Notes on nomenclature in “HRXRD study of the Theoretical Densities of Novel Reactive Sintered Boride Candidate Neutron Shielding Materials”

This README concerns the Excel documents “**JMM\_Novel\_BorideArchive.xlsx”**, “**cWC-RSB-XRD\_raw data.xlsx**” and “**JNM\_Novel\_RSB.xlsx**”

**Part 1: Materials data (JMM\_Novel\_BorideArchive.xlsx)**

This spreadsheet records the recipes and the recipe generator for cWC and RSB material development over a period of 9 months from June 2016 until March 2017.

This is a partial data set as it does not record the properties of cWC standards and the properties of the wear test sample geometries.

**Nomenclature**

Overview:

RSB – Reactive Sintered Boride – refers to compositions which can be sintered in that mass loss, shrinkage and porosity are similar to that of cWCs but which do not contain any WC at weigh-in.

cWC-RSB – any material with boron additions and WC powder.

**RSB Nomenclature**

Sample names are determined by their composition determined at weigh-in. RSBs and cWc-RSBs are named by the following convention. Some exceptions occur which are explained in detail. in Sheet 2 (1450 DMK Vacuum).

**BxTEy(W)**

**B** Identifies the material as having some (any) B4C additions at weigh-in. Boron additions from all samples within this spreadsheet vary from 0.5wt% - 9wt%.

**X** This represents the wt% of boron additions into the sample

**TE** Dummy initials that separate the boron content from the Fe-8Cr content given by digit **y.** In the case of fractional additions, a numerical value can replace the E in TE, e.g B5T522W.

**Y** Represents the wt% of Fe-8Cr at weigh-in

**W** Identifies the sample as having W metal additions. These can be any quantity of W metal from 4wt% to balance for true RSB samples.

Other identifiers are discussed in the description for Sheet 2(1450 DMK Vacuum).

**Sheet 1: Composition Calculator**

Three calculators are present within this spreadsheet from left to right:

1: Density calculator – calculates the weighted average density of the reagents at weigh in. This is suitable for calculating theoretical density of cWCs but not RSBs due to the chemistry that occurs during sintering. This calculator was used to estimate possible theoretical densities for trial cWC-RSB and RSB compositions.

2: Atomic calculator – calculates the atomic abundance from weigh-in reagents.

3: Carbon additions for cWCs: This is a calculator to determine the carbon additions required for stoichiometric cWCs based on the carbon/oxygen content of WC powder. This calculator can be used to assess nonstoichiometry of cWC materials.

**Sheet 2: 1450K DMK Vacuum – mixed cWC-RSB and RSB compositions**

This sheet is an archive of the first attempts at adding boron to cWCs and the initial round of RSB materials. Raw data incorporating mass-loss, magnetism (where applicable) and basic mechanical data is incorporated within the spreadsheet. All samples on this page were vacuum sintered with an Ar/H bleed for organic binder removal.

**Column A** has a summary of all the sample compositions with their reagent weigh-in densities calculated in **column B.**

**Column C** has the composition and the temperature at sintering (1450°C or 1520°C).

**Column D** has the WC type – this gives the average sintered WC grain diameter if the WC powder was in a typical sintered WC-Co body. For example WC060 refers to WC powder with a target sintered grain size of 6µm, WC008 refers to WC powder with a target sintered grain size of 0.8µm.

**Column E** has the Fe-8Cr content in wt% at weigh-in.

**Column F** has the pressing pressure (MPa) of cutting die and square cuboid geometry samples.

No samples from this page were featured as **Sheet 2** is concerned with the preliminary study which determined whether RSB materials were feasible when sintered as for cWCs. Samples from compositions B5T522W and B9TE25W are present as these represent the first sinterable non-WC containing RSBs.

All samples above the red line (row 120) comprise of mixed cWC-RSB materials. All samples with no W in the name had WC up to balance when Fe-8Cr and B4C additions are taken into account – e.g B1T515 consists of 1.5 wt% B4C, 15wt% Fe-8Cr.

**Non-canon sample compositions**

**B1TEB –** 1 wt% B4C, 10 wt% Fe-8Cr – WC008 WC (0.8 µm) carbide type. This is the same composition as **B1TE10.** Note that the cWC baseline composition **JBTE10** has the same WC type and Fe-8Cr content for **B1TEB/B1TE10** but without the B4C addition.

**B1TEE –** 1 wt% B4C, 10 wt% Fe-8Cr – WC080 WC (8 µm) carbide type.

**BNSJ1 –** 0.8 wt% B4C, 18 wt% Fe-8Cr, 4wt% W metal – WC060 WC (6 µm) carbide type. This composition was an attempt to make pore-free cWC-RSB material

All samples with W in the suffix have W metal additions at least 4 x wt% of the B4C additions. For example, B4TE25W true has a total W metal content of 16 wt%. B4TE25W 6W has W metal equivalent to 24 wt%.

Any composition in brackets - revised composition when investigating weigh-in errors.

Cr suffix – sample with 2.5wt% Cr3C2 to investigate the effect of enhanced Cr additions on sintering – compositions generally sintered poorly – large porosity and friable materials. All samples with Cr suffix have this in addition to the standard W addition based on B4C content.

Due to loss of some of the raw data, not all compositions are accounted for directly but the general composition rules follow the sample nomenclature.

**Sheet 3: 1450K SinterHIPP - Mostly RSB compositions**

This data sheet contains all the samples examined during this study. Sample convention and nomenclature follows that defined for **Sheet 1** and **Sheet 2,** along with the layout established in **Sheet 2.**

cWC-RSB compositions are above the red line. B05TE8 0.15 C is a cWC-RSB composition with 0.15g carbon additions. B1TE8C has carbon additions calculated using the carbon addition calculator in Sheet 1.

Compositions below the red line contain no added WC at weigh-in. All compositions and samples evaluated in this work are highlighted in purple text.

**Sheet 4: Evaluation 1**

This sheet contains a summary of the physical properties of cWC-RSB and RSB compositions featured in **Sheet 2** and **Sheet** **3** alongside early qualitative evaluation of possible phase presence from samples via EDX.

**Part 3: Raw XRD data (**“**cWC-RSB-XRD\_raw data.xlsx**”**)**

This Excel document contains raw XRD data obtained from the powder and solid sample evaluated in this study. For all samples Co radiation was used to mitigate Fe fluorescence.

Each composition has its own page with each page comprising of four columns:

**Column A** 2θ values 20° - 140°

**Column B** Yobs Raw XRD data output from scan

**Column C** Ycalc Output from the best fitting (lowest Rwp/closest atomic abundance) TOPAS model

**Column B** Difference taken as Ycalc - Yobs

Constituent phases used in TOPAS models and their relative abundances derived from the diffracted intensity fraction are shown in the spreadsheet **JNM\_Novel\_RSB.xlsx.** Data is also available for each scan in individual \*.csv files.

**Part 3: Processed XRD data (**“**JNM\_Novel\_RSB.xlsx**”**)**

This spreadsheet contains the composition data measured from (1) direct measurements via ICP and EDX and (2) Phase data obtained from XRD scans. Most of these sheets contain tabulated and graphical data from the models and the phase abundance determined from XRD modelling.

**Sheet 1: phase abundances + PDFs**

This sheet has the complete selection of candidate phases, their PDF numbers and their unit cell parameters alongside compilations of the detected phases from RSB compositions.

**Sheet 2: Composition difference**

This sheet has some test tables exploring composition trends and also the difference in atomic abundance (at%) between the direct (ICP-EDX) abundance measurements and XRD-derived measurements. Data on the fitting values Rwp, Rexp and GoF are also present in this sheet. This also shows the differences in atomic abundance pre- and post-sintering for all compositions.

**Sheet 3: Composition difference**

This sheet has the direct measurements of ICP and EDX data and the calculations of the true direct sintered compositions of RSB and cWC samples. This also includes new data measured during this project (not the densities seen in “JMM\_Novel\_BorideArchive.xlsx”). The real compositions and measured densities used as constraints for the XRD models.

**Sheet 4: JBTE10 powder**

This is the template for processing the phase abundance, XRD-derived and atomic abundance for all other compositions. First stable shows the candidate phases, their empirical formulae, densities and their mol%, alongside the elemental breakdown of the empirical formulae from the PDF files.

The tables serve as a calculator of atomic composition and weighted average density from the phase abundance as calculated from the spread sheet when the phase quantities are entered in mol %, as seen in **Column H.**

This spreadsheet calculator is copied for all powder and solid XRD spectra which include **Sheet 4 (JBTE10 powder) – Sheet 13 (B9TE25W solid)** inclusive.

**Sheet 14: Boride pies (2)**

This sheet has the compilation of all the phases identified from RSBs and arranged in the form of pie charts. These include major (the top 8 phases in terms of diffracted intensity) and minor (the remaining < 10% of diffracted intensity) to evaluate visual representation of phase data.

Data on the 7 main phase groups (defined as FeWB/FeW2B2, Fe2B/FeB, WB/W2B5, WC, Fe7W6/Fe2W, M12C/M23C6, W2C/W2B) is represented in bar chart form directly comparing phase abundance between powder and solid samples.