

Tungsten Carbide Thin Film: Fabrication by Employing Plasma Focus Device

N. J. Dutta^{1*}, N. Bharali², S. R. Mohanty²

¹Department of Physics, Tihu College, Tihu (Assam) 781371, India

²Center of Plasma Physics-Institute for Plasma Research, Sonapur, (Assam) 782402, India

*Email: nilamjadu889@gmail.com

Article history : first Received March 4; Final May 20

ABSTRACT

Tungsten carbide thin film has advantageous properties like high melting point, high hardness value, high wear resistance capacity, low friction, more chemical stability, etc.

In literature, a wide range of techniques has been reported for the synthesis of tungsten carbide, including direct carburization of tungsten powder, high-energy mechanical milling, spray conversion methods, solid-gas phase reactions, sol-gel processing, in-situ carburization, and combustion synthesis. But in this work, a low-cost, tabletop pulsed plasma accelerator called a plasma focus device is used to fabricate the tungsten carbide thin film. The device plasma focus is well-known as an energetic source of electromagnetic radiations such as ions, electrons, neutrons, etc. As an ion source, the device can be operated with a variety of ion species simply by changing the filling gas environment. For carrying out the present work, we have operated the device in an acetylene gas environment for the implantation of carbon ions on the tungsten substrate. After the imposition to various numbers of carbon ion pulses, the optimum condition for the growth of thin film was investigated with different kinds of characterization tools mainly to ascertain the morphological and structural variations. The FESEM picture infers the development of carbon rich defects on the surface of the tungsten whereas the GIXRD confirms the development of tungsten carbide thin film.

Keywords: Plasma focus, Tungsten carbide, Acetylene, Ion, Thin film.

A part of the work of the MS was presented by the 1st author at the International conference on Electronics and Advances in Science & Technology (IConEAST-24) held at Gauhati University in November 2024.

1. Introduction:

Tungsten offers several significant advantages, making it a peerless material in various industries. One of its most remarkable properties is its exceptional strength and hardness, which make it highly resistant to wear and tear [Erik and Wolf-Dieter 1998]. Tungsten has the highest melting point of any metal, making it ideal for high-temperature applications, such as aerospace, electronics, and manufacturing of tools that operate in extreme environments. Also, tungsten is highly dense, which gives it excellent shielding properties against any radiation. Furthermore, the qualities of tungsten, such as its resistance to corrosive environments and structural stability under extreme conditions, significantly enhance its suitability for advanced applications. Hence, all these properties make tungsten indispensable in the production of various devices and components of industries. On the other hand, tungsten has the high potential to combine with a wide range of elements to produce various alloys and ceramic. It is because of its properties like low sputtering ratio, high chemical resistance, perfect hardness, high thermal conductivity, high melting point and low coefficient of friction, etc as mentioned above [Erik and Wolf-Dieter 1998].

Out of various composites of tungsten, tungsten carbides are utilized in nuclear and general engineering sectors, cutting and drilling industries, aerospace, and marine activities etc. In literature, there are several processes used to prepare tungsten carbide, typically involving the combination of tungsten powder and carbon sources some of those are Direct Carbonization Method [Xiaoxin et al., 2015], Co-precipitation Method [Qi et al., 2017], Hydrothermal Method [Thangavelu et al., 2023 4], etc.

The Direct Carbonization Method involves mixing tungsten powder with a carbon source, such as graphite, and heating the mixture in a vacuum or inert atmosphere to temperatures between 1400°C and 2000°C. During the reaction, carbon combines with tungsten, leading to the synthesis of tungsten carbide (WC).[Erik and Wolf-Dieter 1998].

In Co-precipitation Method tungsten oxide (WO_3) is first dissolved in a suitable solvent, such as ammonium hydroxide or sodium hydroxide, to form a precursor. Carbon is then introduced, often in the form of carbon black or graphite. The solution is subjected to precipitation and then heated in a furnace under a controlled atmosphere to produce tungsten carbide [Qi et al., 2017 3]. Whereas Hydrothermal Method involves using a high-pressure vessel (autoclave) to treat tungsten oxide or tungsten salts with carbon-containing agents (like glucose) in a solvent at elevated temperatures and

pressures [Thangavelu et al., 2023]. Tungsten carbide (WC) can also be synthesized using advanced deposition techniques such as Chemical Vapor Deposition (CVD) [McAuliffe and Low, 1974] and Physical Vapor Deposition (PVD) [Saito and Matsumoto, 2001, 6].

In the chemical vapor deposition (CVD) technique, tungsten carbide films are produced through the interaction of gaseous precursors, typically involving a tungsten-bearing compound such as tungsten hexafluoride (WF_6) or tungsten carbonyl ($W(CO)_6$), with a suitable carbon source (such as methane, CH_4 , or acetylene, C_2H_2) under controlled temperature and pressure conditions [McAuliffe and Low, 1974 5]. PVD involves the evaporation of a tungsten material (usually tungsten metal or tungsten oxide) in a vacuum chamber, followed by the reaction of the vaporized tungsten with a carbon source. The tungsten vapour reacts with the carbon to form tungsten carbide, which is then deposited as a thin film on the substrate [Saito and Matsumoto 2001]. Here tungsten metal or tungsten oxide is evaporated or sputtered in a vacuum chamber and a carbon source, such as methane or acetylene, is introduced into the chamber.

Out of all the above mentioned methods, PVD method has the advantages such as for precise control over the thickness and composition of the tungsten carbide films and also suitable for producing thin coatings on complex shapes or components that cannot be coated using other contemporary methods. Keeping the advantages of PVD process, here in the present work, we have used a tabletop, low cost device called Plasma focus device for preparing the tungsten carbide thin film.

2. Experiment

As mentioned earlier, a plasma focus device is used to accomplish the current work. The device basically is a coaxial plasma accelerator that produces high temperature, high-density and short lived plasma [Rawat 2013]. Figure 1 depicts a schematic representation of the experimental setup employed for the fabrication of the tungsten carbide film.

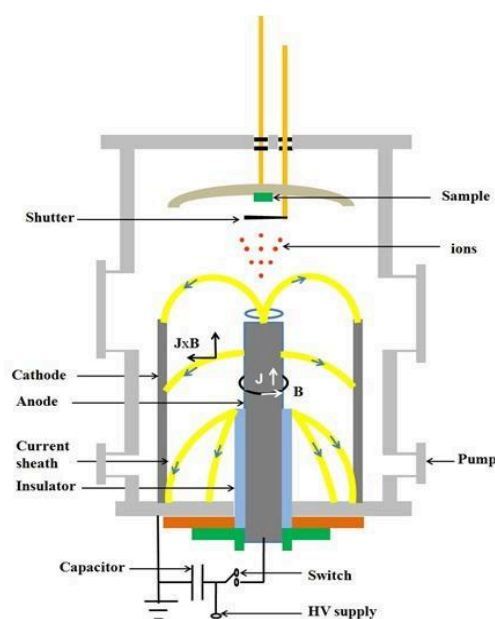


Figure. 1: Schematic diagram of experimental set up.

The device mainly consists of a hollow chamber that houses an electrode assembly, a pump, a capacitor, and other components. The electrode assembly includes a central anode surrounded by twelve cathodes arranged in a squirrel-cage structure, and the both electrodes are separated by an insulating sleeve [Bhuyan et al., 2004]. Initially, the chamber is evacuated using a rotary pump, after which acetylene (C_2H_2) gas is introduced to achieve a pressure of 0.5 Torr. The capacitor is then charged to its maximum value and discharged between the electrodes. This discharge causes the gas medium in the lower part of the chamber to ionize, forming a current sheath. The sheath is propelled upward due to the self-generated Lorentz force, eventually collapsing at the anode's top, creating a short-lived plasma pinch characterized by extremely high densities and temperatures. This plasma column disintegrates within nanoseconds (in a pulse) due to $m=0$ instabilities, leading to emission of carbon ions which is directed upward from the electrode. A polished tungsten sample of dimension 1 cm x 1 cm and thickness of 0.3 cm is introduced into the path of carbon ions at a distance of 06 cm from the anode top. The measured ion energy of the device for the present work is in the range of 25 to 750 keV as measured by biased ion collectors [Bhuyan et al., 2011 9]. The tungsten samples are irradiated with different numbers of ion pulses by simply varying the number of Plasma focus (PF) shots. After irradiation the both reference and irradiated samples were examined with the help of various characterisation tools such as Optical Microscope

(OM), Field Effect Scanning Electron Microscope (FESEM), X- Ray Diffraction (XRD). The detailed surface morphology was analyzed using FESEM, while structural variations were examined through Grazing Incidence X-ray Diffraction (GIXRD). Here, in this work we have used a BX Olympus OM, a Sigma Zeiss FESEM and Bruker K alpha XRD at 3° glancing incidence angle.

3. Results and Discussion:

After irradiation to different PF shots, the irradiated samples are initially visualized through naked eye and clear differences have been observed the shining surface of the reference samples becomes blue in colour. Immediately after primary inspection those are examined under OM. In Figure. 2, an optical micrograph of 30 PF shots irradiated sample is shown.

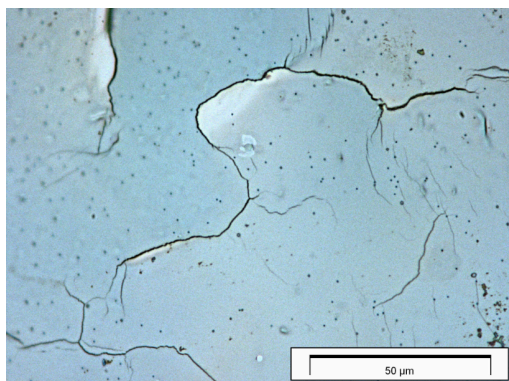


Figure. 2: Optical micrograph of 30 PF shots irradiated sample.

At lower PF shots (such as 10 to 20 PF), the irradiated samples shows the formation of fine grain structures, surface roughness, or erosion patterns whereas at higher PF shots (greater than 30 PF shots) the sample surface depicts the formation of cracks, fractures, along with the other forms of surface damages that are persistence in case of lower PF shots irradiated samples. To get a better insight the irradiated samples are viewed under FESEM. Figure 3 represents the FESEM picture of PF shots irradiated samples.

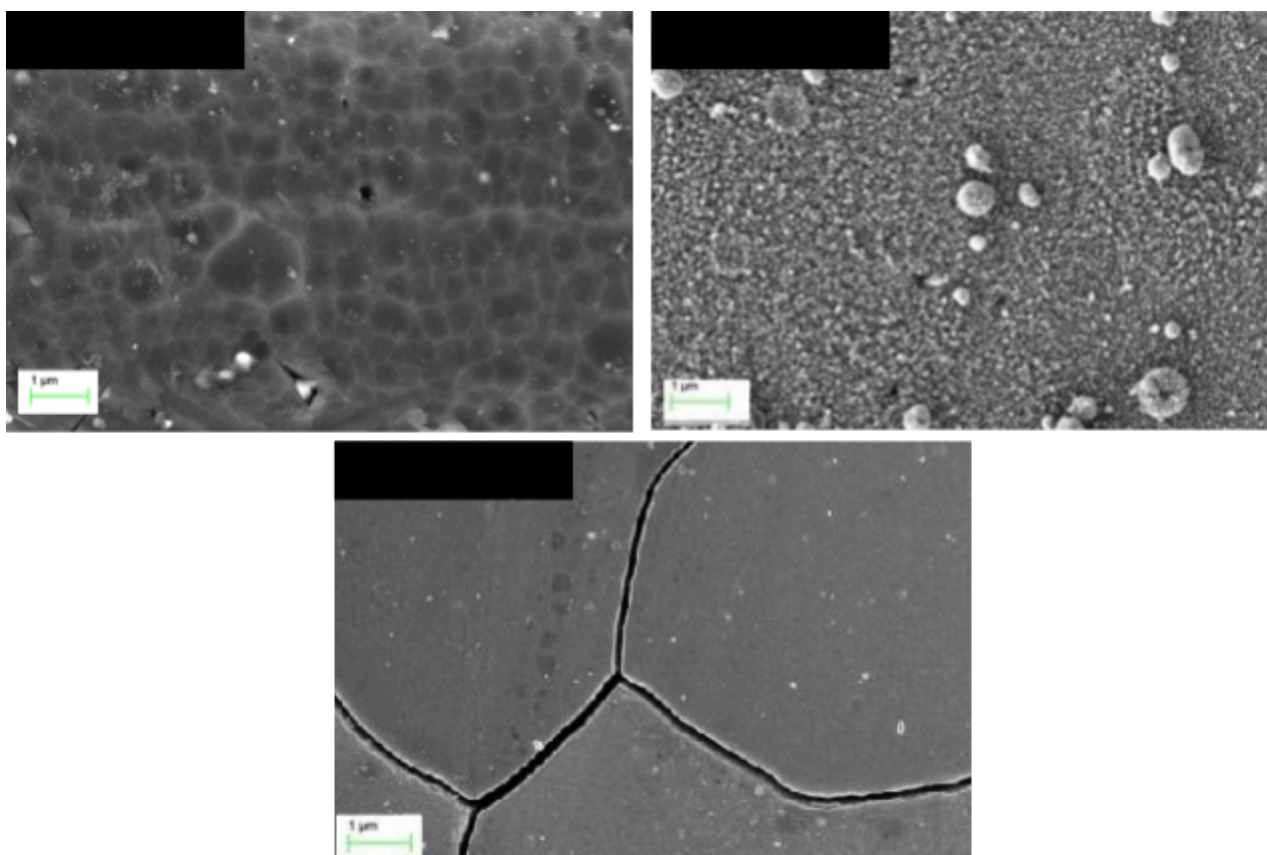


Figure. 3 FESEM picture of irradiated sample to various PF shots.

The FESEM result also corroborates the finding of Optical micrographs. It also confirms that the tungsten particles have completely penetrated into the carbon-rich defects and are visible on the substrate surface. The carbon-rich defect appears to be a highly stable viscous liquid crystal and it is believed that the liquid form of the defect is due to the hydrocarbon structure and it contains high concentration of carbon and hydrogen along with low content of oxygen. Moreover, the GIXRD spectra of reference and 30 PF shots of irradiated samples are shown in Figure. 4. The irradiated spectrum clearly shows the appearance of tungsten carbide peak along with tungsten. This signifies that on the surface of the irradiated sample tungsten carbide thin films are developed.

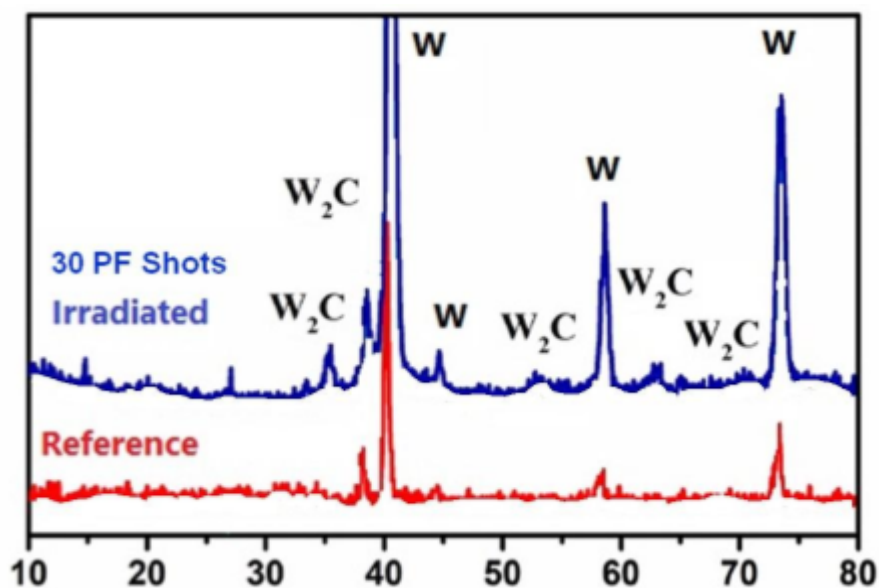


Figure. 4: GIXRD spectra of reference and 30 PF shots irradiated sample

4, Conclusion:

This paper presents an investigation into the deposition of tungsten thin films in an acetylene environment utilizing a low-energy plasma focus device. The carbon ions emitted from Plasma focus devices are used to irradiate the tungsten substrate at different experimental conditions by varying the number of carbon ion pulses (10, 20, 30 PF shots). A clear difference has been observed in case of surface morphology as well as in structural analysis. The results of FESEM characterisation infer the presence of carbon-rich defects in case of irradiated samples. On the other hand the GIXRD spectra confirm the presence of tungsten carbide (W_2C) peaks at the irradiated samples.

Reference:

Erik Lassner and Wolf-Dieter Schubert, (1998) *Tungsten Properties, Chemistry, Technology of the Element, Alloys, and Chemical Compounds*, Plenum Publishers, New York, ISBN 0-306-45053-4

Xiaoxin Ye , Tao Liu , Yongda Ye , Haibo Wang , Guoyi Tang , Guolin Song (2015), **RETRACTED** : Enhanced grain refinement and microhardness of Ti–Al–V alloy by electropulsing ultrasonic shock, *Journal of Alloys and Compounds* 621 , 66-70

Qi Wang , Haihua Wang , Yajie Zhang , Guodong Wen , Hongyang Liu , Dangsheng Su (2017) , Syntheses and catalytic applications of the high-N-content, the cup-stacking and the macroscopic nitrogen doped carbon nanotubes, *Journal of Materials Science & Technology* 33, 843-849

Thangavelu Kokulnathan ^a, Tzyy- Jiann Wang ^a, Faheem Ahmed , Thamraa Alshahrani (2023) Hydrothermal synthesis of ZnCr-LDH/Tungsten carbide composite: A disposable electrochemical strip for mesalazine analysis, *Chemical Engineering Journal*, 451, 138884

McAuliffe C. A. and S. Low, (1974), Chemical Vapor Deposition of Tungsten Carbide Coatings, *Journal of Vacuum Science & Technology* 11, 467–473

Saito, M. and K. Matsumoto, (2001), Physical Vapor Deposition of Tungsten Carbide Coatings for Tooling Applications, *Journal of Vacuum Science & Technology A*, 19, 2154–2158

Rawat R.S, (2013) *IEEE Trans. Plasma Sci.* 41 , 701–713

Bhuyan, H. , S.R. Mohanty, N.K. Neog, S. Bujarbarua, R.K. Rout, (2004) *J. Appl. Phys.* 95 ,2975–2981

Bhuyan, M. , N.K. Neog, S.R. Mohanty, C.V.S. Rao, P.M. Raole, (2011), *Phys. Plasmas*, 18 033101.