SUPPLEMENT

Evolution of the microstructure and phase composition of the products formed in the reaction between iridium and W_2B

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1. Experimental

The XRD measurements were calibrated using Al_2O_3 as a standard (NIST 1976a). Quantitative phase analysis and lattice parameter refinement were performed using the Rietveld method (Topas 4.2 software, Bruker AXS, Germany). The phases analyzed were cross-referenced against the Inorganic Crystal Structure Database ICSD (FIZ Karlsruhe, Germany, 1996). The crystal structure data for WB, WIr, WIr_3 and W_xIr_{1-x} solid solution were referenced after Springer Materials database.

The neutron flux was $5 \cdot 10^6$ $^{nu}/_{cm^2 \cdot s}$, and the neutron flight path length was 24.45 m [1]. Because of the large cross sections of absorption for boron and iridium (σ_{abs}(B) = 767 barn, σ_{abs}(Ir) = 425 barn), the spectrum was accumulated for 12 h. Spectra were measured at θ = 85.8° (neutron backscattering). The resolution was Δd/d ≈ 1%; the range of neutron wavelengths was ~1−12 Å with the maximum intensity at ~1.9 Å. The TOF-ND patterns were fitted using the GSAS-II software[2].

The morphology and elemental composition were studied using a Hitachi TM 1000 scanning electron microscope coupled with a SwiftED^{TM} energy dispersive X-ray spectroscopy (EDS) detector (Oxford Instruments Analytical Ltd., UK, accelerating voltage of 15 kV).

Additionally, the SEM/EDS studies were performed with a Mira 3 LMU high-resolution scanning electron microscope (TESCAN) equipped with an INCA Energy 450 XMax 80 EDS detector with accelerating voltage of 20 kV. Cross-sections of the samples were prepared for in-depth analysis of the microstructure and local elemental composition of the powdered products. The samples were embedded into epoxy resin, cut with a diamond saw, and carefully polished using a set of polycrystalline diamond suspensions with particle size ranging from 9 to 1 μm (Monosyn Duo, Synercon, Germany). The probe current and the energy shift of the recorded spectrum were calibrated using metallic cobalt as a standard. A SU8220 scanning electron microscope (Hitachi, Japan) equipped with QUAD and Quantax 60 EDS detectors (Bruker, USA) was used for detecting boron (K-line) together with tungsten (M-series) and iridium (M-series) at an accelerating voltage of 6 kV (shared-use facilities of the "Nanostructures" Center, Institute of Semiconductor Physics, SB RAS, Novosibirsk) [3,4]. Low accelerating voltage provides a sufficient peak to ground ratio for quantification of boron [3]. In accordance with Bruker recommendation, Ni_3B was used as a calibration standard. SEM images were processed using the ImageJ software (National Institute of Health) to evaluate the sizes of product particles and aggregates.

2. RESULTS

2.1. The 1 : 1 mixtures

Scanning electron microscopy

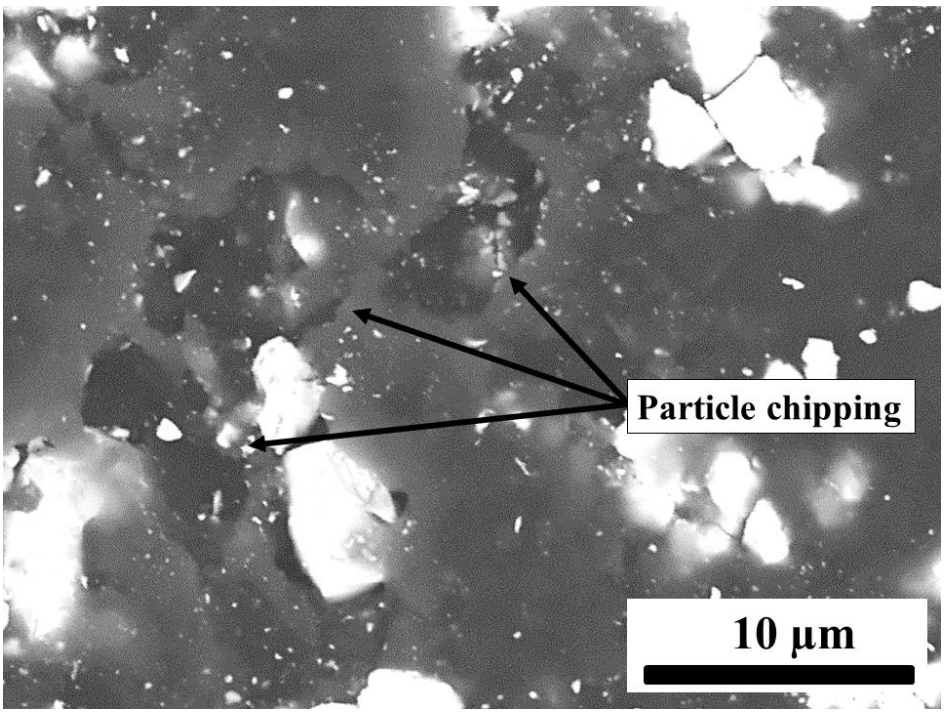

Figure S1. SEM image of the sample heat-treated at 1300°C after preparation of cross-section

2.2. The 3 : 1 mixtures

Phase composition

X-Ray diffraction analysis

Table S1. The unidentified *d* spacing and the corresponding 2Θ

2Θ	23.11	26.67	30.64	31.77	35.78	44.47	49.27	50.48	51.18
D, Å	3.84	3.34	2.91	2.81	2.50	2.03	1.84	1.80	1.78

The TOF-ND analysis

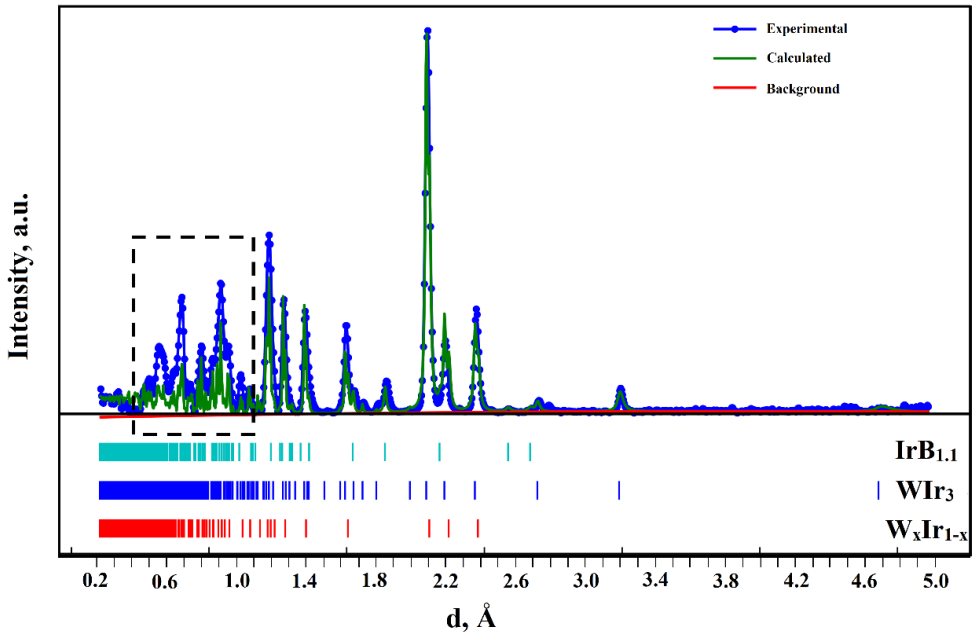

Figure S2. The TOF-ND of the products obtained in the 3 : 1 mixture at 1600°C. The *d* range in which the unidentified peaks were detected is denoted by dashed line.

Elemental composition

The BSE SEM/EDS results, accelerating voltage of 6 kV

Spectrum: WIr 8

Element	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (3 Sigma) [wt.%]
Boron	K-series	0.00	0.00	0.00	0.00
Tungsten	M-series	25.23	30.41	31.36	3.36
Iridium	M-series	57.74	69.59	68.64	7.39
	Total:	82.96	100.00	100.00	

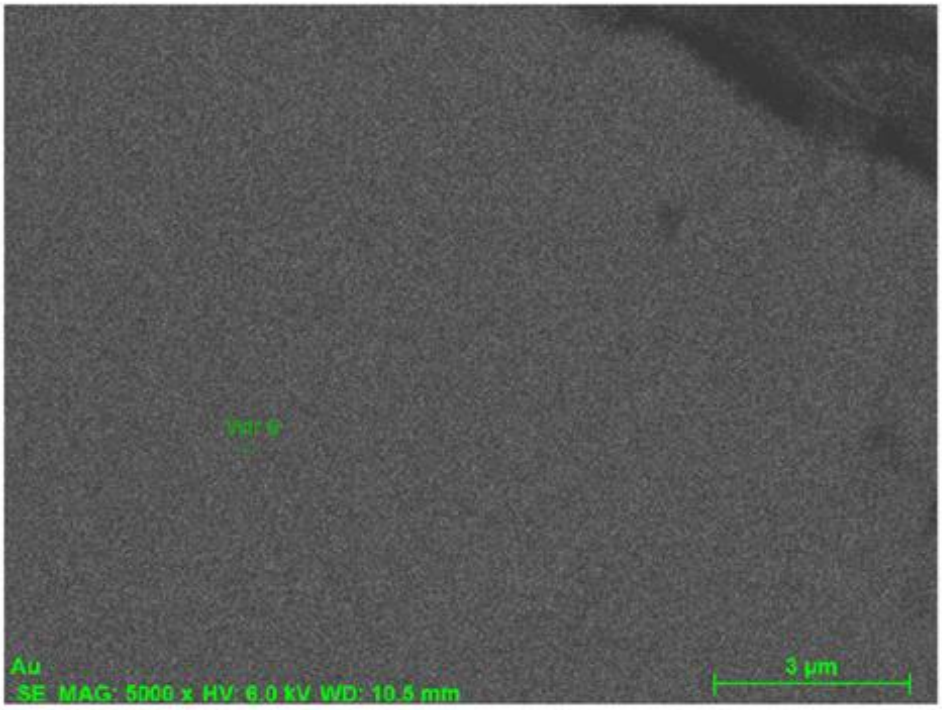

Figure S3. The BSE SEM/EDS image and elemental composition of the area belonging to the W_xIr_{1-x} intermetallic phase (where $x \approx 0.33$)

Spectrum: WIr 1

Element	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (3 Sigma) [wt.%]
Boron	K-series	0.00	0.00	0.00	0.00
Tungsten	M-series	22.96	31.02	31.98	3.07
Iridium	M-series	51.05	68.98	68.02	6.56
	Total:	74.01	100.00	100.00	

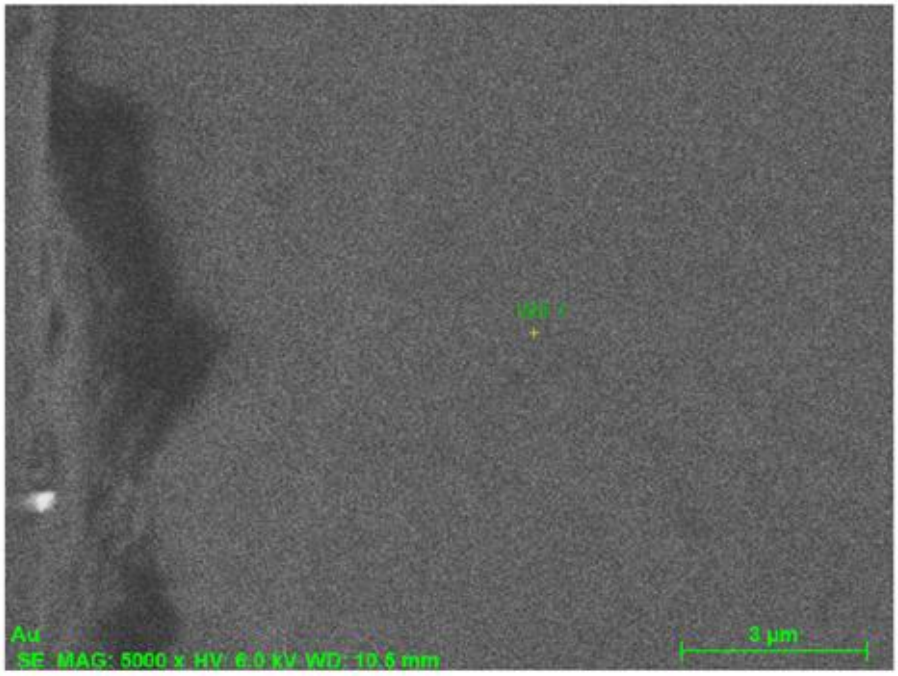

Figure S4. The BSE SEM/EDS image and elemental composition of the area belonging to the W_xIr_{1-x} intermetallic phase (where $x \approx 0.33$)

References:

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