument stay stable and horizon.



FIG. 10. AC 10rpm Motor

The FIG. 10 showed the AC motor which would take the holder inside the chamber doing the rotation at 10 RPM while doing the deposition.



FIG. 11. O-Ring

The FIG. 11 is a bronze O ring which would ensure the connection between different part sealed perfectly.

### **CHAPTER IV**

## SUMMARY AND CONCLUSIONS

The instruments we designed connected and worked as we expected. We would demonstrate the deposition on the glass first in order to check and ensure the quality of deposition. After several adjustments, we could find out the right deposition parameters and condition for TiN. In the following days, we are planning to demonstrate this new self-design on TiN coating and observe its processing. Furthermore, we would go further to explore the TiN coating for the application of fuel cladding tube.

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