Micro-mechanical testing: a quantitative method for measuring local mechanical properties in hardmetals

"MicroMech"

Stage 1: Exploratory study

EPMA European Hard Materials Group EHMG
Research Proposal – March 2015

Proposal

The proposed project is an exploratory study to assess the ability of microsample testing to measure mechanical properties of hardmetals at the local scale. This includes the behaviour of individual features such as WC grains, WC-WC interfaces and the binder. For this purpose microsamples of different geometries (beams, pillars) will be machined using FIB and tested with a nanoindenter system to measure force–displacement information. This project is the first stage of what could be an ambitious programme aiming at the development of robust metrology for the mechanical characterisation of key microstructural features in hardmetals.

The proposed work can be summarised in the following objectives:

Objective 1
To demonstrate repeatability of the experimental setup by testing microsamples machined from a very fine grained material (<0.2 μm grain size) or submicron grade (0.5-0.1 μm). A set of microsamples starting from the same material will be produced to similar dimensions and geometries and tested by the contractors. Results obtained at the different sites will be compared.

Deliverable: Report on the repeatability of the experimental outcome (load vs. displacement curves) for microsamples with similar dimensions tested using the same parameters.

Objective 2
To evaluate the ability of microsample testing to capture the effect of the orientation of the WC grains on its mechanical behaviour. A coarse grained sample will be mapped by EBSD and microsamples (beams and pillars) with different orientations relative to the crystal orientation will be machined and tested. This is the first step to describe the behaviour of WC grains.

Deliverable: Report on the experimental outcome for WC microsamples machined from coarse grains within a hardmetal with different orientations relative to the crystal orientation.

Objective 3
To evaluate the ability of microbeam testing as described in [15] to discriminate interfaces. Similar microsamples will be machined from two hardmetal grades with coarse grains and known to have different interfacial properties (different C activity and/or different impurities). The load-displacement curves will be compared and related if possible with the observed failure/crack path and the grain orientations.

Deliverable: Report on the experimental outcome from first testing strategy attempted to discriminate interfaces in hardmetals.

All the deliverables will be gathered in a single Final Report. No FEM modelling to define precise stress values is included in this project, but would be used in further stages.

Rationale and Introduction

Hardmetals exhibit an outstanding combination of hardness, toughness and wear resistance which makes them suitable for a wide variety of applications (i.e. metal cutting or shaping, mining or even as structural elements). A deep knowledge of the mechanisms that control the final properties is needed for improving their in-service life and these mechanisms depend mainly on their microstructural characteristics. There are a number of works that study the correlations between hardness, fracture and fatigue in hardmetals having the WC grain size distribution, the binder phase mean free path and the initial population of defects as critical microstructural parameters [1]. However, for a proper description of crack initiation and propagation phenomena it is critical to understand the mechanisms related to WC intergranular and intragranular fracture, the rupture of Co ligaments and the properties of the Co-WC interfaces [2, 3]. During the last years, several micromechanical models have been developed to study crack propagation phenomena in hardmetals based on the plastic deformation of the binder phase [4], the contiguity of WC grains, their shape and grain size distributions [5] and even the anisotropy of the different phases [6]. Nevertheless, these models use the bulk properties of WC and Co in their constitutive equations, which are far from those of WC grains and Co ligaments in the hardmetal microstructures. It is also important to note the effort made within the EPMA-EHMG to develop a Finite Element (FE) micromechanical model for crack propagation under fatigue (Projects Simucrack I & II) [7, 8].

There are many works in literature which use micro samples to study the size effect in plastic deformation of small volumes of material. Compression of micro pillars [9], tensile testing [10] and cantilever bending [11] are typical examples of these types of tests and are applied to several metallic alloys. These techniques have been also applied in microelectronics for the study of brittle and ductile fracture in thin films [12, 13] and recently even to ultrafine WC-Co hardmetals [14] for studying the test piece size effect in their fracture strength. In this work the cantilevers had sizes from about 16x10x60 μm to 6x9x40 μm and were machined from hardmetals with ultrafine WC grains (0.2-0.5 μm). This means that the hardmetal can be considered as a homogeneous material during the test. A first attempt to characterise individual hardmetal features is presented in [15] by CEIT, where a hardmetal with a grain size of 6 μm is used to machine cantilevers with a section of ~1 μm^2, placing a WC grain at the clamping. Nevertheless, so far no information has been published on the mechanical behaviour either of Co ligaments or WC grains as a function of their characteristic size [16].

In summary, despite the important effort at developing new micromechanical models to study in-service behaviour of hardmetals, reliable information on the mechanical properties of the different phases and interfaces at their characteristic size is still lacking. **The present proposal is aimed at exploring the potential of microsample testing for analysing the local properties of the different phases present in hardmetals** as a first stage to develop robust metrology. Microsamples with different geometries will be machined using a focused ion beam and tested with a nanoindenter system to provoke fracture of the feature of interest. Results will be interpreted in terms of load-displacement curves and fracture loads:

no FE modelling will be performed to extract the properties of individual features. The main advantage of this approach is that microsamples are machined from actual materials (fig. 1), that is the effect of different processing routes or in-service conditions on the properties of individual features could be assessed.

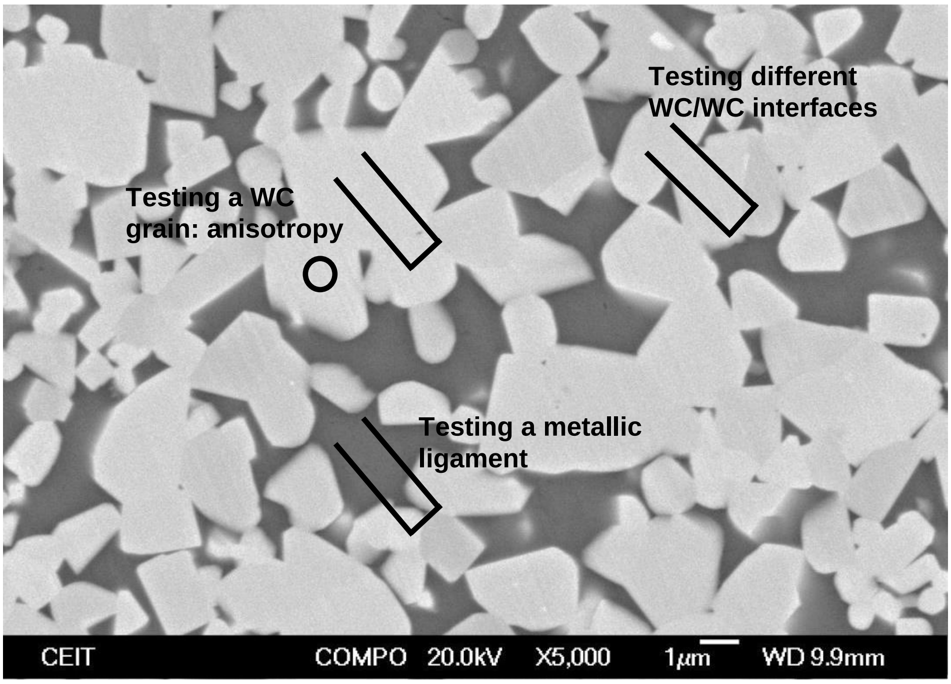
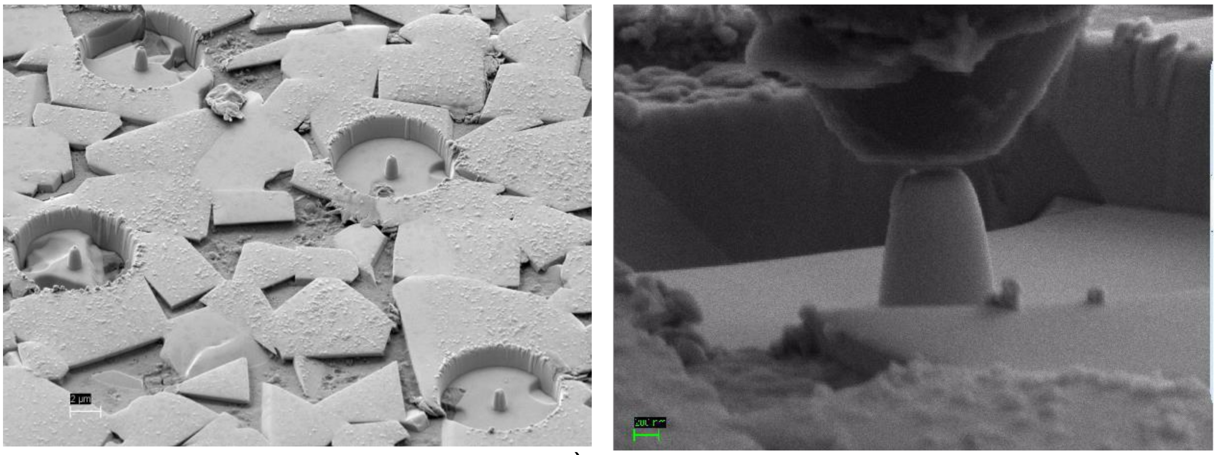


Figure 1. Microstructure of a WC-25wt%Co showing possible locations to machine microsamples with a FIB to study different individual features: WC grains, WC/WC interfaces and metallic ligaments.

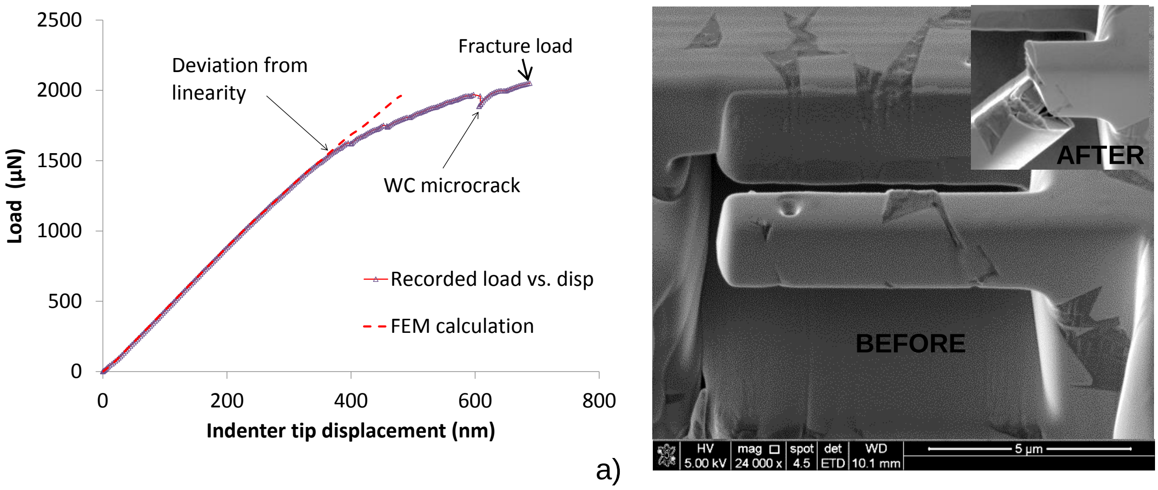
Several factors would affect the repeatability of the tests performed either on cantilevers or pillars, such as dimensional errors, damage induced by the ion beam or errors due to uncertainties in force and displacement measurements. In this project a systematic evaluation of uncertainty budgets is not contemplated but a first study on the repeatability of the measurements in terms of load-displacement curves is mandatory. For this purpose a hardmetal with an ultrafine or submicrometric grain will be used as starting material to machine different microsamples. This way the variability related to the microstructure will be avoided. Samples nominally with the same geometries and dimensions will be machined and tested in different sites, but a full Round Robin is not foreseen.

For WC grains an orientation-dependent behaviour is expected. To evaluate the ability of microsample testing to capture the behaviour of individual features it is proposed to map a coarse grained hardmetal by EBSD, to identify grains covering a wide range of orientations within the inverse pole figure and to mill pillars and beams just within a single WC grain, at least for pillars. Then the microsamples will be tested with a nanoindenter (fig. 2) and results compared in terms of load-displacement curves depending on the dimensions and the orientation relative to the crystal orientation. Results obtained for the same dimensions and testing conditions but milled and tested with different equipments (by the different contractors) will also be compared. A failure analysis will be performed *in-situ* or after the test in a scanning electron microscope (SEM) to relate the load-displacement curves with different deformation/fracture events.



a) b)

Figure 2. a) Hardmetal pillars machined on individual grains within a hardmetal sample and b) tested in-situ at a nanoindenter. Courtesy of NPL.



a) b)

Figure 3. Beam FIBed from a coarse WC-11wt%Co grade with the clamping at a WC grain and tested at a naoindenter. a) Load-displacement curve recorded and b) the beam before and after the test. Fracture occurs at WC/WC interfaces and is related with deviation from linearity and drops in the load-displacement curve (from [15]).

The interfacial properties are one of the key parameters needed to gain insight on the behaviour of hardmetals. Two coarse grain grades known to have different interfacial properties are going to be used as reference materials to evaluate the capability of micro-beam testing to discriminate between different interfaces. The goal is to place a WC grain at the clamping, as done in [15] and shown in figure 3, and to bend the sample using a nanoindenter system to provoke fracture. The beam microstructures will be mapped with EBSD and analysed with a FEG-SEM to determine the microstructural feature responsible for fracture initiation and propagation. This analysis will also be critical to assess the capability to discriminate interfaces as at this stage only the load-displacement curves are going to be compared. Therefore to obtain meaningful results the beams should have the same dimensions and microstructure. The goal will be to obtain similar load-displacement curves at low loads but as the load increases to have deviation from linearity for different load levels and/or different fracture loads.

References

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Proposed Project Management

The EPMA will oversee the project and ensure that partners meet their commitments. The EPMA will use various methods (web, etc) to ensure satisfactory dissemination of the project outcomes.

Proposed Project Costs

The cost to each participating industrial partner would be dependent on numbers taking part but at least 50% of the research at NPL will be co-funded by other NPL projects, reducing their costs to €6k.

The overall cost of the project to the project partners would be €27.12k (excluding VAT if applicable*).

Costs would be **shared equally** between the industrial participants. Payment would be made in several instalments.

** Non UK participants do not pay VAT provided they give their VAT number to the EPMA. UK participants have to pay VAT regardless and then reclaim it.*

The industrial participants will be responsible for the Selection and Manufacture of Test Specimens (WP0).

If you are interested in the project please sign and return to the EPMA the following Consortium Agreement.

If you are not an EPMA member or would like more information please contact Dr Olivier Coube, EPMA Technical Director, oc@epma.com

EPMA European Hard Materials Group
Micro-mechanical testing: a quantitative method for measuring local mechanical properties in hardmetals. Stage 1: Exploratory study

Consortium Agreement

Issued March 2015

The Project - As defined in Annex 1
The Contractors –

- Materials Department CEIT, Paseo de Manuel Lardizabal 15, 20018 Donostia-San Sebastián, Spain: **CEIT**
- Materials Division NPL, Hampton Road, Teddington, Middlesex TW11 0LW, UK : **NPL**
- CIEFMA UPC, Av. Diagonal 647 - Pavellón E, 08028 - Barcelona, Spain. : **UPC**

The Coordinator –

- The European Powder Metallurgy Association, Talbot House, 2nd Floor , Market St., Shrewsbury SY1 1LG, England: **EPMA**

The Members - **paid up corporate <u>EPMA members*</u>** funding the Project
The Participants – The Contractors and the Members

UV = unanimous vote; MV = majority vote of 2/3 members or higher
Project Fee = full fee paid at start of Project Stage 1

Heads

1. The Members and Contractors agree to cooperate in order to complete the Project according to Annex 1.

2. All data generated under the Project will remain confidential to the Members during the Project and for THREE years after delivery of the final written report to Members, and may only be disclosed to third parties (e.g. for dissemination purpose in PM Congress) with UV of the Members.

3. The Contractors agree to not carry out a similar project on hardmetals with organisations other than the Members until the completion of the project (delivery of the final report).

4. The Members agree to share equally the cost of the Project (EUR 27120) through a Project Fee of maximum **EUR 6780** per Member, excluding the Work Package 0 (Selection and Manufacture of Test Specimens). The required minimum number of Members is **four** unless the Members agree to exceed the maximum Project Fee.

5. **VAT**: The Project Fee is excluding VAT if applicable. Non UK participants do not pay VAT provided they give their VAT number to the EPMA. UK participants have to pay VAT regardless and then reclaim it.

6. The Members also undertake to provide the Contractors with the necessary test specimens and their appropriate surface preparations (Work Package 0 “WP 0”). If no agreement on in-kind contribution between the industrial partners can be found, the EPMA will coordinate the WP 0 and charge equally each Member to cover the cost of WP0 plus an administrative fee of 13%.

7. Payment of fees must be made promptly on receipt of invoice by TT to the nominated EPMA account.

    - 50% at the start of the project

    - 50% after completion of the project stage and delivery of the Final report.

8. New paying members may be admitted during the Project by UV on payment of full Project Fee plus a reasonable premium (10%). No Participation and access to the Project's results and deliverables is possible after the completion of the project.

9. Each Participant will retain the Intellectual Property for any other outcomes of the project. The Intellectual Property from the 'Micro-mechanical testing: a quantitative method for measuring local mechanical properties in hardmetals. Stage 1: Exploratory study' reports shall be owned by the Members.

10. The contractor's warranty extends solely to the use of due scientific diligence and to compliance with accepted engineering practice. The contractors do not guarantee that the desired objectives of the research and development project will be achieved.

11. Liability. Each Party is liable solely for wilful actions and gross negligence. Liability for proven damage is limited to the amount of the contractual sum.

12. Coordination will be by the EPMA, who will have responsibility for invoicing, day to day liaison with the Contractors and keeping Members informed. The EPMA will operate under the same confidentiality agreement as Members and the EPMA President will be arbitrator for unresolved disputes by the Members. Should the Parties fail to do so, then such dispute shall be subject to the exclusive jurisdiction of the English Courts. The laws of England and Wales govern all matters arising out of or relating to this agreement, and all transactions contemplated hereby, including, without limitation, its validity, interpretation, construction, performance and enforcement.

13. Except for the terms 4, 7, 8, 10, 11, 12, 13 and 12 all the terms of this agreement may be changed by UV of the Members.

Signatures: signed individually by all Members and Contractors

ORGANISATION:

VAT NUMBER:

NAME:

DATE:

SIGNATURE:

***If you are not an EPMA member please contact Dr Olivier Coube, EPMA Technical Director, oc@epma.com**

Annex 1: Work Packages in the Project:
Micro-mechanical testing: a quantitative method for measuring local mechanical properties in hardmetals. Stage 1: Exploratory study

1. WP 0 Selection of Materials

Programme: Industrial partners and contractors to agree on a set of hardmetals including:

- One very fine grained (<0.2 μm grain size) or submicron grade (0.5-0.1 μm) hardmetal. Three pieces from the same material (for WP1)
- Two coarse grain hardmetals with different interfaces. Three pieces are needed from one grade (for WP2) and one piece from the other (WP3)

Any specific dimensions are required but rectangular shape is preferred with corners at 90º.

Distribution of tasks:

- Selection of materials: **all partners.**
- Supply of material data: **Industrial Partners.**
- Surface preparation: industrial partners to supply blanks for common grinding route. **Industrial Partners**

Cost:
- Contribution by Members (industrial partners): cost not included here. If no agreement on in-kind contribution between the industrial partners can be found, the EPMA will coordinate the WP0 and charge equally each Member to cover the cost of WP0 plus an administrative fee of 13%.

Estimated duration: Months1-2 **Deliverables**: Test samples

2. WP 1 Testing microsamples: repeatability.

Programme: CEIT, NPL and UPC will mill sets of microsamples to similar dimensions and geometries (pillars, beams) to evaluate the repeatability within each Institute. The dimensions and geometries will be chosen at each site depending on the characteristics of the equipment used. Then one geometry and dimension will be chosen to perform one set of tests at each site and compare results. All the results will be compared in terms of load-displacement curves recorded during the test. FEG-SEM will be used to measure the dimensions of the microsamples and for failure analysis. The report will be included in the final report.

Distribution of tasks: CEIT, NPL, UPC:

- Testing sets of pillars with similar dimensions at each site (milling the microsamples, measurement of dimensions, testing and failure analysis)
- Testing sets of beams with similar dimensions at each site (milling the microsamples, measurement of dimensions, testing and failure analysis)
- Testing one set of microsamples at each site with similar geometry and dimensions

Estimated duration: Months 2-5. **Deliverable**: Report on the repeatability of the experimental outcome (load vs. displacement curves) for microsamples with similar dimensions tested using the same parameters.

3. WP 2 Mechanical testing of WC pillars/beams: orientation effect

Programme: CEIT, NPL and UPC will use as starting material a coarse grained sample to locate grains with different orientations mapping by EBSD and then WC pillars with different orientation of the axis relative to the crystal orientation will be milled. The sensitivity of the technique proposed to the orientation of the WC grains will be studied. The repeatability within each institute will also be reported. Moreover, a set of pillars will be prepared at different institutes with the same dimensions and orientations (to be agreed) for a preliminary study on repeatability. Beams with different microstructures (only WC, WC at the clamping) will be evaluated in order to produce controlled crack propagation through the WC. The tests outcome will be interpreted in terms of load-displacement curves. The relation between different events in the curves and failure events will be studied mainly using the *in-situ* testing capabilities available at NPL. In any case failure analysis after the tests will be performed at the FEG-SEM by the contractors. The report will be included in the final report.

Distribution of tasks: CEIT, NPL, UPC

- Identify WC grains with different orientation using EBSD mapping and testing sets of WC pillars with similar dimensions and different orientation at different institutes
- Testing one set of pillars at each site with similar geometry and dimensions
- Explore the experimental setup needed to produce controlled cracking of WC grains when testing beams

Estimated duration: Months 3-8. **Deliverable**: Report on the experimental outcome for WC microsamples machined from coarse grains within a hardmetal with different orientations relative to the crystal orientation.

4. WP 3 Mechanical testing of microbeams: ability to discriminate interfaces

Programme: CEIT will mill microbeams with the same geometry and microstructure starting from two coarse grained hardmetals known to have different interfacial properties. The microstructure of the beam will be characterised by EBSD and fracture analysis after the test will be performed at the FEG-SEM. The load-displacement curves will be analysed in terms of fracture load and load needed for deviation from linearity in order to assess the capability of the technique to discriminate interfaces. Results will be included in the final report.

Distribution of tasks: CEIT

- Characterisation of the beams: microstructure and dimensions.
- Testing the beams at the nanoindenter.
- Fractography.

Estimated duration: Months 4-5. **Deliverables:** Report on the experimental outcome from first testing strategies attempted to discriminate interfaces in hardmetals.

5. WP 4 Project Management and Reporting

Programme: The EPMA will oversee the project and ensure that CEIT and partners meet their commitments. The EPMA will use various methods (web, etc) to ensure satisfactory dissemination of the project outcomes.

Distribution of tasks:

- Project Management: **EPMA**
- Final Report: **EPMA, CEIT, NPL, UPC**

Estimated duration: WP 0-3 + 1 Month for Report

<u>Costs:</u>
CEIT: €12k (WP1, WP2 and WP3); **NPL**: €6k (WP1 and WP2); **UPC**: €6k (WP1 and WP2);
EPMA: ca. 13% Administrative cost of WP 0-3 (WP4 + Travels): €3.12k
Total Cost: WP1-4 = <u>€27.12k</u> (excluding VAT if applicable and WP 0)
Estimated total duration: ca. 8 <u>Months</u>

6. <u>Proposed Project Timetable:</u>

Practical work in the project would commence once the samples as decided in WP0 are available. Two meetings with all contractors would be held, a kick-off meeting and one wind-up meeting at project completion. Work at partner organisations to prepare materials should start as soon as the project has sufficient members to meet the financial requirements.

	Month							
	1	2	3	4	5	6	7	8
Kick-off meeting / Agreement of Programme	■							
Selection of Materials	■							
Supply of Material	■	■						
Final meeting								■
WP1. Testing micosamples: repeatability								
Testing sets of pillars		■	■	■				
Testing sets of beams			■	■				
Testing one set of pillars/beams at each site, same dimensions				■	■			
WP2. Testing WC pillars/beams: orientation effect								
Identify WC grains using EBSD and testing pillars at different sites			■	■	■	■		
Testing one set of nominally identical pillars at each site						■	■	■
Explore beam testing to produce controlled cracking of WC grains						■	■	
WP3. Microbeams: ability to discriminate interfaces								
Characterisation and milling of the microbeams				■				
Testing microbeams					■			
Fractography						■		