

Spring 4-14-2015

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Diletta Sciti

*UHTCMCs: short vs continuous fibers, Italy*

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## Recommended Citation

Diletta Sciti, Yeon Woo Yoo, and Sea Hoon Lee, "Characterization of Ultra High Temperature Ceramic Coatings Deposited by Vacuum Plasma Spraying" in "Ultra-High Temperature Ceramics: Materials for Extreme Environment Applications III", G. Franks and C. Tallon, University of Melbourne Eds, ECI Symposium Series, (2015). <http://dc.engconfintl.org/uhtc-iii/11>

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# Characterization of Ultra High Temperature Ceramic Coatings Deposited by Vacuum Plasma Spraying

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## Introduction

- Materials for Extreme Environment Applications
- UHTC Coatings
- Thermal Spraying & Vacuum Plasma Spraying

## Experimental

## Results and Discussion

- Properties of TaC Coatings Deposited by Vacuum Plasma Spraying at Various Condition
- Properties of HfC Coatings Deposited by Vacuum Plasma Spraying at Various Condition

## Summary

## Materials for Extreme Environment Applications

### Refractory Metals

- High temperature strength
- Good wear & erosion resistance
- Low oxidation resistance
- High density
- High cost

### Carbon

- High mechanical strength
- Highest melting temperature
- Low density
- Vulnerable to oxidation at low temperature

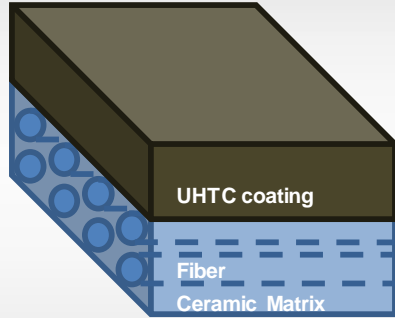
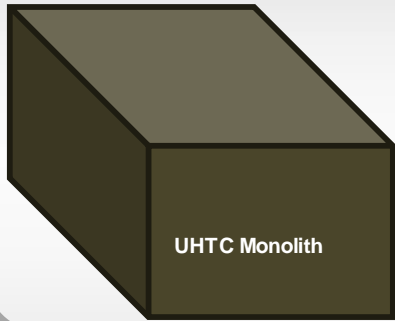
### Ceramic Matrix Composites (SiC)

- High mechanical strength
- High oxidation resistance below 1700 °C (Formation of SiO<sub>2</sub> glass on the surface)
- Low density

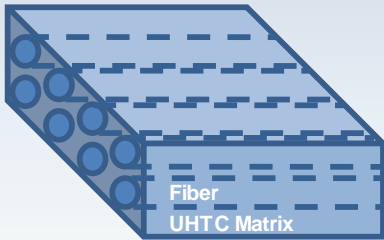
### Ultra High Temperature Ceramics

- High melting temperature (up to 3000 °C)
- Good chemical stability at high temperature
- Easily oxidized
- High density

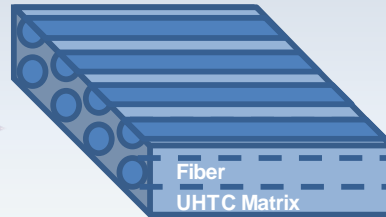
## Why UHTC Coatings?



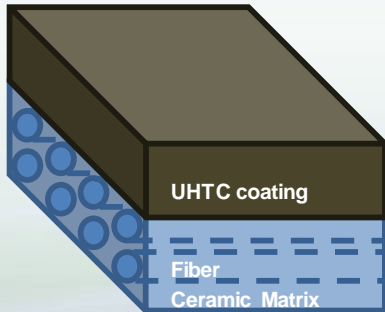
- UHTC monoliths weigh too much because of their high density.
- UHTC coatings can reduce the weight.



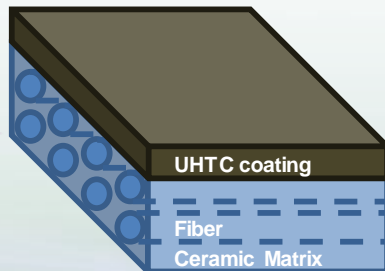
After Ablation



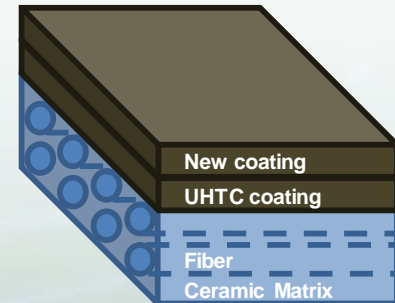
- UHTC coatings prevent damage of CMC from ablation and oxidation.
- Easy to repair damaged coating.



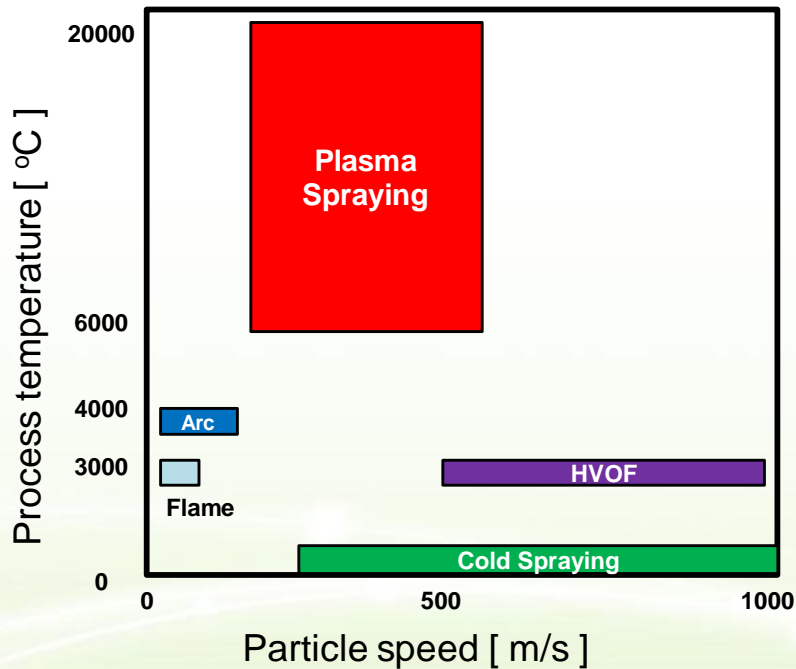
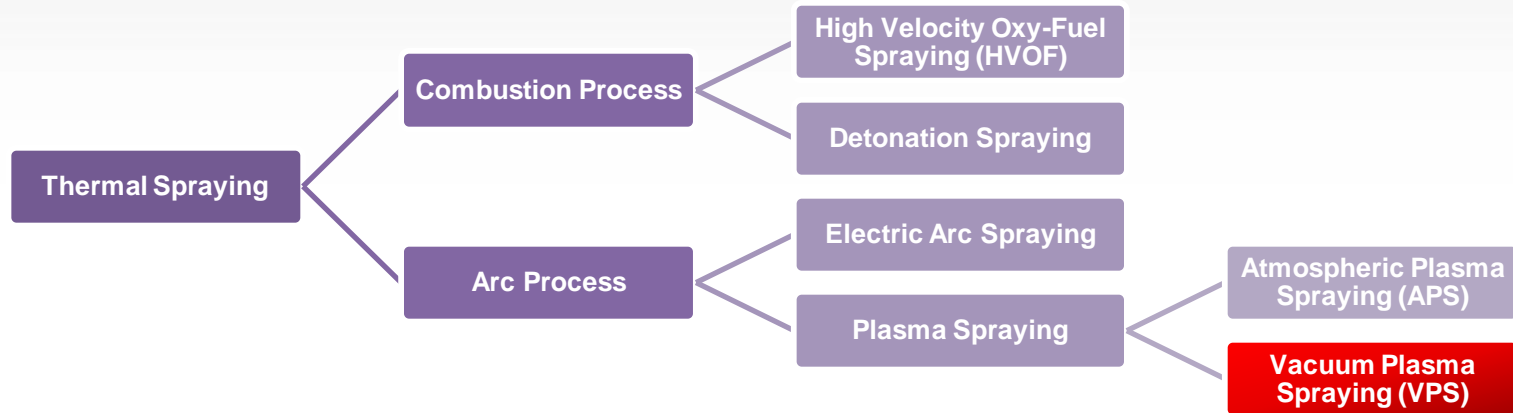
After Ablation



Additional Coating

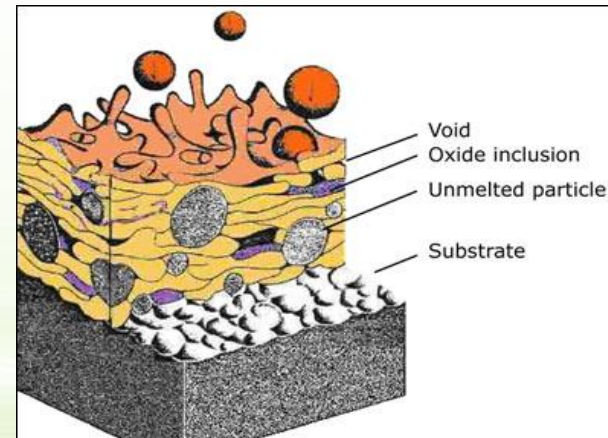
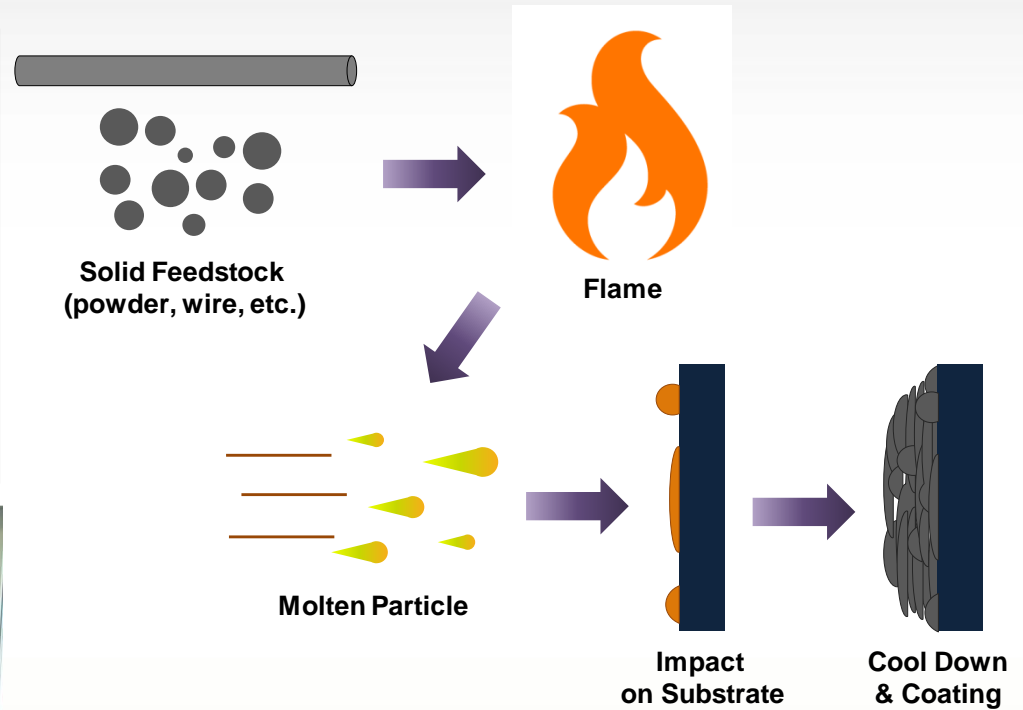
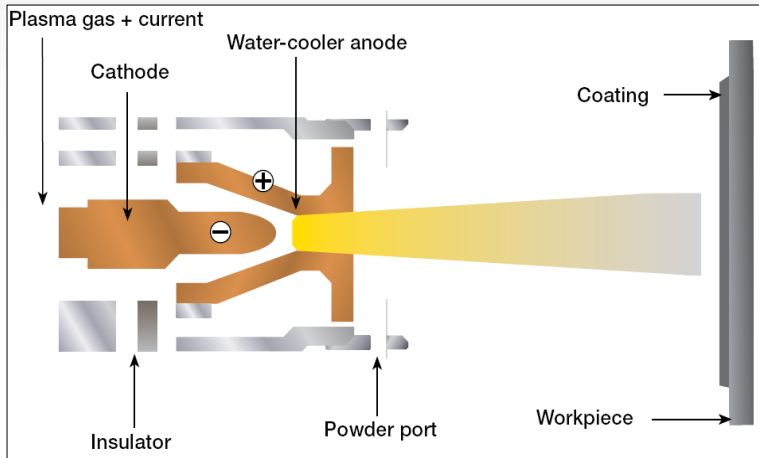


## What Is Thermal Spraying?





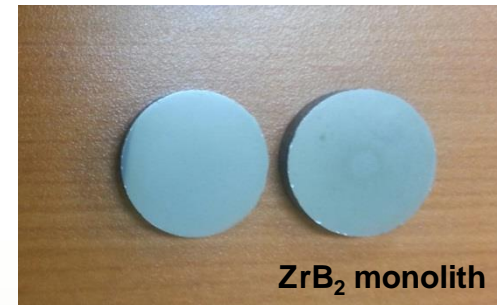
## Vacuum Plasma Spraying (VPS)





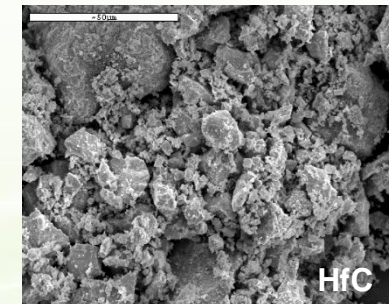
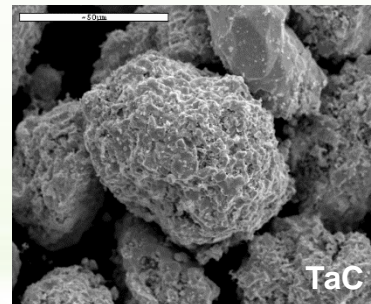
## - Plasma Flame Conditions

- ◆ Primary gas : Ar
- ◆ Secondary gas : H<sub>2</sub>
- ◆ Ar to H<sub>2</sub> ratio : 10 to 3
- ◆ Atmosphere gas : N<sub>2</sub>



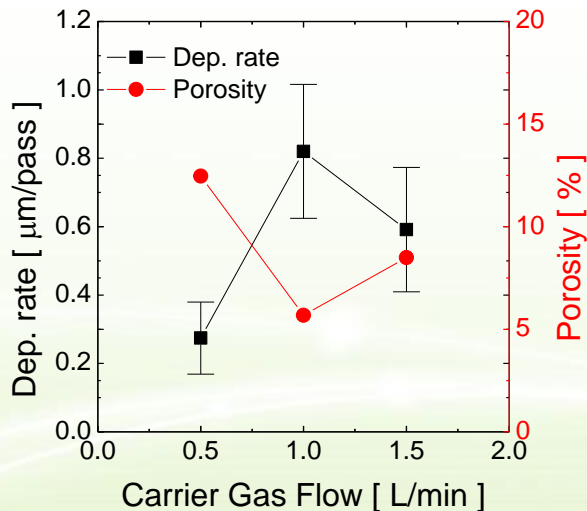
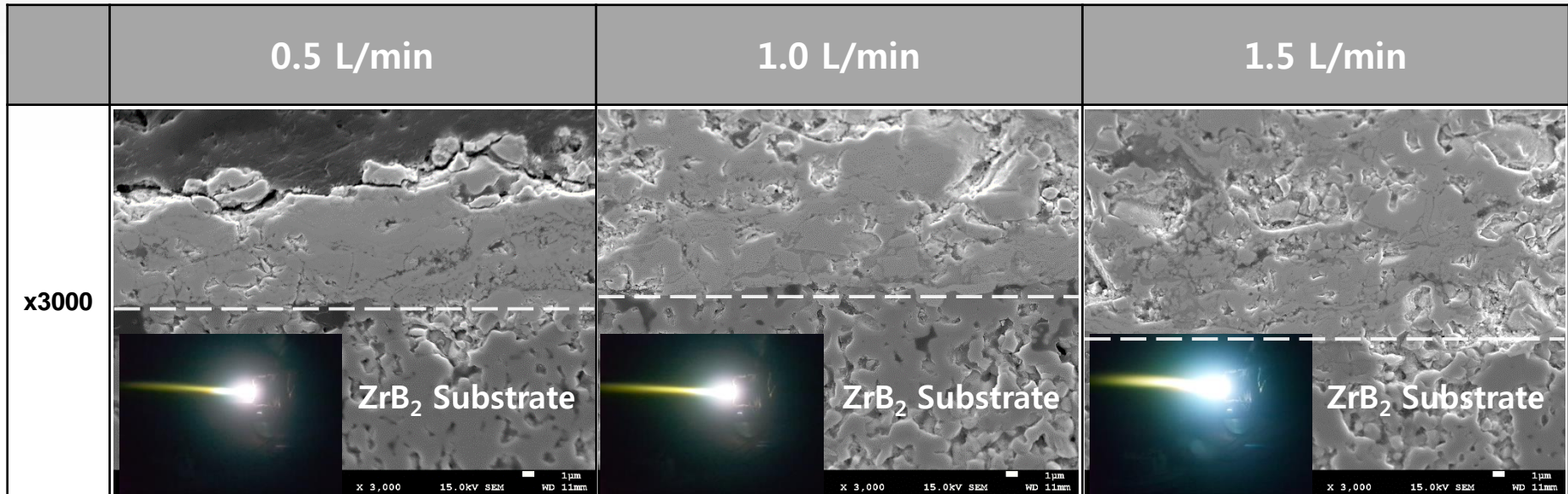
## - Experimental Conditions

- ◆ Feeding powder : TaC and HfC powders  
( size distributed )
- ◆ Substrate : Sintered ZrB<sub>2</sub> monolith
- ◆ Various working pressure
- ◆ Various carrier gas flow





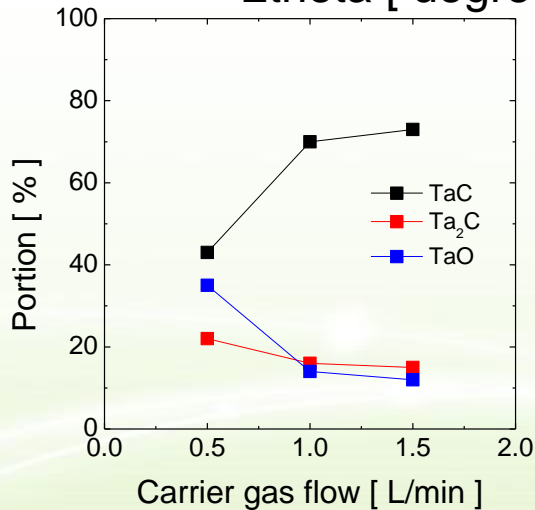
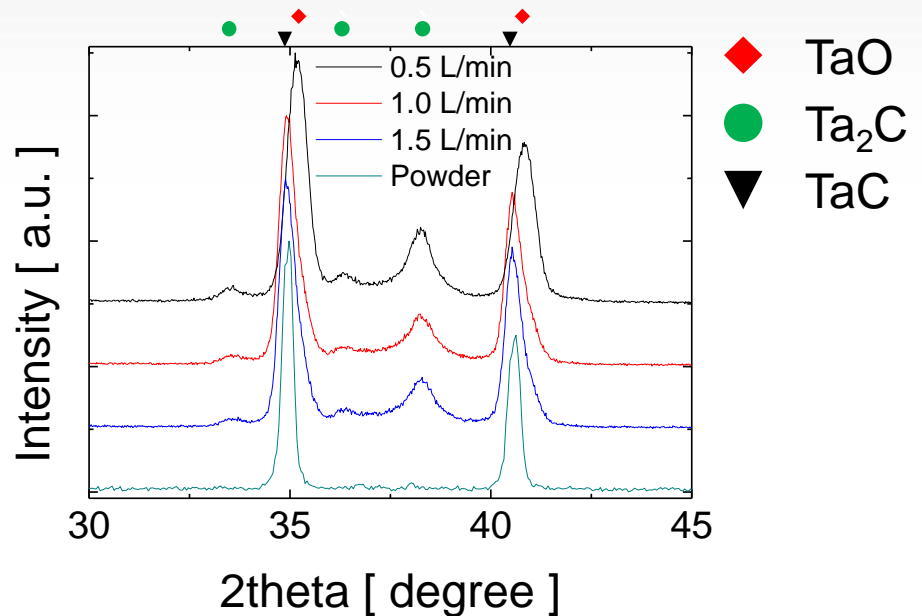
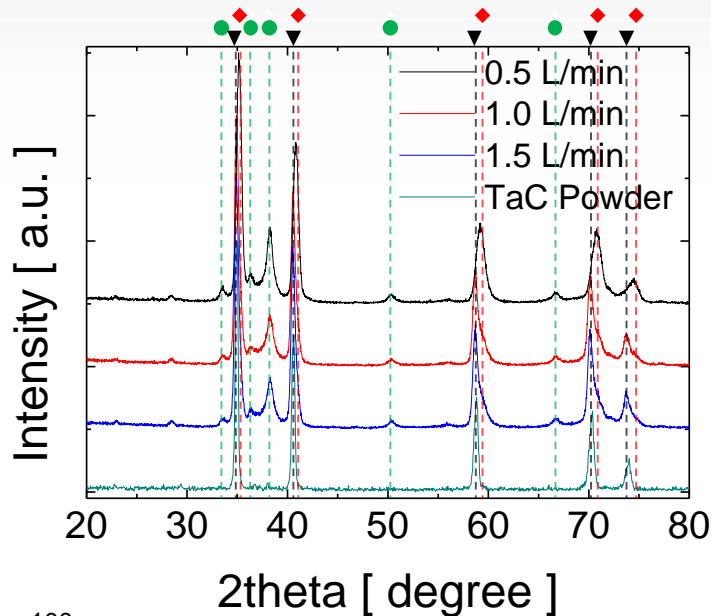
## Microstructure of TaC Coatings at Various Carrier Gas Flow



- Working pressure – 150 mbar

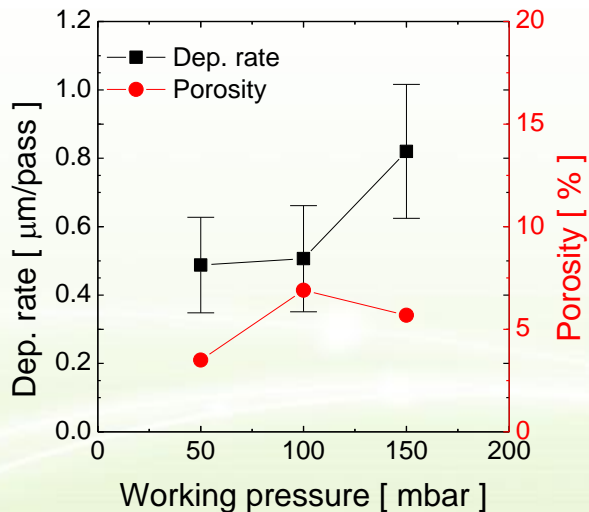
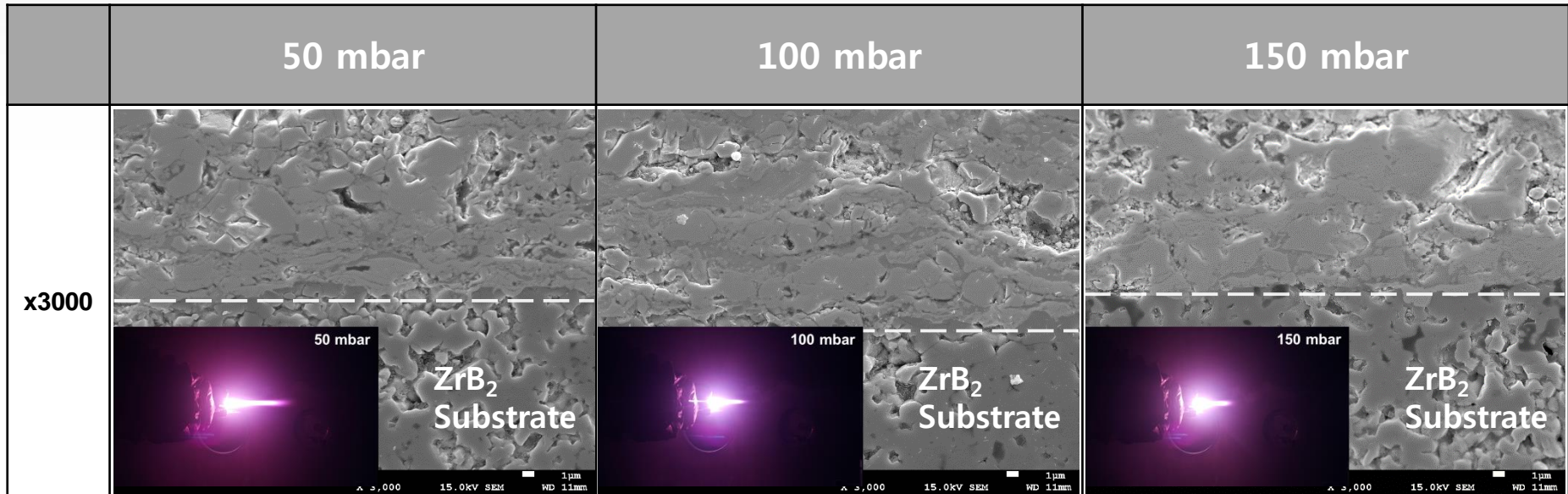
- TaC coatings adhered well to surface of ZrB<sub>2</sub> substrates.
- The highest deposition rate and lowest porosity TaC coating was obtained through proper carrier gas flow level.

## Crystal Structure of TaC Coatings at Various Carrier Gas Flow



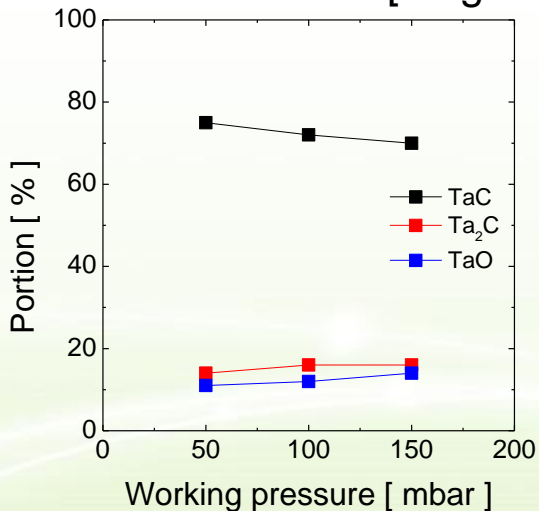
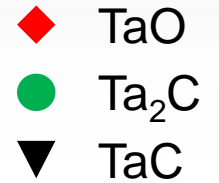
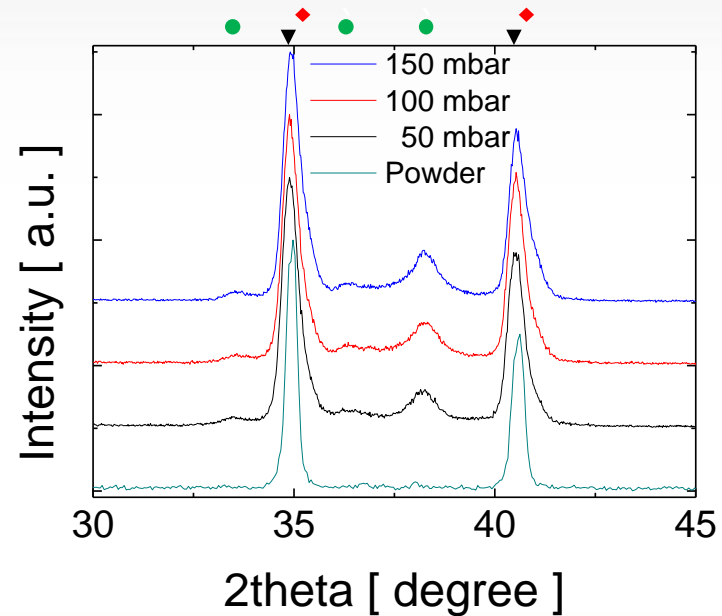
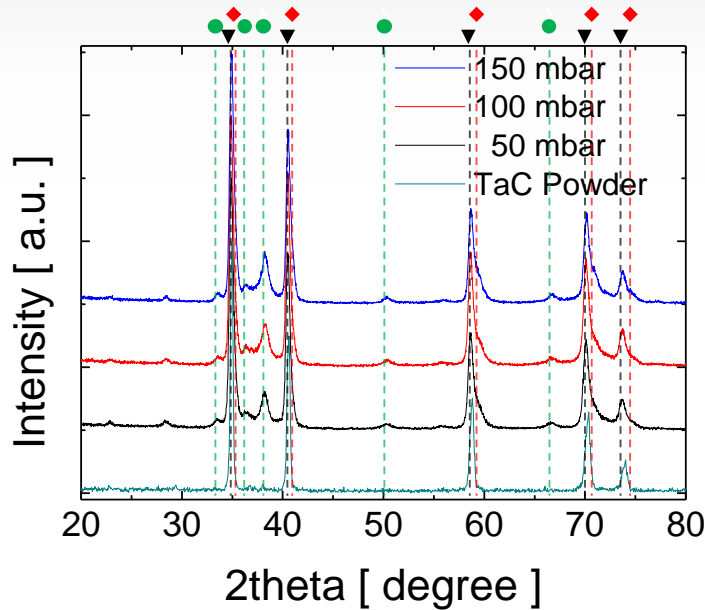
- Decarbonization of TaC occurred during plasma spraying.
- TaO formed during spraying.
- Oxidation of TaC occurred well when carrier gas flow level was not enough for injecting powders into the flame.

## Microstructure of TaC Coatings at Various Working Pressure



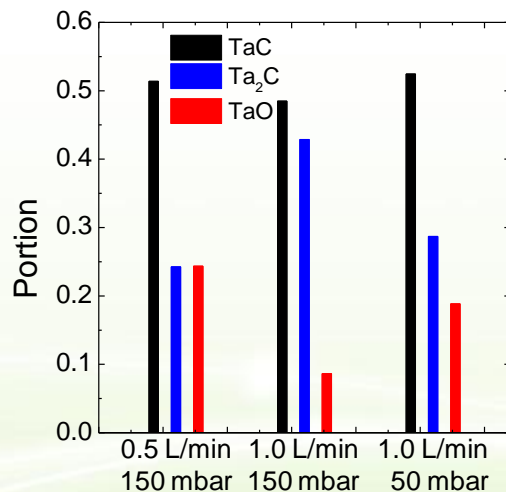
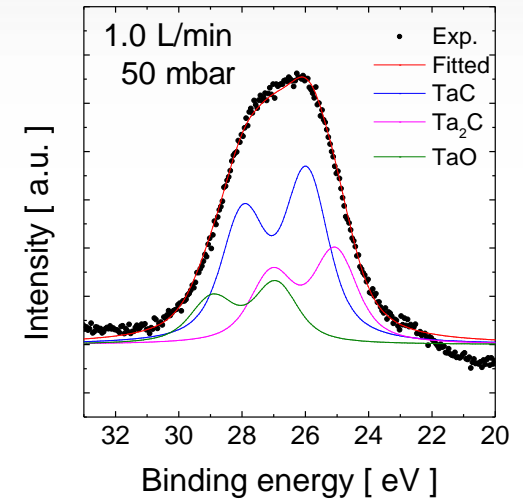
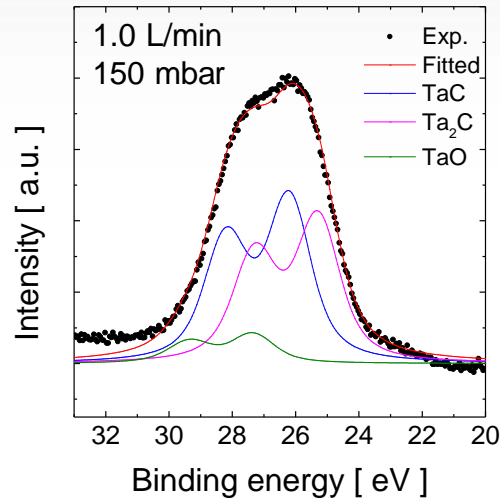
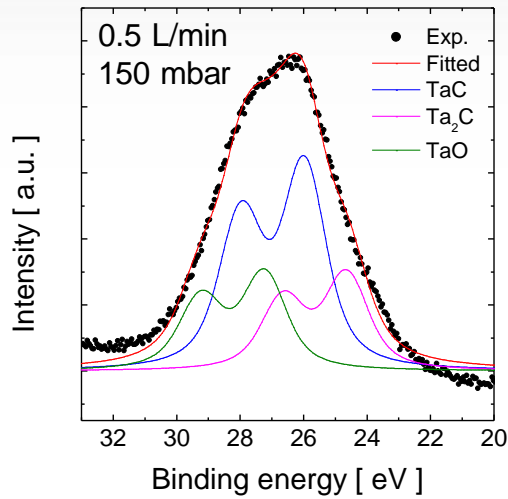
- As changing working pressure, the shape of plasma flame changes.
- Flatter splats were deposited in lower working pressure because of bigger plasma flame.

## Crystal Structure of TaC Coatings at Various Working Pressure



- Content of TaC slightly increased as decreasing the working pressure.

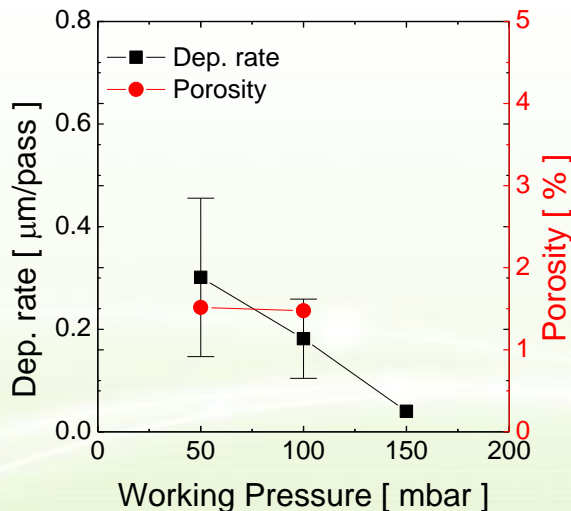
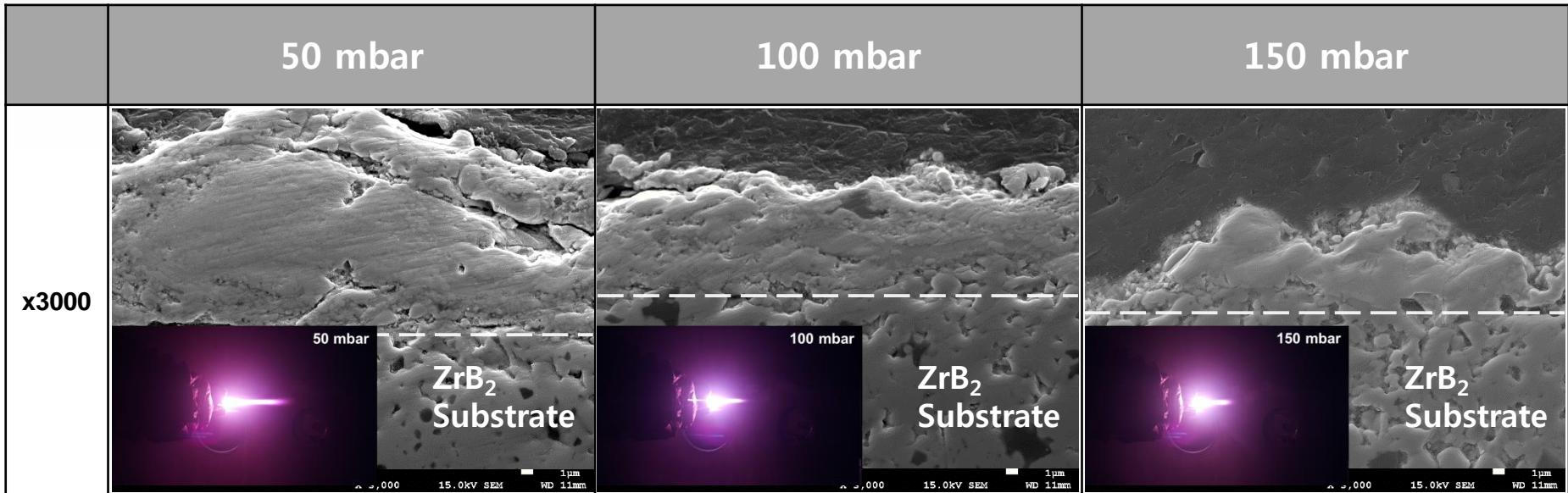
## XPS analysis of TaC Coatings



- Same as XRD analysis, the portion of TaO decreased when powders are injected into the plasma flame.
- Portion of Ta<sub>2</sub>C decreased when working pressure decreased.



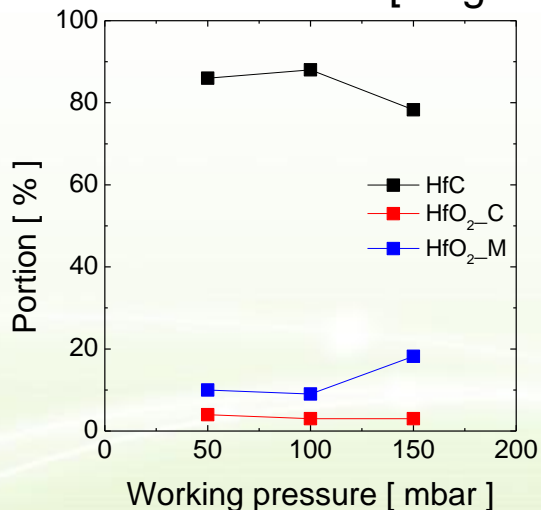
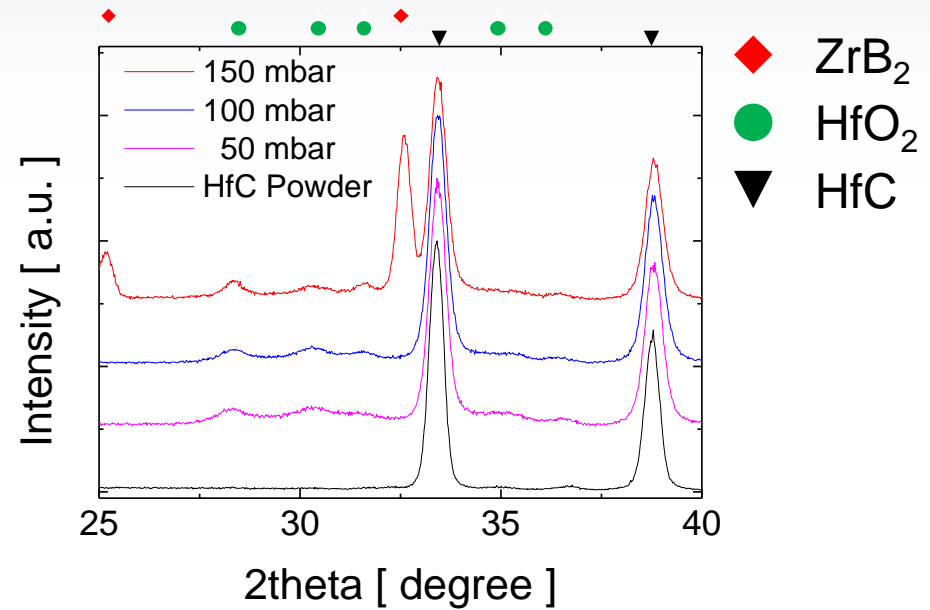
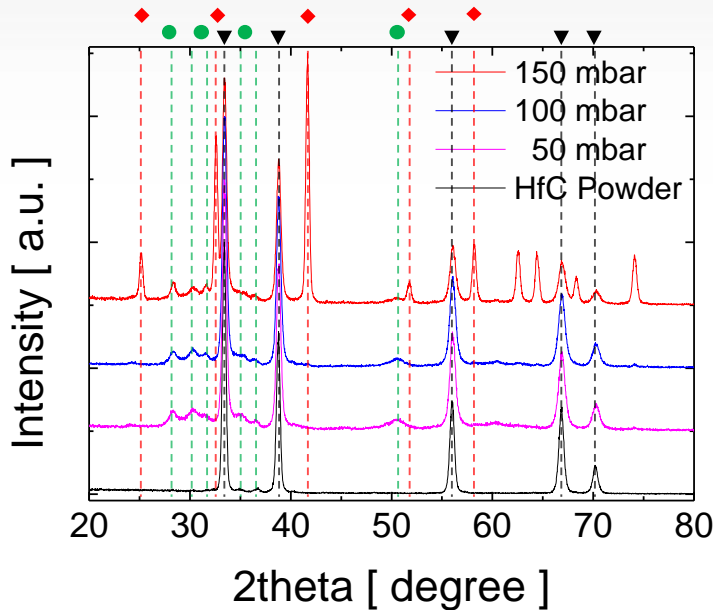
## Microstructure of HfC Coatings at Various Working Pressure



- Carrier gas flow – 0.6 L/min

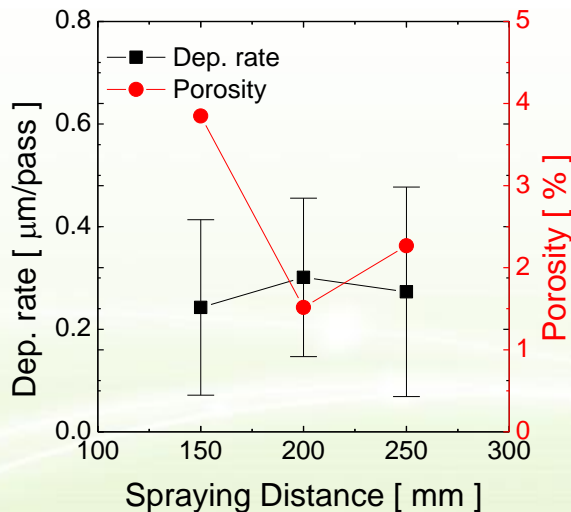
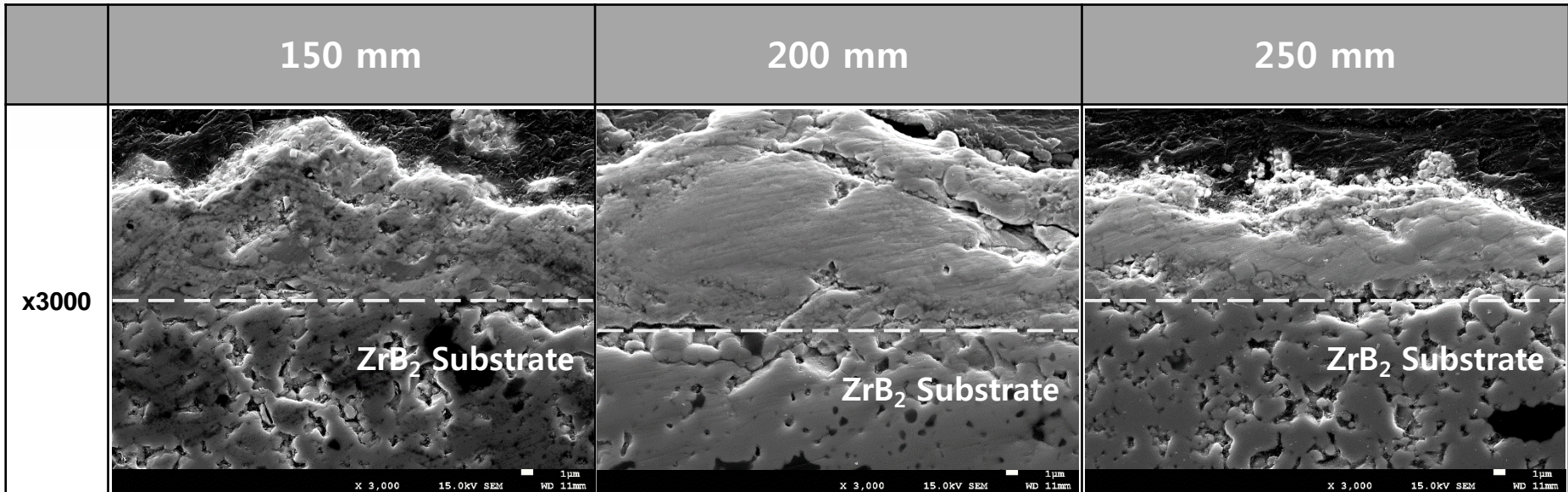
- As increasing the working pressure during HfC spraying, the coating thickness decreased.
- The deposition rate and porosity of HfC coatings decreased compared to TaC coatings.

## Crystal Structure of HfC Coatings at Various Working Pressure



- HfC coatings including various phase of HfO<sub>2</sub> deposited at all working pressure.
- Little amount of HfC was coated on ZrB<sub>2</sub> at 150 mbar, so XRD spectrum showed peaks of ZrB<sub>2</sub>.

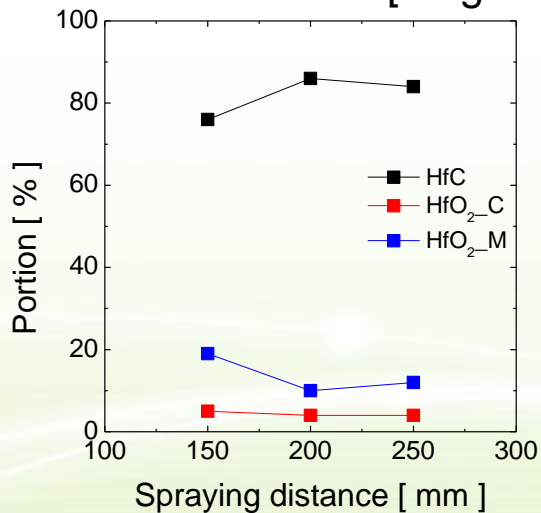
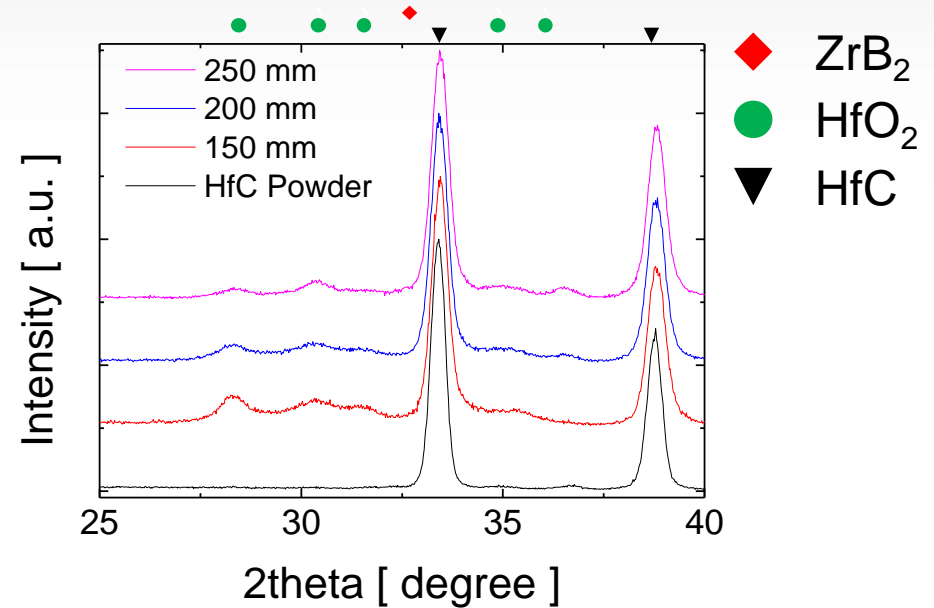
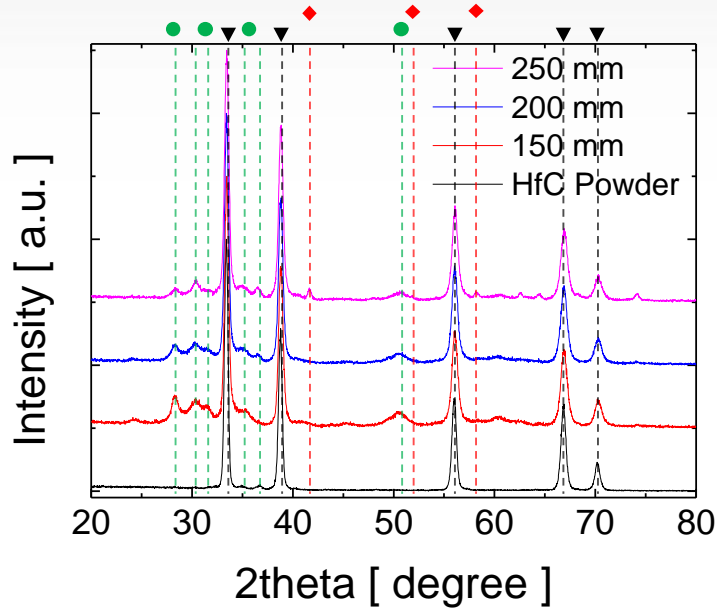
## Microstructure of HfC Coatings at Various Spraying Distance



- Working pressure – 50 mbar

- Porous HfC coatings were deposited when spraying distance was not enough.
- No big difference of deposition rate between various spraying distance.

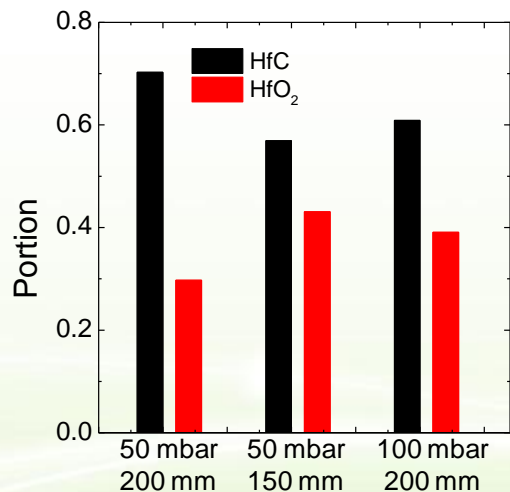
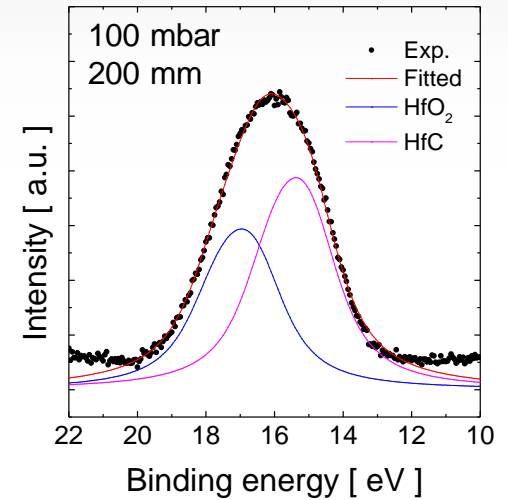
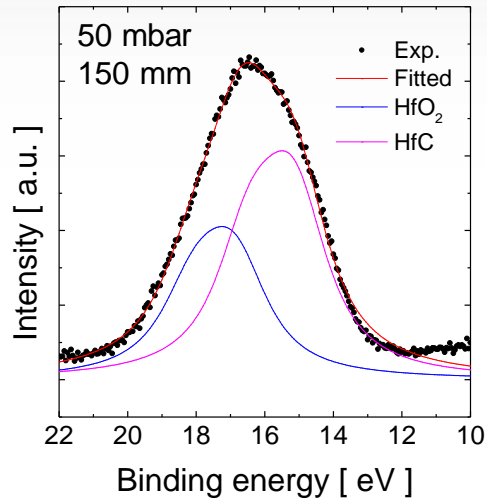
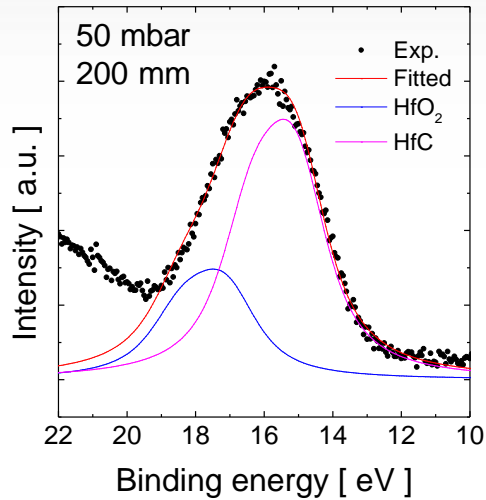
## Crystal Structure of HfC Coatings at Various Spraying Distance



- As increasing spraying distance, the content of HfO<sub>2</sub> decreased.



## XPS analysis of HfC Coatings



- The portion of oxide in HfC coating increased when spraying distance decreased.
- HfC coating deposited at low working pressure showed lower oxide content.



## TaC Coatings Deposited by VPS

- TaC coatings with TaO and Ta<sub>2</sub>C were deposited by VPS
- Enough carrier gas flow was key parameter for reducing oxidation of TaC.
- Deposition TaC coating at low working pressure decreased carbon loss and made flatter splats.

## HfC Coatings Deposited by VPS

- HfC coatings with various phases of HfO<sub>2</sub> were deposited by VPS.
- Coatings deposited at low working pressure showed lower oxide content.
- More oxide content in HfC coatings deposited when spraying distance is shorter than significant distance.

## Comparison between Two Carbide Coatings

- TaC coatings showed that easier to oxidize than HfC coatings.
- Deposition rates of TaC coatings were higher than those of HfC coatings.
- TaC coatings showed more porous microstructure than HfC coatings.

**Thank you  
for  
your attention!**

