

# STUDY OF THE NON-EVAPORABLE Ti-Zr-V FILM GROWN ON DIFFERENT MATERIALS\*

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## Abstract

The non-evaporable (NEG) Ti-Zr-V films were coated on the different vacuum-chamber materials, including the extruded aluminum samples (Al), the extruded seamless stainless steel samples (S.S.), CuCrZr alloys, and oxygen-free copper (OFC) plates. The NEG films were fabricated by using the direct current (DC) sputtering method. The secondary electron microscopy images showed that the morphology of NEG films was different on these various substrates. The thermal analysis (TA) presented that exothermic reaction happened by heating the samples.

## INTRODUCTION

Because the contribution of surface outgassing is evident for an ultrahigh-vacuum (UHV) vacuum chambers, much effort has been studied to decrease surface outgassing using an appropriate method. As we know, the residual gases, including H<sub>2</sub>, CO, CO<sub>2</sub> and CH<sub>4</sub>, still exist after baking. Additionally, a thin-film coating was thought as the good method to decrease thermal outgassing and outgassing induced by particles bombardment.

As these gaseous molecules contact a surface, multiple activities happen, such as adsorption, desorption, surface reaction, backscattering, diffusion, and replacement [1]. While the gases adsorption occurs, the pressure becomes decreased. In order to develop the materials to adsorb gases, the most important properties of these materials are both chemical affinity and bulk diffusivity. Therefore, it was reported that thin films with many species or compounds can be selected for improving the performance of gas adsorption.

A non-evaporable getter (NEG) thin film is used to pump a vacuum chamber with limited conductance. The pumping mechanism of the NEG films for residual gases includes surface adsorption followed by bulk diffusion. However, inert gases and methane are not pumped by NEG [2]. NEG films have been developed at CERN for use in vacuum chambers of a particle accelerator since 1995 [3,4]. Recently, the ring beam pipes of MAX IV have been coated with Ti-Zr-V NEG thin film to fulfil the average pressure requirement of  $1 \times 10^{-7}$  Pa. Previous reports suggested that a NEG film with Ti, Zr and V in the appropriate combination has the lower activation temperature [5,6].

Here, the NEG film was deposited on a substrate by a sputtering method due to its simple and wide application for varied materials and alloys. For each coating experiment, small samples were produced on these

materials that included the extruded aluminium samples (Al), extruded seamless stainless steel (S.S.), CuCrZr alloys, and oxygen-free copper (OFC) sheets. Those materials are applied or designed in the vacuum chambers of the booster and storage rings of Taiwan Photon Source (TPS). We compared the surface morphology and thermal analysis of NEG films on these materials.

## EXPERIMENTS

The NEG Ti-Zr-V films were coated on various substrates by DC (direct current) sputtering method. Before deposition, the substrates were cleaned separately by the standard procedure, as listed in Table 1.

The deposition parameters of NEG films were DC power, the pressure of sputter gas (Ar), deposition thickness, and deposition rate, as shown in Table 2.

Table 1: The Substrate Cleaning Procedure

	Surface Cleaning Procedure
Al	Soap, NaOH, HNO <sub>3</sub> +HF
S.S.	Soap, NaOH, HNO <sub>3</sub> +HF
CuCrZr	Soap, Citranox
OFC	Soap, Citranox

Table 2: The Deposition Parameters of NEG Films

	Deposition Parameters
Power	250 W
Ar pressure	0.53 Pa
thickness	0.5-1 μm
Deposition rate	0.034 nm.s <sup>-1</sup>

The surface morphology, cross-sectional structure, and composition of the films were investigated by the field emission scanning electron microscope (FESEM, JSM-6500F). The thermal analyzer (TA, Mettler-Toledo, 2-HT) and gas chromatography-mass spectrometry (GC-MS, Agilent, 7890GC/5975 MSD) were measured to explore reaction and thermal desorption gas of the Ti-Zr-V films.

## RESULTS AND DISCUSSION

The surface morphology of Ti-Zr-V films, grown on Al, S.S., CuCrZr, and OFC substrates, are shown in Figure 1(a)-(d), respectively. For Al substrates, the NEG films have porous structures. The small pores, in the range of

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50 -100 μm, are surrounded with the large holes. For S.S. samples, the NEG films appear the flat surface with a few scratches and concaves. For CuCrZr case, the NEG films have the rough surface with large fluctuation in height. For OFC samples, the NEG films are smooth with the polished direction. The NEG films on these four materials have different surface morphologies. The cross-sectional structure of a NEG film is indicated in Figure 2. The NEG film has a columnar structure.

Figure 3-6 indicated the composition and spectrum of the NEG films on Al, S.S., CuCrZr, and OFC substrates, respectively. The acquired data are detected from the selected area. The NEG films consisted of Ti, Zr, and V.

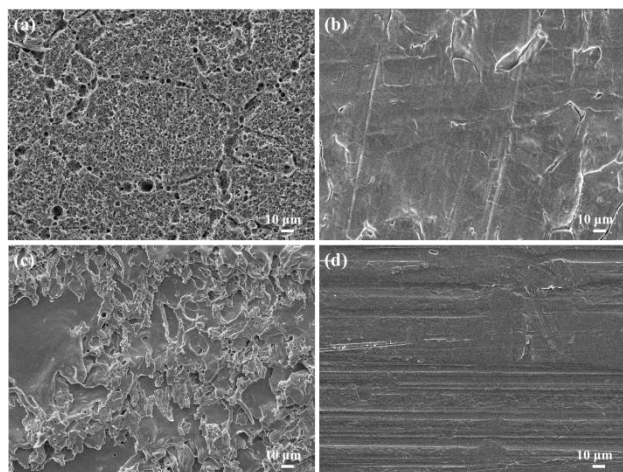


Figure 1: The surface morphology of Ti-Zr-V films on (a) Al, (b) S.S., (c) CuCrZr, and (d) OFC substrates.

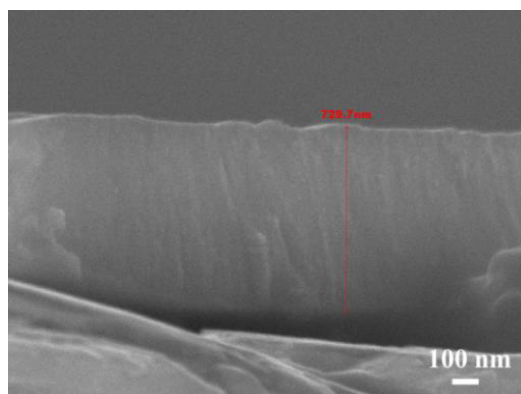


Figure 2: The cross-sectional structure of a NEG film.

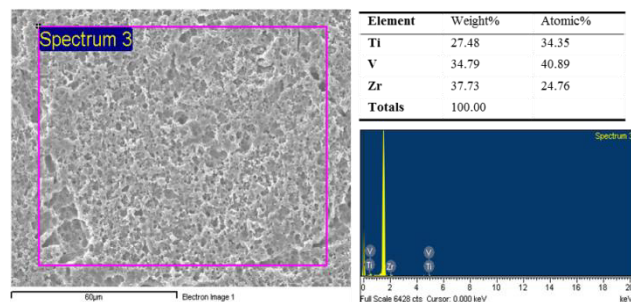


Figure 3: The composition and spectrum of the NEG films on Al substrates.

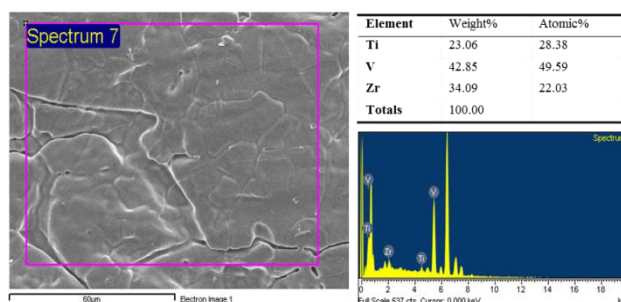


Figure 4: The composition and spectrum of the NEG films on S.S. substrates.

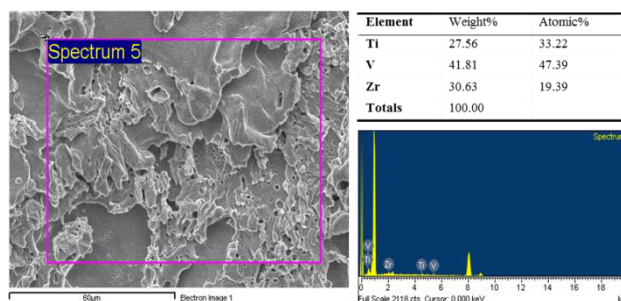


Figure 5: The composition and spectrum of the NEG films on CuCrZr substrates.

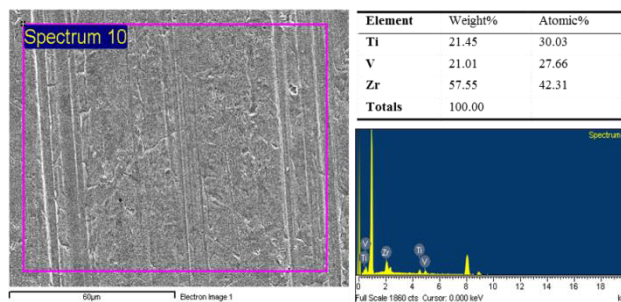


Figure 6: The composition and spectrum of the NEG films on OFC substrates.

The thermal properties of the NEG films on the S.S. and OFC substrates were measured with a thermal analysis (TA), including differential scanning calorimetry (DSC) and mass spectrometer (MS). For DSC experimental procedure, the samples (3 mm × 3 mm) were heated from 30 °C to 420 °C at rate 10 °C/min. The MS data were acquired at 100 °C, 200 °C, and 350 °C. Figure 7-8 show the DSC and MS results of S.S and OFC substrates, respectively. As the NEG films were heated, the enthalpy was changed, shown in the DSC curves. For the case of NEG films on the S.S. and OFC substrates, the enthalpy decreased and exothermic reaction happened by heating the samples. The enthalpy variation for OFC substrates is greater than that for S.S. substrates. The exothermic reactions generally could result from gas adsorption, phase transformation, dissolution, and some chemical reactions. In our case, the gas adsorption could happen during thermal activation of NEG films.

Besides, the gas desorption can be investigated by MS measurement. By means of the library database of the TA software, the outgassing source is contributed from the mass 44. It is suggested that the mass 44 presented CO<sub>2</sub>.

However, the reason why CO<sub>2</sub> outgassing produced is still not clear.

### CONCLUSION

This paper primarily focused on the non-evaporable (NEG) Ti-Zr-V films on the various materials, which are applied in vacuum chambers or vacuum devices. The substrates include the extruded aluminum samples (Al), the extruded seamless stainless steel samples (S.S.), CuCrZr alloys, and oxygen-free copper (OFC) sheets. The surface morphologies of NEG films were distinct on these various substrates. The thermal analysis (TA) showed that exothermic reaction was observed and mass 44 (CO<sub>2</sub>) outgassing was produced by heating the samples.

### REFERENCES

- [1] J. R. Anderson, *Structure of Metallic Catalysts. Academic*, New York, 1962.
- [2] B. Ferrario, *Vacuum* 47 363, 1995.
- [3] C. Benvenuti, P. Chiggiato, F. Cicoira F, Y. L'Aminot, *J. Vac. Sci Technol, A* 16 148, 1998.
- [4] C. Benvenuti, P. Chiggiato, F. Cicoira, V. Ruzinov, *Vacuum* 50 57, 1998.
- [5] C. Benvenuti, P. Chiggiato, P. Costa Pinto, A. Escudeiro Santana, T. Hedley, A. Mongelluzzo, V. Ruzinov, I. Wevers, *Vacuum* 60 57, 2001.
- [6] A. E. Prodromides, C. Scheuerlein, M. Tadorelli, *Vacuum* 60 35, 2001.

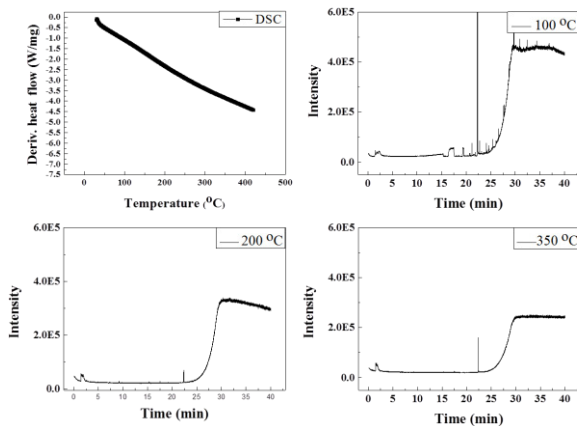


Figure 7: DSC and MS results of S.S. substrates.

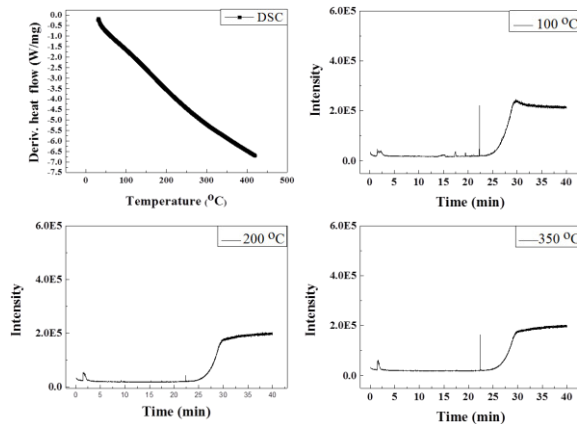


Figure 8: DSC and MS results of OFC substrates.