Micro-mechanical testing: a quantitative method for measuring local mechanical properties in hardmetals - Stage 2

“MicroMech 2”

EPMA European Hard Materials Group EuroHM
Research Proposal – May 2018

<u>Consortium Agreement</u>
Issued May 2018

The Project - As defined in Annex 1
The Contractors –

- Division of Materials and Manufacturing Ceit-IK4, 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>**[1] funding the Project
The Participants – Paid up EPMA Members 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

<u>Heads</u>

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 60000) through a Project Fee of maximum **EUR 15000** 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. The Members who did not participate in the project “MicroMech” may participate in the MicroMech 2 project by settling a “MicroMech results access premium” of EUR 3,874 at the start of the

[1] **<u>If you are not an EPMA member please contact Dr Olivier Coube, EPMA Technical Director, oc@epma.com</u>**

MicroMech 2 Project. The premium less a 13% EPMA Management fee will be used to decrease the MicroMech 2 Project Fee for the Kinetic Consortium Members.

6. **VAT**: VAT will be added to the Project Fee as appropriate but may be reclaimed according to local arrangements (e.g. “Reverse Charge” mechanism). All VAT numbers are to be provided to the EPMA.

7. 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%.

8. 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.

9. 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.

10. Each Participant will retain the Intellectual Property for any other outcomes of the project. The Intellectual Property from the “MicroMech 2” reports shall be owned by the Members.

11. 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.

12. 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. The Member is not liable for this project.

13. 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.

14. Except for the terms 4, 9, 11, 13, 14 and 13 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:

Annex 1

Micro-mechanical testing: a quantitative method for measuring local mechanical properties in hardmetals - Stage 2

"MicroMech 2"

Project Proposal

The proposed project focus on two aspects of microsample testing to measure mechanical properties of hardmetals at the local scale: the effect of geometry on the repeatability of the tests and the characterisation of interfacial strength. In a previous exploratory study, microsamples of different geometries (beams, pillars) were machined using FIB and tested with different nanoindenter systems. The measured force-displacement information obtained demonstrated good repeatability, captured the effect of WC orientation for fracture and discriminated WC-WC interfaces with different relative orientation. This project is the second 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 evaluate sources of errors, focusing on geometry, by testing micropillars machined from small grained samples and WC microbeams machined from ultra coarse grained materials. Microsamples will be milled by the three contractors to similar dimensions and geometries and tested at one institution. Some analytical and FEM modelling will be performed to evaluate the error budget of different geometric parameters.

Deliverable: Report on the effect of the geometry on repeatability of the experimental outcome (load vs. displacement curves) for microsamples with similar dimensions milled by different institutions and tested at one institution.

Objective 2
To characterise the strength of WC-WC interfaces with different relative orientation. EBSD maps will be performed on ultra coarse grained materials to identify CSL2 and no CSL2 interfaces and then microbeams will be milled with these interfaces close to the fixed end and perpendicular to their longitudinal axis. The load-displacement curves will be compared and related to the observed failure/crack path. Strength will be calculated using analytical models and some tests will be modelled using FEM to quantify the limitations of the analytical models.

Deliverable: Report on the strength of WC-WC interfaces with different relative orientation.

All the deliverables will be gathered in a single Final Report. FEM modelling is not going to be used extensively to extract properties from the experimental outcome but to identify trends and quantify limitations of analytical approaches to calculate stress values from load-displacement curves.

Some of the contractors propose to supervise a Master Thesis, which will be in direct association with the "Micromech 2" project and hence to apply for extra funding provided interested students are found:

1- Ceit-IK4: The Master Thesis will focus on FE modelling to evaluate the geometric budget error and the limitations of the analytical models to extract mechanical properties. The Master Thesis report will be added to the "MicroMech 2" Final Report.

2- UPC/CIEFMA: The Master Thesis will focus on strength of cantilevers. The Master Thesis report will be added to the "MicroMech 2" Final Report.

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-EuroHM to develop a Finite Element (FE) micromechanical model for crack propagation under fatigue (Projects Simucrack I, II & III) [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 was presented in [15] by Ceit-IK4, 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].

Considering the important effort at developing new micromechanical models to study in-service behaviour of hardmetals and the lack of reliable information on the mechanical properties of the different phases and interfaces at their characteristic size it is clear that robust metrology is needed. A first Club Project aiming at exploring the potential of microsample testing for analysing the local properties of the different phases present in hardmetals was conducted. 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. Microsamples with different geometries were machined using a focused ion beam and tested with a nanoindenter system to provoke fracture of the feature of interest. Load-displacement curves were recorded and stress-strain curves were calculated using analytical models to calculate Young's modulus and strength. Results obtained from the **first stage of Micromech Project** were very good. First, r**epeatability was demonstrated** with a fine grain grade for pillars and cantilevers despite the lack of a common protocol to mill samples and specially to measure the dimensions. Moreover, it was noted that the grade chosen was not fully homogeneous at the scale tested and hence the microstructure was introducing some variability in the results. Secondly, the **effect of WC orientation was clearly captured for fracture** and finally, **WC-WC interfaces with different relative orientation were discriminated**. CSL2 interfaces were stronger than no CSL2 interfaces, as predicted from computational studies of WC-WC interfaces performed using density functional theory [17, 18]. In order to progress towards the development of robust metrology at the scale of interest, a **second stage of Micromech Project** is proposed focusing on two objectives. On one hand, we will evaluate the **sources of errors focusing on the geometry** and, on the other hand, we will characterise the **strength of WC-WC interfaces with different relative orientation**. As in the previous stage, the results will be compared in terms of load-displacement curves and stress-strain curves calculated using analytical models, but some FE modelling will be also performed. All the samples will be observed at the scanning microscope to rationalise the results.

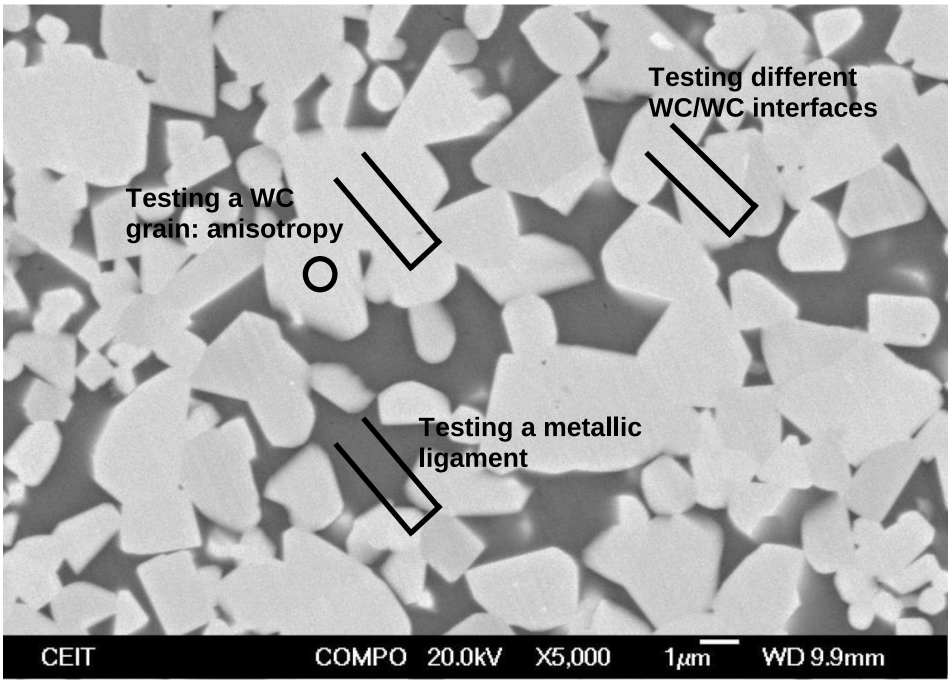

Figure 1. Microstructure of a WC-25wt%Co showing possible locations to machine microsamples with a FIB to study different individual features: WC grains, WC/WC interfaces and metallic ligaments.

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 second stage project, as in the first stage project, a systematic evaluation of uncertainty budgets is not contemplated. However, it was concluded that in order to obtain reliable results geometry was a key factor. Therefore, the first objective in terms of improving repeatability is to establish a protocol to mill microsamples and, more important, to measure their dimensions. For this purpose, a hardmetal with smaller grains will be used as starting material to machine pillars. In the case of cantilevers, a coarse grain grade will be used to machine the beams within a single WC grain. This way, cantilever results will be a reference to the results obtained from WC-WC characterisation. Microsamples will be machined at different sites but tested *in-situ* at one site.

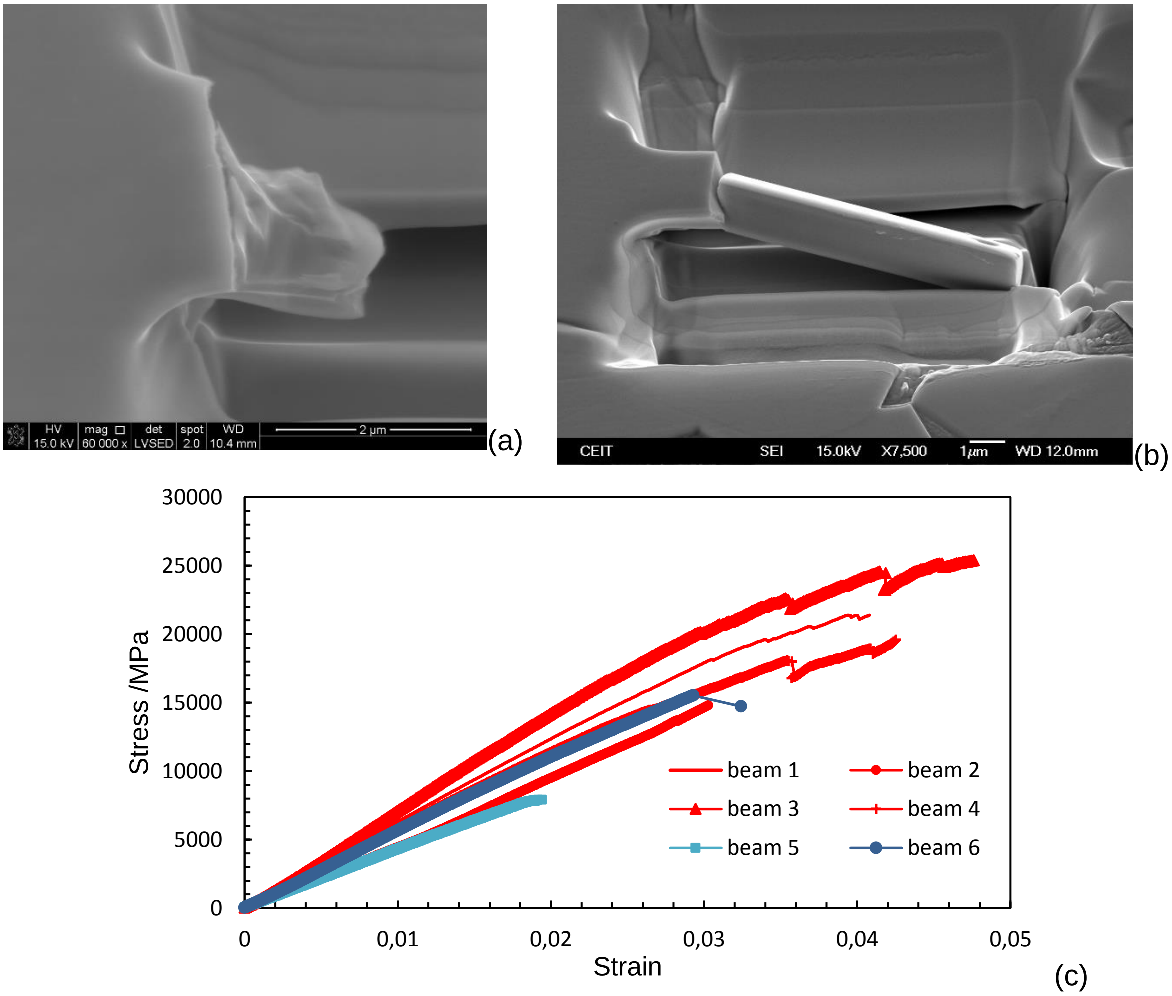

Figure 2. SEM images of cantilevers milled on a coarse grain WC-Co hardmetal with (a) CSL2 interface and (b) no CSL2 interface close to the fixed end after bending test. (c) Corresponding stress-strain data for cantilever beam bending with in red CSL2 interface and in blue no CSL2 interface. Note data for beams 1, 2& 4 is incomplete as they touched the substrate before complete fracture.

The interfacial properties are one of the key parameters needed to gain insight on the behaviour of hardmetals. For this reason, it is considered key to develop metrology for interfacial characterisation. In the first exploratory stage, CSL2 and no CSL2 interfaces were discriminated testing cantilevers with different interfaces placed at the same distance from the fixed end and perpendicular to the cantilever axis. The cantilevers were milled from coarse grain grades. In order to identify the interfaces, the sample was previously mapped with EBSD. Even if a small number of tests were performed, the ability of micro-beam testing to discriminate interfaces with different relative orientation was clearly proven (Fig. 2). In this second stage project, the work will be completed and the strength of CSL2 and no CSL2 interfaces will be measured. The goal is not to develop new testing setups but to study the advantages and limitations of the setup described in the first exploratory project. More cantilevers will be tested to fracture and stress-strain curves will be calculated from the recorded load-displacement curves to extract the interfacial strength. To study the limitations of the analytical results, some tests will be simulated using FEM. The results will also be compared to the cantilever tests performed on single WC grains.

References

1. Roebuck, B and Almond, EA, *International Materials Reviews* 33: 90-110 (1988)
2. Sigl, LS and Exner, HE, *Metall Trans A* 18A: 1299-308 (1987)
3. Mingard, KP, Jones, HG, Gee MG, Roebuck, B and Nunn, JW, *Int. Journal of Refractory Metals and Hard Materials* 36: 136-142 (2013)
4. McHugh, PE and Connolly, PJ,. *Computational Materials Science* 27: 423-436 (2003)
5. Kim, C-S, Massa, TR and Rohrer, GS, *Int. Journal of Refractory Metals and Hard Materials* 24: 89-100 (2006)
6. Kim, C-S., Massa, TR and Rohrer, GS, *J. Am. Ceram. Soc.* 90: 199-204 (2007)
7. Özden UA, Bezold A, Broeckmann C, *Procedia Materials Science* 3: 1518-1523 (2014)
8. Özden UA, Mingard KP, Zivcec M, Bezold A and Broeckmann C, *Int Journal of Refractory Metals and Hard Materials* 49: 261-267 (2015).
9. Uchic, MD, Dimiduk, DM, Florando, JN and Nix, WD, *Science* 305: 986-9 (2004)
10. Kiener, D, Grosinger, W, Dehm, G, and Pippan R, *Acta Mater* 56: 580-92 (2008)
11. Motz, C, Schöberl, T, Pippan, R, *Acta Mater* 53: 4269-79 (2005).
12. Matoy, K, Schönkerr, H, Detzel, T, Dehm, G, *Thin Solid Films* 518: 5796-5801 (2010)
13. M. Trueba, D. Gonzalez, M.R. Elizalde, J.M. Martínez-Esnaola, M.T. Hernandez, H. Li, D. Pantuso, I. Ocaña, *Thin Solid Films* 571: 296-301 (2014).
14. Klünsner, T, Wurster, S, Supancic, P, Ebner, R, Jenko, M, Glätzle, J, Püschel A, Pippan R, *Acta Mater* 59: 4244-4252 (2011).
15. M. Trueba, A. Aramburu, N. Rodríguez, I. Iparraguirre, M.R. Elizalde, I. Ocaña, J.M. Sánchez, J.M. Martínez-Esnaola, *Int Journal of Refractory Metals and Hard Materials* 43: 236-240 (2014).
16. Greer, JR, De Hosson, JM, *Progress in Materials Science* 56: 654-724 (2011).
17. Petisme M.V.G., Johansson S.A.E. and Wahnström G., *Modelling Simul. Mater. Sci. Eng.* 23:045001 (2015).
18. Johansson S.A.E., Petisme M.V.G. and Wahnström G., *Computational Materials Science* 98: 345-353 (2015).

Work Packages – Budget – Timetable

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). Three pieces from the same material (for WP1)
- One very coarse grain hardmetals. Three pieces are needed from the same material (for WP1 & WP2)

Any specific dimensions are required (prismatic or cylindrical) but top and bottom surface should be parallel.

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: errors introduced by geometry.

Programme: CEIT, NPL and UPC will mill pillars from very fine grained samples and WC cantilevers from ultra coarse grained samples. In all cases, microsamples will have similar dimensions as determined by NPL. The microsamples will be measured at each site as agreed. All the microsamples will be tested *in-situ* to fracture at NPL in order to study reproducibility. All the results will be compared in terms of load-displacement curves recorded during the test and stress-strain curves calculated from analytical models. Some analytical and FE modelling will be performed at CEIT to study the error introduced by different geometric parameters The report will be included in the final report.

Distribution of tasks: CEIT, NPL, UPC:
- CEIT: Milling pillars and cantilevers with similar dimensions and measurement of their dimensions. Analytical and FEM modelling.
- NPL: Milling pillars and cantilevers with similar dimensions, measurement of the dimensions of all the microsamples (milled also by CEIT & UPC). Testing *in-situ* all the microsamples (milled also by CEIT & UPC).
- UPC: Milling pillars and cantilevers with similar dimensions and measurement of their dimensions.

Estimated duration: Months 2-11. **Deliverable**: Report on the evaluation of the effect of errors in geometry measurement on the reproducibility of microsample testing.

3. WP 2 Testing microbeams to extract interfacial strength of WC-WC interfaces with different relative orientation

Programme: CEIT and UPC will mill microbeams from an ultra coarse grade with WC-WC interfaces close to the fixed end and perpendicular to the cantilever axis. The WC-WC interfaces will have different relative orientation: CSL2 and no CSL2. The microstructure of the samples will be maped by EBSD in order to select the location of the cantilevers. At each site, the dimensions of the cantilevers will be measured following indications from WP1 and tested to fracture. After the test, all the samples will be analysed at the FEG-SEM. Stress-strain curves will be calculated from the recorded load-displacement curves. CEIT will simulate some tests using FEM to evaluate the limitations of the analytical model. Finally, a limited number of cantilevers milled at CEIT and/or UPC will be tested *in-situ* at NPL. Results will be included in the final report.

Distribution of tasks:
- **CEIT**: EBSD mapping, milling of the cantilevers, testing cantilevers, fractographic analysis, analytical modelling, FE modelling of selected cases.
- **UPC**: EBSD mapping, milling of the cantilevers, testing cantilevers, fractographic analysis, analytical modelling.
- **NPL**: Testing in-situ a limited number of cantilevers milled at CEIT and/or UPC.

Estimated duration: Months 4-11. **Deliverables:** Report on the strength of WC-WC interfaces with different relative orientation in hardmetals.

4. WP 3 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.

The contractors CEIT, NPL, UPC should provide the consortium with short progress reports every three months, an interim report at mid-term, minutes and presentations after each meeting or teleconference and a final report at the end of the project.

Distribution of tasks:
- Project Management: **EPMA**
- Minutes, Progress, Interim and Final Reports: **CEIT, NPL, UPC**

Estimated duration: 12 months WP 0-2 + 1 Month for Report

5. Budget

The following budget is planned for the MicroMech 2 project:

CEIT: €23.85k (WP1 and WP2); **NPL**: €18.55k (WP1 and WP2); **UPC**: €10.6k (WP1 and WP2); **EPMA**: ca. 13% Administrative cost of WP 1-3: €7k

Total Cost: WP1-3 = €60k (excluding VAT if applicable and WP 0)

6. Proposed Project Timetable and Reporting:

Practical work in the project would commence once the samples as decided in WP0 are available. Three meetings with all contractors would be held, a kick-off meeting, a mid-term meeting (physical or teleconference) 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.

Estimated Timetable for MicroMech 2

	Month											
	1	2	3	4	5	6	7	8	9	10	11	12
Kick-off meeting / Agreement of Programme	■											
Selection of Materials	■											
Supply of Material	■	■										
Reports/meetings	■						■					■
WP1. Testing microsamples: errors introduced by geometry												
Milling sets of beams		■	■	■								
Testing beams			■	■	■					■	■	
Milling sets of pillars						■	■	■				
Testing pillars							■	■	■			
Analysing results/modelling			■	■	■	■	■	■	■	■	■	■
WP2. Strength of WC-WC interfaces with different relative orientation												
Identify WC-WC interfaces using EBSD				■								
Milling cantilevers with different WC-WC interfaces					■	■	■		■	■		
Testing cantilevers						■	■	■	■	■		
Fractographic analysis								■	■	■	■	
Analysing results/modelling							■	■	■	■	■	■

Estimated total duration: ca. 12 Months