A Plasma Approach to Superhard Tantalum Borides

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Introduction

Tantalum borides have become a subject of interest due to their excellent properties: high hardness, wear resistance, chemical inertness, among others. Previously studied methods of boriding (powder boriding, diamond anvil cell, sintering, chemical vapor deposition (CVD) coating) have drawbacks: contamination, limited volumes, extreme conditions, or coating delamination.

In this study, we use a microwave plasma CVD (MPCVD) system to boride tantalum substrates, with a feedgas mixture of diborane (B_2H_6) and hydrogen gas (H_2). This presents a **novel method** that relies solely on diffusion of boron into the substrate without introducing contaminants.

We vary two growth parameters – substrate temperature and substrate bias – in order to understand the effect on structure and hardness. Our motivation for the use of bias is based on prior studies on CVD coatings reporting that ion bombardment mechanisms lead to enhanced hardness.

Methods

Reactor conditions:

- Pressure = 15 Torr
- Diborane flow rate = 0.6 ± 0.15 sccm
- Hydrogen flow rate = 500 sccm
- Reaction time = 6 hours

Characterization Techniques:

- X-ray Photoelectron Spectroscopy (XPS)
- X-ray Diffraction (XRD)
- Nanoindentation
- Scanning Electron Microscopy (SEM)

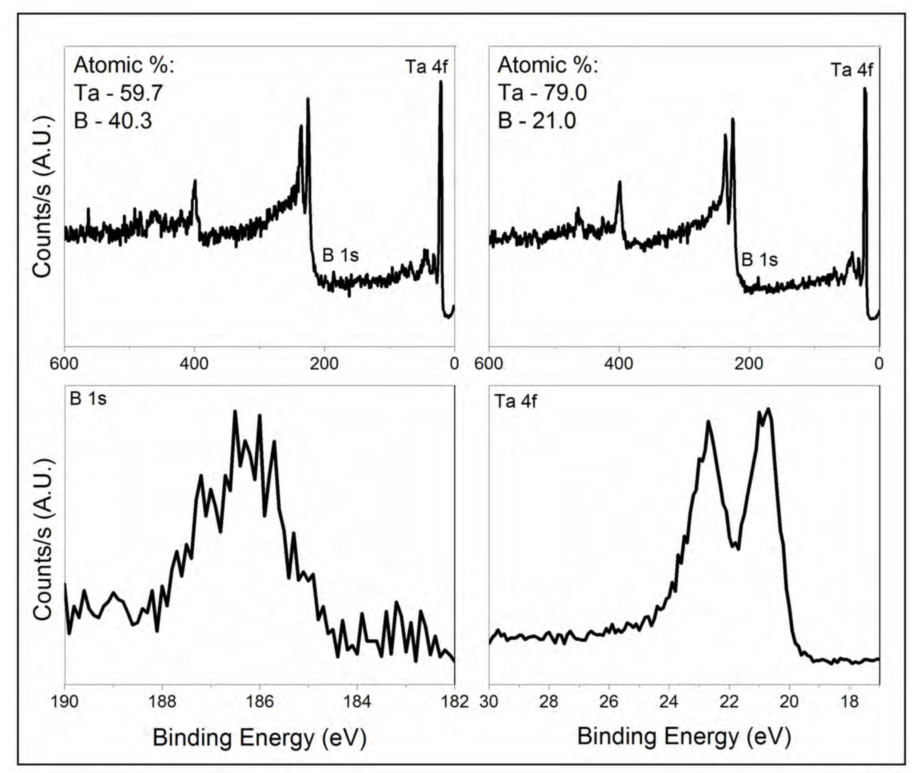
Sample ID	Power/Reflected Power (W)	Approx. Average Temperature (°C)	Applied Bias (V)
A	600/50	700	0
B	700/50	775	0
C	1000/20	850	0
D	600/80	800	-50
E	600/30	800	-150
F	600/40	750	-250
G	600/50	775	-350

Table of sample conditions.

Results

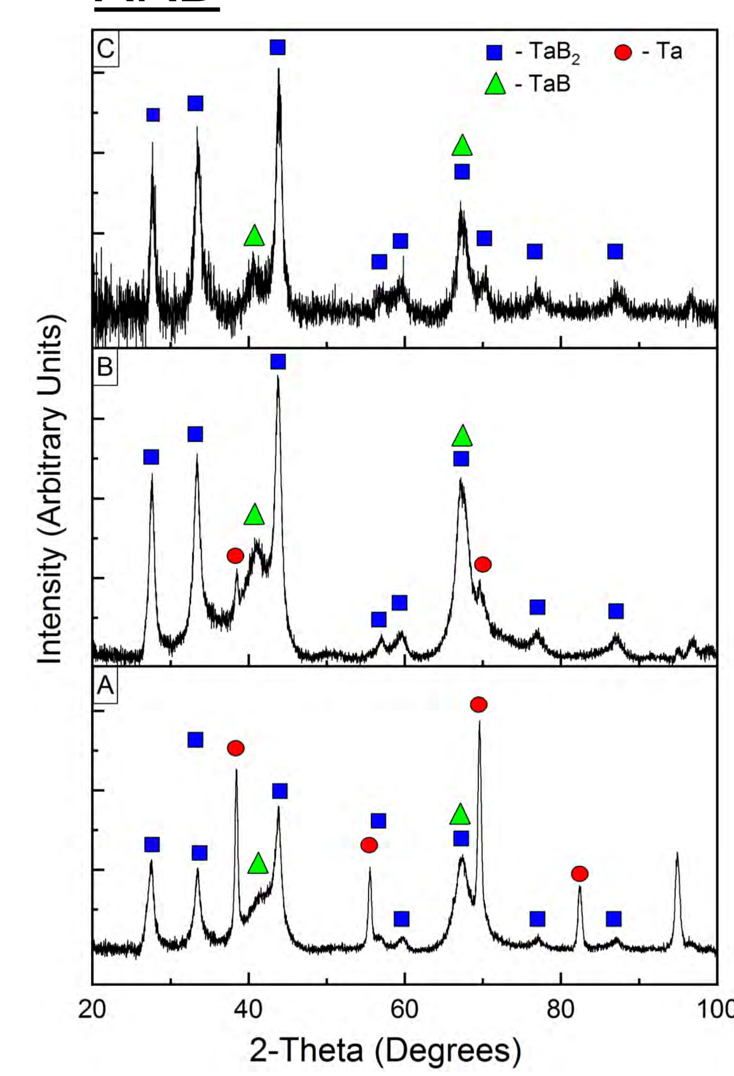
Temperature Set (A, B, C)

XPS

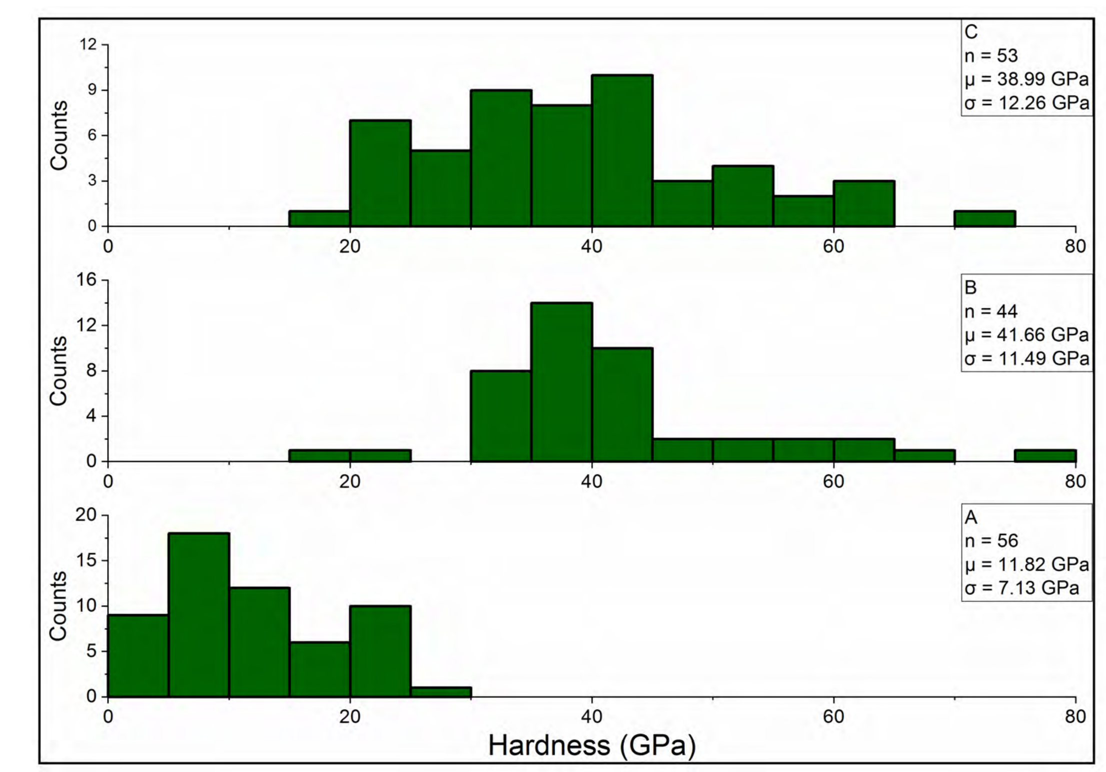


XPS survey scans from two close regions (top), with high resolution scans of boron and tantalum (bottom) showing no Ta-B bonding. However, the XRD patterns (right) show the presence of boride phases.

XRD



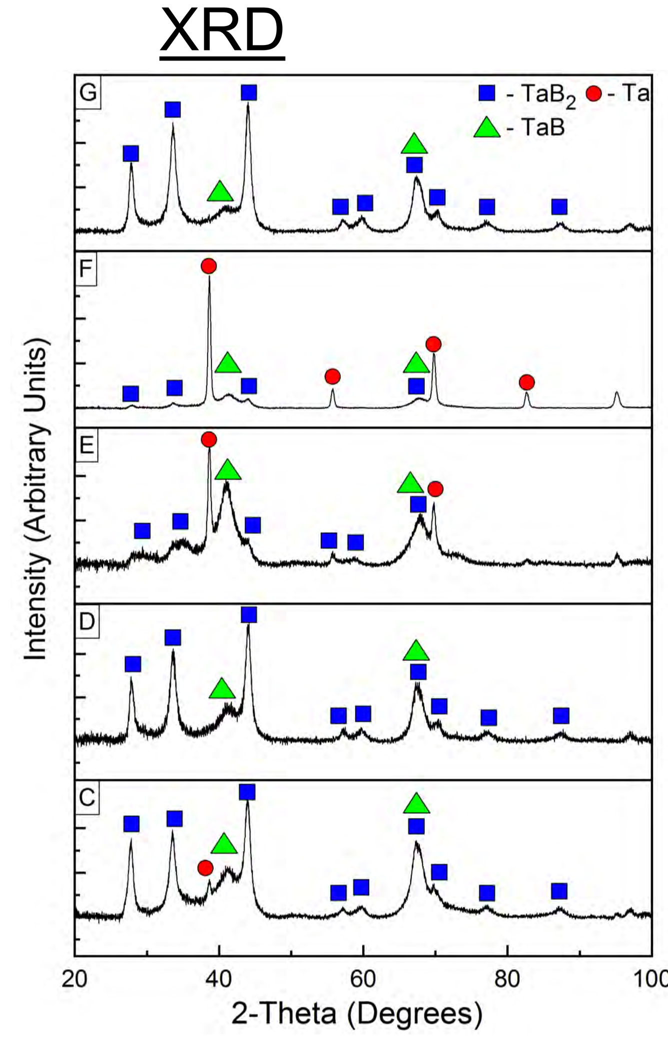
Nanoindentation



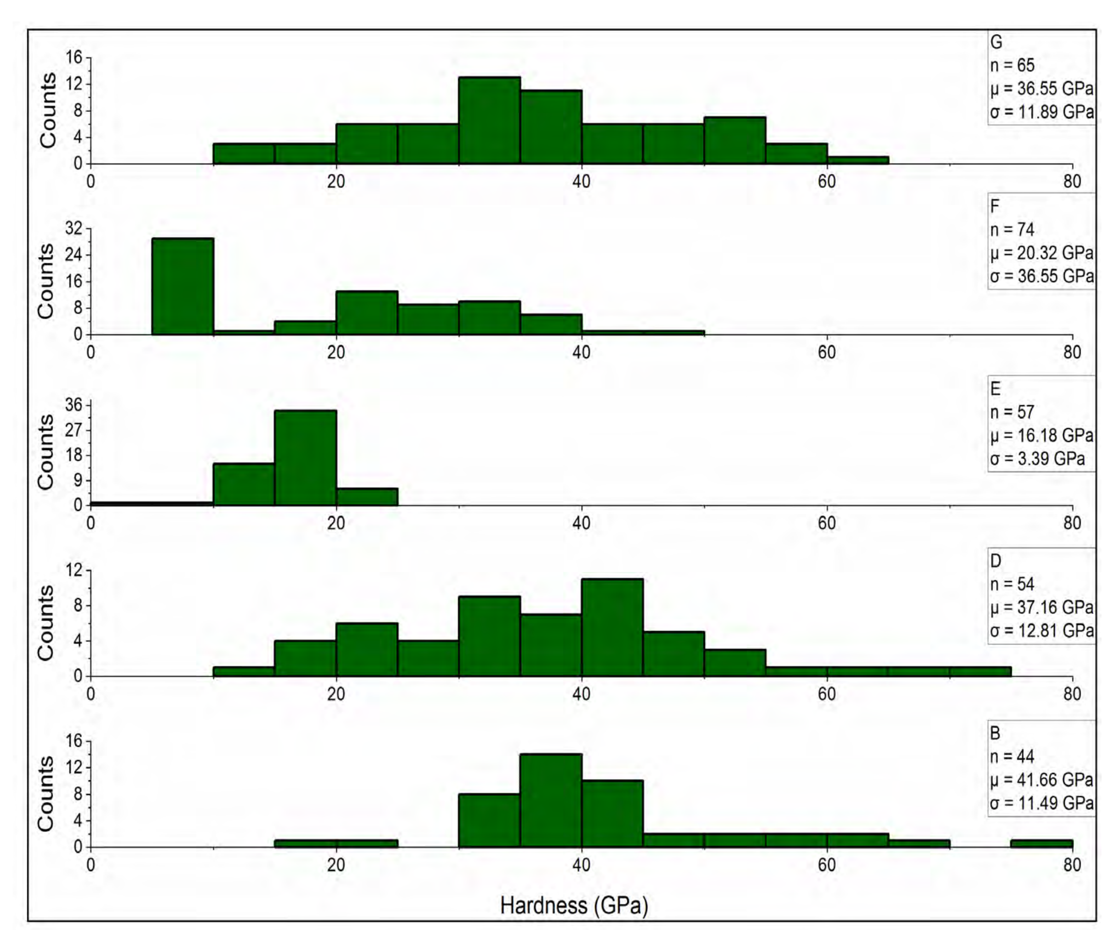
Nanoindentation shows hardness values with a mean of around 40 GPa for samples B and C, a significant improvement to literature values of approx. 33 GPa for tantalum boride coatings deposited via CVD. The large standard deviation confirms the inhomogeneity shown in XPS.

Bias Set (D, E, F, G)

XRD



Nanoindentation



The XRD pattern (left) shows an increase in Ta signal until sample G (-350V), where the TaB_2 signal returns. These changes in the XRD pattern are mirrored by the nanoindentation values (right) – as the Ta signal increases, the mean hardness decreases. For sample F, the lowest values were found on the edge of the sample, where the bias is known to be most active.

Conclusions

- MPCVD is a viable method for producing hard and superhard tantalum borides, in a way that has not been reported to date.
- Higher substrate temperatures are more favorable for TaB_2 production.
- The discrepancy between XPS and XRD indicates that a solid solution of boron in tantalum close to the surface, while the borides are deeper.
  - These both serve to harden the material, allowing for the superhard values measured by nanoindentation.
- The presence of DC bias is not effective at increasing hardness.
  - This may be due to increased ion bombardment that is offset in sample G by a measured increase in boron (not shown).
- Future work will go towards improving film homogeneity and finding a better implementation method for the DC bias.

Acknowledgements

Support provided by the National Aeronautics and Space Administration (NASA)-Alabama Space Grant Consortium, Research Experiences for Undergraduates (REU) award to University of Alabama – Birmingham (UAB). This material is also based upon work supported by the National Science Foundation (NSF) EPSCoR RII-Track-1 Cooperative Agreement OIA-1655280. Any opinions, findings, and conclusions or recommendations expressed in this material are those of the authors and do not necessarily reflect the views of the National Science Foundation.