Advanced Physical Research
Vol.5, No.2, 2023, pp.95-102

EFFECTS OF HELIUM ION IRRADIATION ON TUNGSTEN-BASED COMPOSITES: NEUTRON DIFFRACTION; AS A REAL-TIME DIFFRACTOMETER

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Abstract. The weight ratio 6% was formed for this paper by B4C-2% (99.5% purity, 20 µm particle size), TiC-1% (99.5% purity, 15 µm particle size), C (99.5% purity, 21 µm particle size) and the remaining part is tungsten W (99.5% purity, 17 µm particle size) structure had been established. In order to get homogeneous sinter, it was subjected to a temperature gradient of up to 1750 °C for 2 hours with a temperature step of 5 °C per minute. In the study, neutron diffraction (ND) was performed using the time-of-flight technique (TOF) in the IBR-2M research reactor. The obtained data were refined with the FULLPROF Rietveld suite. The results convey that it is different from the results obtained by X-Ray diffraction.

Keywords: *Tungsten composites, helium irradiation, neutron diffraction, lattice parameters.*

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Received: *26 January 2023;* ***Accepted:*** *28 April 2023;* ***Published:*** *23 June 2023.*

1. Introduction

There is a constant demand for cost-effective materials with properties such as hardness and super-hardness in order to utilize use in various industrial applications (Darziyeva *et al.*, 202; Utamuradova *et al.*, 2023; Orujlu, 2020; Vera-Serna *et al.*, 2022). Transition - metal compounds - carbide, nitride, especially borides show mechanical, optical, electronic, etc. with higher level properties, these ceramic materials continue to serve as a potential source for new research (Hashimov *et al.*, 2019;

How to cite (APA): Neov, D., Beskrovnyi, A.I., Abiyev, A.S., Mirzayeva, D.M., Demir, E., Valizade, A.H., … & Mamedov, F. (2023). Effects of helium ion irradiation on tungsten-based composites: Neutron diffraction; as a real-time diffractometer. *Advanced Physical Research, 5*(2), 95-102.

Mirzayev *et al.*, 2020*;* Popov *et al., 2020;* Bykova *et al*., 2022). Tungsten-boride structures are distinguished among ceramic compounds due to their easy synthesis and high mechanical peculiarities (Gong *et al.,* 2019; Demir *et al.,* 2021). However, despite being investigated for more than half a century, new structures and more accurate investigations in recent years illustrate that there are still gaps in their fundamental understanding and rational features, which preserves their scientific appeal (Belichko *et al*., 2022; Mirzayev *et al*., 2022; Aliyev *et al*., 2019). Therefore, multiform synthesis and experimental methods were used to reveal the structure and features of structures with complex multi-atom composition (Agayev *et al*., 2022). In the given literature, the synthesis of WB_4 crystals with a hexagonal structure and the changes in their mechanical properties when subjected to pressure up to 54 GPa by the X-ray diffraction method - investigation of parameters such as single crystal compressibility, hardness, Yung's modulus - have been reflected (Bykova *et al*., 2022). It was found that the hexagonal polycrystalline WB_4 tungsten tetraborides replaced tungsten atoms along the c axis with B-B covalent bonds, which increased its anisotropy property. In the obtained single crystal, tungsten atoms fully occupy their positions and exhibit hardness under pressure conditions of about 36 GPa. Determining the systematically optimal structure of tungsten boride compounds, as well as thermodynamically stable and metastable structures with a majority of boron atoms, shows itself as a priority. In the experiments conducted on this basis (Li *et al*., 2013), it was determined that tungsten borides with *P63=mmc-2u* WB_2, *R-3m-6u* WB_3 and *P63=mmc* WB_4 structures are thermodynamically stable. Obtaining and researching enriched and complex structures of tungsten-based transition-metal compounds is still relevant. In this regard, the obtained results suggest that $WB_{4.2}$ is the most energetically best case with a minimal energy of 3 MeV per atom among the formations with different structures obtained under temperature and pressure (Kvashnin *et al*., 2020).

As a result of obtaining and characterizing solid materials included in the tungsten boride class, all space groups and lattice parameters of certain structures are known in detail. The fact that the structures it creates are even comparable to diamond (953 GPa along the *hP6-WB₂* c-axis) makes us believe that it can really eliminate some of the challenges facing physics (Chrzanowska *et al*, 2016). Properties such as Vickers hardness, elastic anisotropies, and ideal stiffness are some of the properties produced by the strong 3-way covalent bonds formed by the B-B and W-B covalent bonds that form the basis of the structure. Moreover, the density of electrons located at the Fermi level resulted in metallic properties in these structures. After the synthesis, W_2B (gamma phase) with a tetrogonal structure, *I4/mcm* space group, and WB, α and β space groups appear as *$I4_1$ /amd* and *Cmcm*, respectively, in which case their volumes are V_γ = 148.05 $Å^3$, V_α= 167.16 $Å^3$ and V_β= 83.43 $Å^3$ manifested in the form **(**Chrzanowska *et al*, 2016). W_2B with tetragonal phase of *I4/m* symmetry is considered to be the most mechanically and dynamically stable tungsten boride class (Qin *et al.,* 2018). Furthermore, based on the band structure it has, it can be said that this compound is a metal. Because of this conductivity property and mechanical properties, W_2B promises to be a key material for today's hard coatings and electrical measurements.

One of the main problems of physics is related to increasing the efficiency in obtaining of ecologically clean energy. It is known that thermonuclear reactors have been the most important issue in the steps to be taken for this purpose for years. Diverter stages are considered one of the main stages during construction (Aldabergenova *et al.,* 2016), and the selection of raw materials for their construction is the most important priority. Here, when the front walls are created, the maintenance of

plasma columns based on superconducting magnets is one of the primary factors. If we look for a material with this enthusiasm, it is natural that tungsten will take place in the first ranks in terms of compatibility (Petaccia *et al.,* 2019). Tungsten is the best example as a substance resistant to all physical and chemical effects, such as corrosion, high temperature and neutron radiation, in order to create longevity in the front walls. Studies have shown that (Aldabergenova *et al.,* 2016) tungsten exhibits qualitatively the same properties despite being irradiated under different conditions than alpha rays. After the irradiation, the bubbles created by the alpha particles in the regions inhibited by the particles cause a sharp decrease in the microhardness of the material.

Recently, there is much renewed interest on tungsten borides. Due to their comparatively elevated cost, they seek prospective use a radiation shielding and constructive materials in high-end applications like contemporary and future fusion reactors (Windsor, 2021). Tungsten itself is a metal with notable high melting point and density but also high brittleness. Tungsten borides poses even higher hardness (micro hardness in the range 20-30GPa (Okada *et al.,* 1995) but together with the metal binder they possess increased fracture toughness. Moreover, element (B) adds to neutron shielding capacity. Tungsten borides possess high density, high wear resistance and more importantly, high sputtering resistance at high temperatures and irradiation conditions. Their synthesis however remains as a challenge since it requires high temperature solid state sintering from precursor materials. In recent years, spark plasma sintering or electric current assisted sintering has been applied for alleviate requirements on temperature and pressure needed. Applying electric current (pulsating or not) is believed to improve homogeneity of the sintered material, both as chemical composition and particle size.

Neutron diffraction is valuable tool for studying the processes gone during high (temperature, pressure) synthesis and alloying, including tungsten alloys. It allows looking in the bulk of the material, where conditions (temperature, pressure) may differ from those on the surface. Applicability on borides remains somehow a challenge, because of the very high absorption of thermal neurons by element B. Boron possess high capture cross section and is known constituent of neutron shields. Therefore, most of the research completed up to today is either by Monte Carlo simulation (Windsor, 2021) or XRD experiment (Okada *et al.,* 1995).

2. Materials and methods

Specimens were prepared by from precursor materials in weight percentage 6% B4C–2% TiC–1% C and the remainder – tungsten in the form W (99.5% purity, 17 μm particle size), B_4C (99.5% purity, 20 μm particle size), TiC (99.5% purity, 15 μm particle size), and C (99.5% purity, 21 μm particle size). In this case high-temperature mechanical alloying was employed for sintering achieve homogeneous sintering, the samples are subjected to a temperature gradient for 2 hours to 1750 °C, at a temperature step of 5 °C per minute. Procedure is described in (Demir *et al*., 2020). Following such a procedure, we have expected that the material is formed by tungsten (W) metal matrix with nondispersive distributed tungsten boride particles in the volume. From similar research we conclude this should include W_2B, but other boride phases are also possible (α-WB, β-WB, WB_2, W_2B_5, WB_4) as well as tungsten carbide (WC). Conceivably, existence phase content of these borides should depend strongly on the conditions and at which synthesis is a performed. Neutron diffraction (ND) measurements were performed on RTD (real time diffractometer) (Balagurov *et al.,* 2016) installed on IBR-

2M research reactor using Time-of-flight (TOF) technique. The flight path of the neutrons was about 24m and the time FWHM (full width at the half maximum) of the neutron pulse after the moderator was about 320 μs. The data was fit using FULLPROF Rietveld suite (Rodriguez-Carvajal *et al., 1990*). High boron content in the specimens cuases high attenuation of neutron intensity and hence measurement times of up 10h per specimen. Some of the as synthesized specimens was irradiated with with 2.5 MeV helium ions (fluence 5.0×10^{20} ion/cm^2) and measusred with the same procedure (Azimova *et al.*, 2020; Mirzayev *et al.*, 2021).

3. Results and discussion

The experimentally obtained TOF neutron spectrum and the Rietveld fit shown in Figure 1.

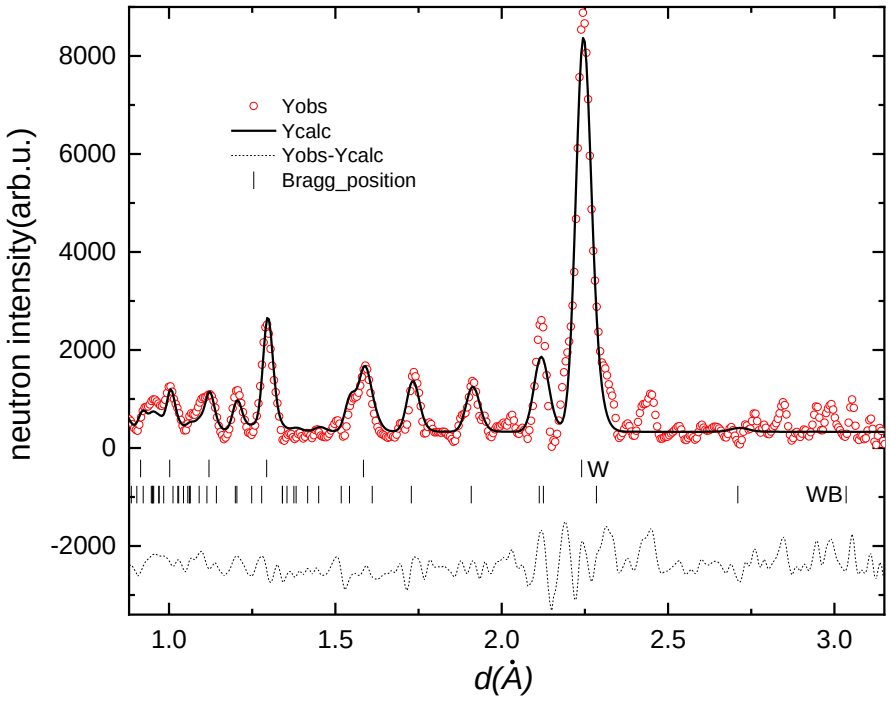

Fig. 1. Measured TOF neutron spectrum of non-irradiated specimen (as synthesized), Rietveld fit, peak positions of two crystal phases – metallic W and δ-WB as well as difference plot)

Two main phases contribute to it – unreacted metallic tungsten (W) and tungsten boride (δ-WB) (Petaccia *et al.*, 2019; Qin *et al.*, 2018). Of the tungsten phase with high symmetry (SG I m$\bar{3}$m) several peaks exist and, two main peaks dominate, namely (110) at 2.24 Å and (211) at 1.29Å. In the BCC crystal lattice where tungsten atoms occupy (0, 0, 0) positions. δ-WB phase has tetragonal space group I41/amd where tungsten atom occupies position (0.1/4.0/178) and boron (B) atom occupies (Mirzayev *et al.*, 2019) (0.1/4.0/031) position. Main results of the fit are listed in Table 1.

The quality of measured spectrum is notably worse at high-d values of the spectrum, because of the scarcity of diffracting neutrons (spectrum of neutrons from the moderator roughly has Maxwell distribution). Spectrum was normalized by dividing by incoherent scattering spectrum of vanadium, time channel per time channel. There is a peak at 2.2Å overlapping with W(110) which remains unaccounted for. We tried to incorporate several other possible phases into the Rietveld fit (Fig. 2).

Table 1. Main result obtained by Rietveld analysis

Phase	SG	Cell parameters	Phase fracture, weight %	Chemical units per unit sell
W	I m$\bar{3}$m	a = b = c = 3.168 Å V = 31.8 Å^3	78%	2 W
δ-WB	I4$_1$/amd	a = b = 3.084 Å c = 17.002 Å V = 161.794 Å^3	22%	8 WB

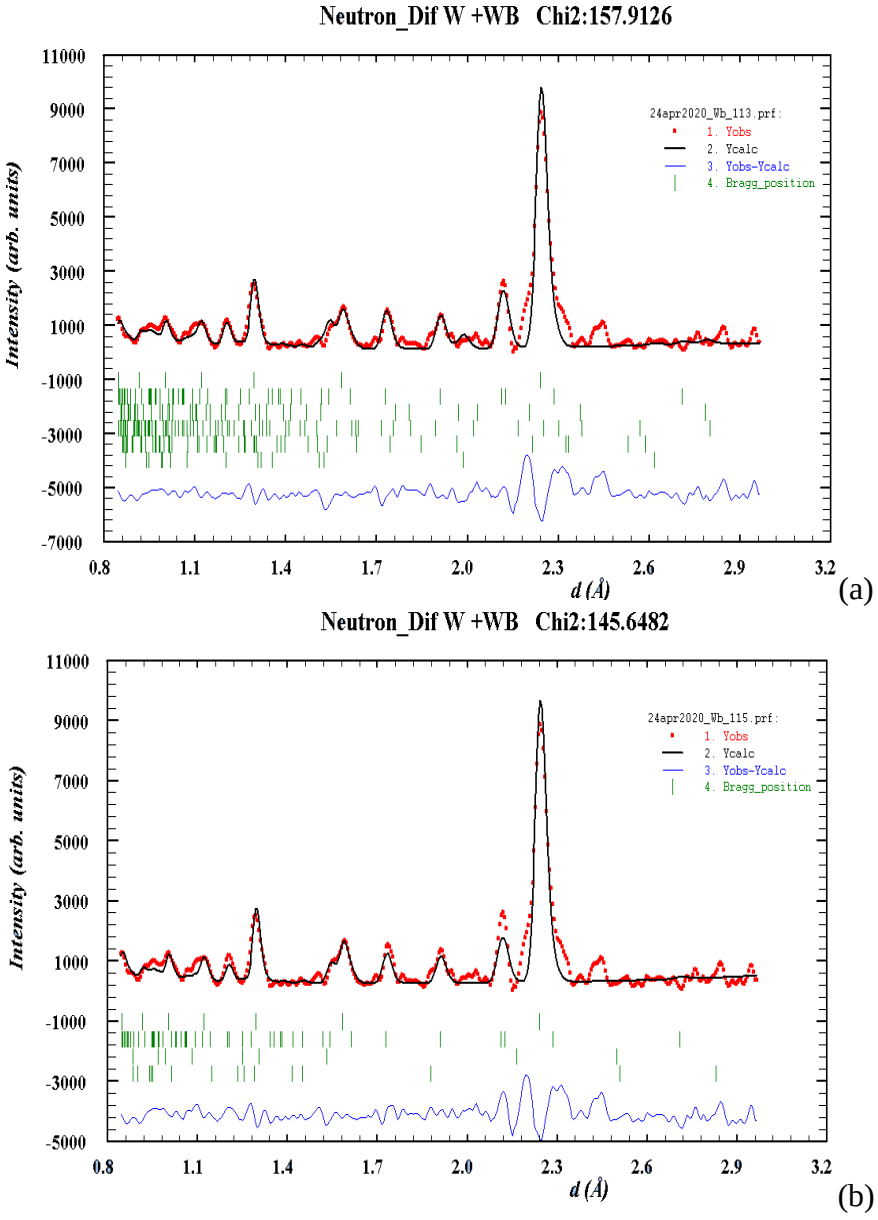

Fig. 2. Rietveld refinement attempt of the same spectrum with and addition of W_2B, B_4C, W_2B_5, WB_2 (a) and TiC and WC (b) phases with designated peak positions

Notably, there is indications that small fraction of W_2B exists, much lower fraction than δ-WB, but this does not contribute to significant improvement and lowering χ^2. Other possible phases as B_4C, W_2B_5, WB_2, TiC and WC were ruled out. We alsi tried to obtain

measurable effect of the influence of irradiation with high intensity He ions on the structure of tungsten –tungsten borides (Abdullayeva *et al.*, 2020). Some specimen was irradiated at 5.0×10^{20} ion/cm^2 fluence of $^4He^+$ ions of 2.5 MeV energy and measured by TOF diffraction (Mirzayev *et al.*, 2020). Relying on high irradiation fluence combined with relatively low ion energy however is expected that high degree of radiation damage will occur, but at very thin layer close to the surface (Tashmetov *et al.*, 2019; Thabethe *et al.*, 2022) According to the rough estimations, irradiation induced damage region in heavy tungsten compounds causes by light He ions has characteristic depths of order of few μm. Such shallow depth is not expected to contribute to any significant changes in the diffraction profile. Similar study conducted by us by X-ray diffraction but on specimen sinthesized at higher temperatures (T = 2550^{o}C) (Neov *et al.*, 2022) shows different composition of the surface layer, namely existence of predominantly W_2B phase and small addition of δ-WB. Even at the surface layer exposed to high radiation damage, XRD detects only diminutive changes namely phase content and crystal imperfection (Mirzayev *et al.*, 2023).

4. Conclusions

The discovered deviation of the results obtained by neutron diffraction from those obtained previously by X-ray diffraction implying that the conditions differ significantly on the surface and the bulk of the specimen. This is however admissible since the process of synthesis under high temperatures and pressure and applied current delivers different physical conditions on the surface where the contact with the applied pressure head is applied. In addition, the process is self-running in sense that homogenization on the surface leads to change of the conductivity and further complete synthesis. The voids and the porosity in the volume remain higher. This also proves usefulness of the neutron diffraction as an indispensable tool for investigating the processes in the bulk of the material. This however is crucial because physical properties of the material are determined by the structure of the bulk material with the exception of the sputtering resistance determined by the surface structure. Altogether, this imposes rigorous requirements on the conditions on which synthesis is done. Most probably, the combination of temperature/duration in current study is not sufficient for complete synthesis of tungsten boride.

Acknowledgments

This work was supported by the Azerbaijan Science Foundation-Grant № AEF-MCG-2022-1(42)-12/03/1-M-03.

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