FINAL BACHELOR'S PROJECT

Materials Engineering Degree

FRACTURE AT SMALL SCALE OF WC GRAINS OF HARDMETALS

Report

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Abstract

Hardmetals are used in different industrial sectors in demanding applications such as cutting and mining tools because of their good combination of properties such as hardness, fracture toughness and high resistance to wear. These are a direct consequence of their composite structure of interpenetrating networks of the ceramic phase, usually tungsten carbide, and a metallic binder, usually cobalt.

In order to improve these material properties and prevent from failures, a deeper knowledge of mechanisms controlling strength and toughness are needed. These mechanisms depend mainly on their microstructural characteristics. For a proper description of crack propagation, it is important to understand the mechanisms related to tungsten carbide intergranular and intragranular fracture. Moreover, finite element micromechanical models have been developed for analysing crack propagation under fatigue conditions. Micro-sample testing appear to be a promising technique for providing reliable information of mechanical properties of different phases of the material.

The purpose of this final Bachelor’s project is to perform micromechanical tests in tungsten carbide grains of a hardmetal in order to obtain results that will allow to understand these mechanisms of crack propagation and determine fracture toughness values.

For the determination of these values, micro-pillars and micro-cantilevers where prepared with focused ion beam technique in basal and prismatic orientation of tungsten carbide grains. The samples were tested by the nanoindenter based techniques of pillar spitting and cantilever bending.

Different results have been obtained depending on the location of the applied load because of the low nanoindenter precision, in micro-pillars, and depending on the dimension of the notch in cantilevers. Obtained values of fracture toughness have been compared between different techniques used in this project and with other values obtained by calculation of fracture toughness with nanoindentation technique.
Resum

L’ús dels metalls durs en diferents sectors de la indústria en aplicacions tan exigents com eines de tall o mineria, es degut a la seva bona combinació de propietats com l’alta dureza, tenacitat a fractura i excel·lent resistència al desgast. Aquestes propietats són conseqüència de la seva estructura formada per una fase ceràmica, normalment de carbur de tungstè i un lligant metàl·lic, normalment el cobalt.

Per tal de poder millorar les propietats d’aquest material i prevenir de possibles fallades, és necessari un ampli coneixement dels mecanismes que controlen la resistència i la tenacitat. Aquests mecanismes depenen, en major part, de les seves característiques microestructurals. Per obtenir una descripció apropiada sobre la propagació d’esquerdes, és important entendre els mecanismes relacionats amb la fractura intergranular i la intragranular dels carburs de tungstè. A més, s’han desenvolupat models micromecànics d’elements finits per analitzar la propagació d’esquerdes en condicions de fatiga. Per obtenir informació fiable de les propietats micromecàniques de les diferents fases, els assajos en micro-mostres semblen ser els més prometedors.

El propòsit d’aquest treball de fi de grau és la realització d’assajos micromecànics en els grans de carbur de tungstè d’un metall dur per obtenir resultats que permetran entendre aquests mecanismes de propagació d’esquerda i obtenir valors de tenacitat a fractura.

Per obtenir aquests resultats s’han preparat micro pilars i micro bigues en voladís amb el Focused Ion Beam en els grans amb orientacions basal i prismàtica de carbur de tungstè en un metall dur. A aquestes mostres se’ls ha realitzat assajos de ruptura de pilars i flexió d’una biga en voladís amb un nanoindentador per obtenir valors de tenacitat a fractura.

S’han obtingut diferents resultats en funció de la localització de la càrrega aplicada degut a la baixa presció del nanoindentador, en el cas dels pilars, i en funció de la dimensió de l’entalla en les bigues en voladís. Els valors obtinguts han estat comparats entre les dues diferents tècniques utilitzades en aquest projecte i amb altres valors obtinguts per càlcul de tenacitat a fractura per nanoindentació.
Resumen

El uso de los metales duros en diferentes sectores de la en aplicaciones tan exigentes como herramientas de corte y de minería, es debido a su buena combinación de propiedades como alta dureza, tenacidad a la fractura y excelente resistencia al desgaste. Estas propiedades son consecuencia de su estructura compuesta por una fase cerámica, normalmente el carburo de tungsteno, y un ligante metálico, normalmente cobalto.

Para poder mejorar las propiedades de este material y prevenir fallos, es necesario un amplio conocimiento de los mecanismos que controlan la resistencia y la tenacidad. Estos mecanismos dependen, en su mayor parte, de sus características microestructurales. Para obtener una descripción apropiada sobre la propagación de grietas, es importante entender los mecanismos relacionados con la fractura intergranular e intragranular de los carburos de tungsteno. Además, se han desarrollado modelos micromecánicos de elementos finitos para analizar la propagación de grietas en condiciones de fatiga. Para obtener información fiable de las propiedades micromecánicas de las distintas fases, los ensayos en micro-muestras parecen ser los más prometedores.

El propósito de este trabajo de fin de grado es la realización de ensayos micromecánicos en los granos de carburo de tungsteno de un metal duro para obtener resultados que permitan entender estos mecanismos de propagación de grieta y obtener valores de tenacidad de fractura.

Para obtener estos resultados se han preparado micro pilares y micro vigas en voladizo con el Focused Ion Beam en los granos con orientación basal y prismática de carburo de tungsteno de un metal duro. A estas muestras se les han realizado los ensayos de ruptura de pilares y flexión de una viga en voladizo con un nanoindentador para obtener los valores de tenacidad a la fractura.

Se han obtenido distintos resultados en función de la precisión del nanoindentador, en el caso de los pilares, y en función de la dimensión de la entalla en los vigas en voladizo. Los valores obtenidos han sido comparados entre las dos distintas técnicas usadas y con los valores obtenidos en otros proyectos por cálculo de tenacidad a la fractura por nanoindentación.
Acknowledgments

First of all, I would like to express my special thanks and gratitude to my supervisor Emilio Jiménez Piqué for trusting me and giving the opportunity of learning promising techniques that would really help me in my future career. Also, for his patience and positivism.

I want to thank Trifon Todorov Trifonov for the patience during my training. And I also want to Acknowledge to Núria Cuadrado Lafoz for staying with me in firsts tests with the nanoindenter.

To finish, I am very thankful to my friends and family that supported me every day.
Glossary of terms

**FIB:** Focused Ion Beam

**SEM:** Scanning Electron Microscope

**EBSD:** Electron Backscattered diffraction

**WC:** Tungsten carbide

**$K_c$:** Fracture toughness

**E:** Young's modulus

**H:** Hardness

**P:** Applied load
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1. Introduction

1.1. State of the art

On this Bachelor’s project, a determination of fracture toughness is done using a nanoindentation system of micro-pillars and micro-cantilevers micromachined by focused ion beam (FIB). The objective is to obtain a value of fracture toughness of WC grains in WC-Co hardmetals.

The use micro-pillar and micro-cantilever with nanoindentation techniques in micro-sized faces of WC has not been done yet, but there are several studies of this techniques for different materials, specially for thin coatings.

Here are some articles covering the field of calculation of fracture toughness by the use of different nanoindentation-based techniques and the preparation of micro-cantilever and micro-pillars by the use of the FIB microscope.

N. Cuadrado [1] studied the mechanical behaviour of small-volumes and micro-sized phases with nanoindentation technique is done. Different materials were chosen in this work and a study of the influence of crack morphology on nanoindentation fracture toughness was done.

The values of fracture toughness found for WC grains in hardmetals by cube-corner indentation technique will be used to compare the results obtained with indentation techniques done in this project. Also, the evaluation of the crack morphology will be useful to relate the results of tests with the fracture mechanisms of this material in the micro-scale.

In 2014, M. Sebastiani and coworkers [2] proposed a novel and relatively simple method for measuring the fracture toughness of thin films based on sharp indentation testing of FIB-milled micro-pilars. A finite element analysis was used in three-dimensional simulations of a rigid Berkovich indenter contacting an isotropic, elastic-perfectly plastic, brittle and cylindrical pillar. Simulations were used to estimate instability loads as a function of pillar radius and fracture toughness for different materials. They found that the load drop occurs at a unique value and proposed an equation for fracture toughness evaluation with pillar splitting results. They also propose an aspect ratio (height/diameter) of around 1.

The technique, the equation and the aspect ratio they propose was used in this project.
Recently, M. Ghidelli and coworkers [3] found that pillar splitting experiments revealed a linear relationship between the splitting load and the indenter angle. The results provide a simple and reliable way to measure fracture toughness over a broad range of material properties.

The curves found expressing the lineal relationship between splitting load and indenter angle were used to calculate the fracture toughness for the material of this project.

Another method was developed before by D. Di Maio and S.G. Roberts [4]: The micro-cantilever method for measuring fracture toughness was proposed. For the determination they used the equation for fracture toughness with fracture stress, crack length and a dimensionless shape factor dependent on sample geometry. They milled micro-cantilevers with house shape cross section and calculated a relation between applied load and specimen/crack geometry with simulations and found an equation for the shape factor for this geometry.

In this project the same house shape cross section geometry and same equation for the micro-cantilever bending tests has been used.
1.2. Objective

On this Bachelor’s project, the objective is to obtain a value of fracture toughness for WC grains with basal and prismatic orientations in WC-Co hardmetal. Furthermore, in order to find the most accurate value, the techniques of pillar splitting and cantilever bending for fracture toughness evaluation at the micro-scale have been tested and compared with the results of cube-corner indentation method in WC grains found by N. Cuadrado [1].

Thus, the project is divided in three different objectives:

1. To get a correct understanding of the FIB micromachining in order to mill micro-pillars and micro-cantilevers specimens with appropriate ratios and dimensions.
2. To carry out successful splitting and bending tests in order to determine a fracture toughness value.
3. To compare different fracture toughness values between different techniques and different grain orientations.

In general terms, the main scope of this project is to find the most accurate value of fracture toughness of WC grains by finding the best dimensions of specimens and the best conditions for tests.
2. Background

2.1. Hardmetals

Hard metals are a group of materials with a composite structure of ceramic phase (usually WC or TiC) in a tough and ductile metallic binder (usually cobalt or nickel) [5]. It is a versatile material because of his properties, that combine the hardness and stiffness of the ceramics and the toughness of the metallic phase.

Hard metals are widely used as cutting, forming and machining tools in different areas of industry due to their high hardness, wear-resistance and fracture strength, as well as drilling tools. When used under impact conditions, an improvement of the fracture toughness is highly demanded for cemented carbides to prolong the service life of the components.

Tungsten Carbide (WC) is a non-oxide ceramic where hexagonal closely packed layers of W atoms are separated by closely packed layers of C filling one-half of the interstices, giving rise to a six-fold trigonal prismatic coordination for the atomic structures.

The lattice shape is hexagonal, with lattice parameters $a=0.2906\text{nm}$ and $c=0.2837\text{ nm}$. The WC grains generate three types of facets: two types of prismatic facets and the basal $(0,0,0,1$ faced which delimit the flat triangular prism [6].

![Image of crystalline structure of WC and planes generated](image)

Figure 3.2.1. Crystalline structure of WC and planes generated [6].

WC grains in WC-Co system are crystals which mechanical properties depend on the crystalline orientation and grain size [7]. Knowing the relation between mechanical properties and crystalline
Fracture at small scale of WC grains of hardmetals

orientation allows a better optimization of microstructure and an improvement in hardness and fracture toughness.

The deformation characteristics of ceramic phase WC have been seldom reported in the literature. Some researchers proposed that WC grains may accommodate the deformation of WC-Co cemented carbides, because dislocations and/or stacking faults were observed in WC. Moreover, the anisotropic and inhomogeneous deformation were found in WC grains, which were related to certain slip systems of the WC crystal [8]. However, a complete understanding of the mechanics of this phase is still missing.

2.2. Fracture toughness

The concept of toughness as a measure of resistance emerged almost a century ago through the work of Griffith, who identified the crucial role of energy release rate in determining the structural integrity of brittle materials [9]. Griffith observed that a body containing a crack and subjected to external or residual stress acts like a spring that stores elastic strain energy. In order for the crack to grow, the volumetric elastic strain energy must be released, and then consumed to create fresh fracture surfaces.

Griffith wrote the relationship

$$\frac{\sigma^2 \pi a^4}{E} = G_C$$  

using the usual notation $E$ for Young’s modulus, and $a$ for half-length of a planar crack and introduced the term "material toughness", $G_C$.

Further work by Irwin proposed the alternative measure known as fracture toughness. Irwin placed the focus on the stress-strain distribution in the crack tip. Furthermore, a direct relationship was established between the two measures of resistance to cracking, toughness and fracture toughness:

$$\sigma^2 \pi a^4 = G_C E = K_{IC}^2$$  

In practice, for complex sample shapes, corrective geometric scaling factor $Y$ needs to be introduced. So crack extension occurs when the stress intensity factor $K_I$ expressed in terms of the stress $\sigma$ and crack half length $a$, reaches the critical value $K_{IC}$.

$$K_I = Y \sigma \sqrt{\pi a} = K_{IC}$$  

This criterion connects the material property, problem geometry, and loading conditions [10].
2.3. **Cube-corner Indentation fracture toughness method**

Sharp indentation-based models for fracture toughness evaluation rely on the direct measurement of radial cracks when indented with the sharp indenter such as Vickers or Berkovich diamond. The original Lawn-Evans-Marshall approach leads to the equation (4), where \( c \) is the average crack length, \( H \) is the hardness, \( P_{\text{max}} \) is the maximum load during the indentation and \( E \) is the Young's modulus [11].

\[
K_c = \frac{E}{H} \cdot \frac{P_{\text{max}}}{c^{3/2}}
\]

Several studies have shown that the application of the original model was appropriate only for the case of brittle bulk ceramics, where dimensions of the radial cracks are usually much larger than the size of the indentation mark ('half penny' crack geometry). Several other models have been proposed in the last few decades, to make different possible crack geometries and material properties into account.

A variation of the technique is to use a cube-corner indenter instead of a Vickers tip. The sharper cube-corner indenter produces much higher stress and strain in the vicinity of the contact, which is useful in producing very small, well defined cracks around residual imprint in brittle materials like ceramics materials and specially suited for nanoindentation.

When cube-corner indenters are used, the equation of Laugier [12] may be used to determine the \( K_c \). Where \( l \) is the crack length from the indentation corner, \( a \) is the indentation diagonal \( c = l + a \), see Figure 3.2.1.1.

![Figure 3.2.1.1. Crack parameters.](image)
The values for fracture toughness for tungsten carbide (WC) have been evaluated with other techniques that will be explained in the following chapters. The values reported by N. Cuadrado [13] are showed in Table 2.3.1.

Table 2.3.1.

<table>
<thead>
<tr>
<th></th>
<th>H (GPa)</th>
<th>E (GPa)</th>
<th>$K_c$ (MPa·m$^{1/2}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Prismatic facet</td>
<td>17.2 ± 0.1</td>
<td>564 ± 26</td>
<td>9.9 ± 2.0</td>
</tr>
<tr>
<td>Basal facet</td>
<td>25.6 ± 0.2</td>
<td>532 ± 23</td>
<td>7.5 ± 0.8</td>
</tr>
</tbody>
</table>

The observed anisotropy in values of $K_c$ is rationalized in terms of the different plastic deformation capability ascribed to each crystallographic plane. Figure 3.2.1.2 show a detail of the slip lines generated in a prismatic and basal plane.

Figure 3.2.1.2. SEM images of cube-corner indentations on different WC crystal planes in hardmetal: (a) prismatic facet (0.25 N), (b) basal facet (50 mN) [13].
2.4. Micro-pillar splitting method

The micro-pillar splitting method for micro scale fracture toughness determination was developed by Sebastiani et al. [2] as a technique for the determination of fracture toughness. The method is based on the use of sharp indenter to indent micro-pillars, micromachined by focused ion beam milling with an aspect ratio (height/diameter) larger than 1. An example of a pillar before and after the splitting and the curve obtained is showed in Figure 3.2.1.1.

Figure 3.2.1.1. (a) Example of a CrAlN/Si3N4 pillar before splitting. (b) Example of a CrN pillar after splitting. (c) Load-displacement curves for CrN pillars, highlighting the critical load (Pc) corresponding to the crack ‘popping out’ to the side surface, and pillar splitting [14].

Under these testing conditions, $K_C$ can be estimated on the basis of the critical splitting load $P_C$, the pillar radius $R$, and a calibration coefficient $\gamma$ with the equation (5). The load at which a displacement burst is called pop-in and is detected in the load-displacement curve (Figure 3.2.1.1).

$$K_C = \gamma \frac{P_C}{R^{3/2}}$$

(5)

A finite element analysis was made also by Sebastiani et al. [3] in order to provide a way to measure fracture toughness for different materials. They found a relationship between the ratio young's modulus/hardness and the calibration coefficient $\gamma$, see Figure 3.2.1.2.
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Figure 3.2.1.2. Coefficient $\gamma$ as a function of $E/H$ for different indenter angles [3].

Also, studies about the relation of the calculated $K_c$ as a function of the position of the indenter offset from the centre of the pillar have been done [15]. As it can be seen in Figure 3.2.1.3, a lower value for fracture toughness is determined when the indenter is at a higher distance from the indenter.

Figure 3.2.1.3. Apparent fracture toughness as a function of distance to the center of the pillar with the offset orientation direction and the indenter tip radius noted [15].
2.5. Micro-cantilever bending method

The first indentation experiments using micro-cantilever geometry were reported for coatings in 2005 [4]. This approach was then applied to various geometries, such as double cantilevers and clamped beams. The method has been used to calculate fracture toughness as well as strength and Young’s modulus. In case of strength and Young’s modulus determination, specimens are unnotched and equations of elastic regime are used. This type of test has been made to cantilevers milled in grain boundaries [16], in ceramic phase with binder zones [17] and also in grains of the ceramic phase [18].

In case of fracture toughness determination, cantilevers are milled with a notch since the fracture toughness concept is based on a body containing a crack of known length. This test has been mostly made for hard coatings and thin films since determining the fracture toughness of coatings is complicated due to their low thickness.

The bending test is performed using a nanoindenter. During the test the applied load as a function of the displacement of the indenter is recorded. The fracture is produced at the load at which the slope changes, see Figure 3.2.1.

![Figure 3.2.1. Curve load-displacement of the cantilever bending test in [4].](image)

The fracture toughness can be determined using the Equation (6) [4], where $\sigma$ is the fracture stress, $a$ is the crack length and $F(a/b)$ is a dimensionless shape factor. The shape factor is a polynomial that depends on $a/b$ and it is estimated by calculating the relation between applied load and specimen/crack geometry via simulations.
There are two different geometries of cantilevers depending on the shape of cross section, see Figure 3.2.1. The cross section will depend on the inclination of the stage inside the SEM stage chamber. If the stage is rotated to -54º, the rectangular section is possible, and if it is rotated to -9º the house shape section will be obtained. That is because the ion beam is inclined 54º and -10º is the stage limit. To obtain the rectangular section, an special holder is required and it must be done in a corner of the sample.

Figure 3.2.1. Schema of cantilevers with a) rectangular[19] and b) house shape cross section [4]
Fracture at small scale of WC grains of hardmetals
3. Experimental procedure

3.1. Materials

Two samples of WC-Co hardmetal with different grain sizes provided by Sandvik company were used. One of the specimens with similar grain sizes and the other, resinterized, with grain sized up to 30 μm (Figure 3.2.1.1). Characteristics of the samples used are presented in Table 3.2.1.1.

Table 3.2.1.1.

Grain size and composition of the samples used.

<table>
<thead>
<tr>
<th></th>
<th>WC</th>
<th>Resinterized</th>
</tr>
</thead>
<tbody>
<tr>
<td>WC (in wt %)</td>
<td>94.0</td>
<td>88.7</td>
</tr>
<tr>
<td>Co (in wt %)</td>
<td>6.0</td>
<td>11.3</td>
</tr>
<tr>
<td>Mean grain size (μm)</td>
<td>5</td>
<td>up to 30</td>
</tr>
</tbody>
</table>

Different grain sizes have been chosen for the specimen preparation depending on the specimen dimensions, regardless of the sample.
3.2. Micro-structural analysis

3.2.1. Field emission Scanning Electron Microscopy (FE-SEM)

Field emission scanning electron microscope (FE-SEM) is a SEM based technique which uses a focused beam of high-energy electrons to generate a variety of signals at the surface of solid samples. SEM employs a beam of highly energetic electrons and the concept field appears by the generation of an electric field, because the surface of the sample is conductor. For this process, the equipment requires an extreme vacuum in the column of the microscope (around $10^{-6}$ Pa). SEM involves an electron emission cathode and anodes. The acceleration voltage between these electrodes is in the range of 0.5 to 30kV [20]. An scheme of the FE-SEM parts is showed in Figure 3.2.1.1.

![Figure 3.2.1.1. Scanning electron microscope scheme][21]

In the sample chamber, the stage can be moved in axis x, y, z, rotated in z-axis $360^\circ$ and inclined from $-10^\circ$ to $90^\circ$ (Tilt angle). The distance of the detectors and columns to the sample must be checked in the camera chamber in every movement in order to prevent them from touching.

The image is formed in the SEM by a primary electron beam that scans the surface. Different types of electrons are emitted from samples depending on the penetration depth, that will depend on the accelerating voltage and sample density, see Figure 3.2.1.2. To distinguish secondary electrons, backscattered electrons or X-rays, different detectors are needed.
The secondary electron detector (SED) is used to produce a topographic SEM image. SED images have high resolution that are independent of the material and is acquired from inelastically scattered electrons close to the surface.

The backscattered electron detector (BSD) detects elastically scattered electrons. These electrons are higher in energy from atoms below the sample surface. Using BSD allows for lower vacuum levels, reducing sample preparation requirements and minimizing beam damage.

X-rays are emitted when the electron beam displaces an inner shell electron that is replaced by an outer shell electron. Because each element has a unique energy difference between outer and inner electron shells, the x-rays that are detected yield an elemental identification. X-rays are detected with the Electron Dispersive X-ray (EDX).

Figure 3.2.1.2. Schema of electrons emitted from the sample depending on the penetration depth [21].

The FE-SEM was used to identify the location of interest for milling afterwards the pillars and cantilevers milling, to measure the final dimensions and to characterise the fracture before the splitting, in case of pillars, and bending, in case of cantilevers. The FE-SEM sample stage movement allows analysing the specimens through different angles.

The model used during this project is shown in Figure 3.2.1.3. The equipmen is a Carl Zeiss Neon40 Crossbeam and equipped with a field emission electron column, gallium ion beam column and gas injection system for gas assisted deposition and milling. The SEM column is indicated with an arrow.
3.2.2. **Electron Back Scatter Diffraction (EBSD)**

Using an EBSD detector coupled to the FE-SEM, crystal orientation of each tested WC grains in hardmetal samples were assessed. The equipment used in this project is a JEOL JSM-7001F with an OXFORD EBSD.

In EBSD, the electron beam of the SEM interacts with a tilted polycrystalline sample and the scattered electrons form a pattern that can be detected with a fluorescent screen. The diffraction pattern is characteristic of the crystal structure and orientation in the sample region where it was generated. Hence the diffraction pattern can be used to determine the crystal orientation, discriminate between different phases, and provide information about the local crystalline order.

When the electron beam is scanned in a grid across a polycrystalline sample and the crystal orientation is measured at each point, the resulting map reveals the grain morphology, orientations and boundaries. This data was used in this project to show the preferred crystal orientation within the sample, see [Error! No se encuentra el origen de la referencia.](#).
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EBSD analysis of grain orientations for pillars (WC2, WC3, WCR2 and WCR3) were made of regions of 100 x 100 μm. For cantilevers milling, a larger region was needed, so a region with dimensions of 600 x 400 μm was analysed. The Figure 3.2.2.2 regions where analysis were made are showed.

Figure 3.2.2.1. EBSD analysis made with Chanel 5.

Figure 3.2.2.2. SEM images of a) normal WC and b) and c) resinterized WC. The localization of EBSDs (WC2, WC3, WCR2 and WCR3) where pillars were made is indicated with an arrow.
3.3. Milling technique

3.3.1. Focused ion beam (FIB)

Focused ion beam (FIB) guns use a gallium Liquid Metal Ion Source (LMIS) instead of a beam of electrons to image the sample. In a Gallium LMIS, gallium metal is placed in contact with a tungsten needle and heated. A large electric field causes ionization and field emission of the gallium atoms. These ions are then accelerated and focused onto the sample by a set of electromagnetic lenses.

Unlike the electron microscope, the FIB can be destructive for the specimen. When the high-energy gallium ions strike the sample, they sputter atoms from the surface. Gallium atoms will also be implanted into the top few nanometres of the surface thus creating an amorphous layer.

The equipment used in this project is the same as in the FE-SEM. In Figure 3.3.1.1 the FIB column is indicated with an arrow.

![Equipment used in this bachelor’s project for the milling, with the FIB column indicated with an arrow.](image)

**Figure 3.3.1.1.** Equipment used in this bachelor’s project for the milling, with the FIB column indicated with an arrow.

**Current and resolution**

The current is an important parameter during the milling with the FIB because of the direct relation between current and resolution. As it is shown in Figure 3.3.1.2 in the example of milling a dot, the lower the current is, the higher resolution can be achieved. When a higher current is used, the milled zone is larger and it creates a halo around the wanted geometry. The sputtering time will depend on the current and dimensions of the geometry milled.
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Figure 3.3.1.2. Schema of the relation between current and resolution in the example of milling a dot.

**Eucentric point**

The eucentric point is a geometry point located in the eucentric height, see Figure 3.3.1.3. This the height at which the sample image does not move when the sample is tilted. It needs to be located for each specific sample exactly. Finding the eucentric point is a very important step, especially for cantilever milling.

![Eucentric Point](image)

Figure 3.3.1.3. Schema of an idea of where the eucentric point must be located.

**FIB position**

The FIB chamber makes an angle of 54° with horizontal axis and the coincidence plane between the ion beam and the electron beam is located at 4,9 mm of the SEM column. To put the sample in FIB position, the stage must be tilted 54° and raised to a work distance (WD) of 4,9 mm. In Figure 3.3.1.4., an schema of the correct position of the sample is represented.

![FIB Position](image)

Figure 3.3.1.4. Schema of the stage in FIB position.
Tilt correction

In this project, the height of cantilevers and pillars was measured by SEM with stage tilted 54°. When the stage is tilted, the dimensions that are seen in the SEM image are the projection in horizontal axis, see Figure 3.3.1.5. To correct the dimensions, a tilt correction of the opposite angle must be made. In the case where the stage is tilted 54°, the tilt correction is 36°.

![Figure 3.3.1.5](image)

Figure 3.3.1.5. Schema of the projection of the dimension in horizontal axis in the SEM image.

3.4. Milling conditions

For the milling of pillars and cantilevers, the following steps were made:

1. Finding the eucentric point. That will help to rotate the sample without losing the region of interest.
2. Rotating the stage to a tilt of 54°, to put the sample perpendicular to the ion beam.
3. Rising the sample, increasing the z axis of the stage, until it is focused at a work distance (WD) of 4,9mm.

After these steps, the grain of interest is identified in the EBSD graphic and the milling of a pillar or a cantilever starts.

All dimensions were measured with sample inclined to54°, Tilt Correction activated and using a tilt angle of 36°.
Micro-pillars

Micro-pillars with diameters of 3μm and 5μm with a ratio height to diameter (h:d) higher than 1 were milled, according to [2].

Preparation of pillars for the splitting test was performed by a FIB procedure based on the ring-core milling approach. For this, the FeatureMill predefined script that allows making complex shapes was used. There are some important variable inputs that affect the final dimensions, such as height and diameter, and the quality of the pillar. These parameters will depend on the hardness and redeposition of the material and are the following.

- Milling current (mA): The milling current will affect on the time of sputtering and the final resolution of the pillar.
- Pixel size and probe size (nm): The probe size is a parameter given by the program that depends on the milling current applied. The pixel size must be lower than 1,5 times the probe size in order to have the adequate resolution.
- Dwell time (ms/pixel): This parameter indicates the time that the ion beam stays in one pixel. The higher the dwell time is, the higher the pillar will be.

For pillars of 5 μm the parameters found to be more suitable are shown in Error! No se encuentra el origen de la referencia. The same conditions were used for prismatic and basal grains. The parameters of pillars of 3 μm were not recorded. The time needed to mill a pillar of 5μm with a ratio h:d≤1 is around 30 minutes.

Table 3.3.1.1.

Parameters of pillar milling.

<table>
<thead>
<tr>
<th>Step</th>
<th>Milling current</th>
<th>Probe size (nm)</th>
<th>Pixel size (nm)</th>
<th>Dwell time (ms/pixel)</th>
<th>Inner radius (μm)</th>
<th>Outer radius (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>3nA</td>
<td>250</td>
<td>160</td>
<td>100</td>
<td>4</td>
<td>10</td>
</tr>
<tr>
<td>2</td>
<td>2 nA</td>
<td>150</td>
<td>66</td>
<td>100</td>
<td>3,5</td>
<td>6</td>
</tr>
<tr>
<td>3</td>
<td>500pA</td>
<td>50</td>
<td>25</td>
<td>50</td>
<td>3</td>
<td>4</td>
</tr>
</tbody>
</table>
Pillars were labeled as presented in Table 3.3.1.2. The label has three parts that correspond to, in the order of given, the diameter, the sample followed by a number and the orientation of the grain followed by a number that numerates them.

In the first part, the diameter is indicated with a 3 or a 5, if it belongs to the group of 3μm or 5μm diameter. In the second part, the sample can be WCR or WC, depending if it is the resinterized sample or not, and the number that follows indicates the EBSD region in this sample. In the third part, the orientation of the grain is indicated with a P or a B, if it corresponds to a prismatic or a basal grain.

Table 3.3.1.2. Identification code for pillars.

<table>
<thead>
<tr>
<th>Pillar</th>
<th>Sample</th>
<th>Facet</th>
<th>Diameter</th>
</tr>
</thead>
<tbody>
<tr>
<td>3-WC2-P1</td>
<td>Normal</td>
<td>Prismatic</td>
<td>3</td>
</tr>
<tr>
<td>3-WC2-P2</td>
<td>Normal</td>
<td>Prismatic</td>
<td>3</td>
</tr>
<tr>
<td>3-WC2-P3</td>
<td>Normal</td>
<td>Prismatic</td>
<td>3</td>
</tr>
<tr>
<td>3-WC2-B1</td>
<td>Normal</td>
<td>Basal</td>
<td>3</td>
</tr>
<tr>
<td>3-WC2-B2</td>
<td>Normal</td>
<td>Basal</td>
<td>3</td>
</tr>
<tr>
<td>3-WC2-B3</td>
<td>Normal</td>
<td>Basal</td>
<td>3</td>
</tr>
<tr>
<td>3-WC2-B4</td>
<td>Normal</td>
<td>Basal</td>
<td>3</td>
</tr>
<tr>
<td>5-WCR2-P1</td>
<td>Resinterized</td>
<td>Prismatic</td>
<td>5</td>
</tr>
<tr>
<td>5-WCR2-P2</td>
<td>Resinterized</td>
<td>Prismatic</td>
<td>5</td>
</tr>
<tr>
<td>5-WCR2-P3</td>
<td>Resinterized</td>
<td>Prismatic</td>
<td>5</td>
</tr>
<tr>
<td>5-WCR3-P4</td>
<td>Resinterized</td>
<td>Prismatic</td>
<td>5</td>
</tr>
<tr>
<td>5-WCR3-P5</td>
<td>Resinterized</td>
<td>Prismatic</td>
<td>5</td>
</tr>
<tr>
<td>5-WC3-P6</td>
<td>Normal</td>
<td>Prismatic</td>
<td>5</td>
</tr>
<tr>
<td>5-WC3-B1</td>
<td>Normal</td>
<td>Basal</td>
<td>5</td>
</tr>
</tbody>
</table>
**Micro-cantilevers milling conditions**

For the milling of micro-cantilevers, the following steps were followed:

1. In the first step, three trenches were made by milling three trapeziums with a current of 15nA, a width of 20μm, a height of 25μm and a depth of 15μm. The trenches draw the first profile of the cantilever.

2. In the second step, the shape of the superior view of the cantilever is machined by milling three rectangles with a current of 2nA, width of 18μm, a height of 9μm and a depth of 15μm, two parallel and vertical ones, to define the width, and the horizontal one, to define the length. To obtain the adequate width, each rectangle must be about 500-600nm of the final dimension wanted.

3. In the third and last step, the sample was tilt to -9° to create the house-shape cross section with a 45° angle, see Figure 3.3.1.1. Then, the cross section is defined by milling a rectangle with a current of 2nA in each lateral of the cantilever, following the same condition of the second step. In this step, dimensions that shows the camera must be recalculated since the sample is no longer perpendicular to the ion beam.

4. For the milling of the notch a line was milled using the mill for time mode with a current of 100 pA during 35s.

![House shape cross section for cantilevers preparation](image_url)
This process lasts about 2 hours and 30 minutes. In the Figure 3.3.1.2. images of the progress of the cantilever milling are shown.

Figure 3.3.1.2. Steps for micro cantilevers milling.
3.5. Splitting and bending tests

For the pillar splitting, a nanoindenter (Figure 3.3.1.1) (MTS Nanoindented XP) was used in XP Basic Hardness Method. Different maximum load were applied and a Load on sample-displacement plot was obtained.

![Nanoindenter set-up used during the project.](image)

**Pillar splitting test**

Pillars with a diameter of 3 μm were tested with a cube-corner tip, using maximum loads between 1 and 3 g. For pillars with a diameter of 5 μm a Berkovich tip was used and the maximum loads applied were between 7 and 8 g. The fracture toughness was calculated with the equation (5) with the γ=0.4 for a berkovich indenter using the curves in Figure 3.2.1.2 and the values of H and E in Table 2.

**Cantilever bending test**

For the cantilever bending the Nanovision interactive scan and indent method was used. The nanovision scan works in the same Nanoindenter but with a piezoelectric holder. In this technique the indenter remains in continuous contact with the surface of the material while scanning it, and creates a topography of the surface. Nanovision provides better precision in the applied load. In Figure 3.3.1.1 there is a topography of the surface of a cantilever.
For the bending test a depth of 5000 nm and a surface approach of 6000 nm were used.

Figure 3.3.1.2. Cantilever topography obtained by Nanovision interactive scan.

The fracture toughness is calculated after the bending test using the equation (6) seen in the second chapter of the project.

The fracture stress $\sigma$ is calculated by the equation (7), where $P$ is the applied load at fracture, $L$ is the distance between the crack and the point where the force is applied, $I$ is the moment of inertia of the beam cross section (8), and $y$ is the vertical distance between the upper surface and the neutral plane.

$$
\sigma = \frac{Ply}{I} \quad (7)
$$

$$
I = \frac{wb^3}{12} + \left(y - \frac{b}{2}\right)^2bw + \frac{w^4}{288} + \left[\frac{b}{6} + (b - y)\right]^2\frac{w^2}{4} \quad (8)
$$

$$
y = \frac{\frac{b^2}{2} + \frac{w^2}{4}\left(b + \frac{w}{6}\right)}{bw + \frac{w^2}{4}} \quad (9)
$$
The dimensionless shape factor, $F$, was provided by Di Maio and Roberts [4] who conducted extensive finite element simulation for a wide range of specimen geometries and provided the equation (10) for $0.3 \leq \left( \frac{a}{b} \right) \leq 0.5$.

$$F(a/b) = 1.85 - 3.38 \left( \frac{a}{b} \right) + 13.24 \left( \frac{a}{b} \right)^2 - 23.26 \left( \frac{a}{b} \right)^3 + 16.8 \left( \frac{a}{b} \right)^4 \quad (10)$$

For the fabricated specimens, the dimensions are showed in Figure 3.3.1.3, where $D$ is the length of the cantilever and $L$ is the distance between the notch and the applied load.

![Scheme of dimensions of cantilevers milled in this project.](image)
4. Results and discussion

In this section, the results of the pillar splitting and cantilever bending are exposed and discussed.

4.1. Micro-pillar splitting

In Figure 3.3.1.1 there is the result of the EBSD analysis in the samples. Grains where the pillars were made are indicated with a circle. Basal grains are the red ones and the prismatic are the blue and green ones.

![Figures 3.3.1.1](image)

*Figure 3.3.1.1. EBSD analysis made with Chanel 5, showing locations where the pillars were made.*
4.1.1. Pillars with 3μm of diameter

Figure 4.1.1 shows the results of pillars with a diameter of 3μm splitting. These pillars, except one, were tested with a cube-corner indenter and the pillar 3-WC2-P2 was tested with a Berkovich indenter.

As it is possible to see, none of the pillars indented with cube-corner split as the ones in the literature. For small maximum loads applied, pillars did not broke. Cracks around the indenter shape appeared in 3-WC2-P1 and 3-WC2-B1. In 3-WC2-B2 the indenter was too far from the centre of the pillar, so that, the crack started at the edge of the pillar and lines of deformation appeared because of the plasticity of WC and pop-ins appeared, but will not be useful for fracture toughness determination. For these tests, the graphic is just the curve of the load on sample without pop-ins.

In the case of 3-WC2-P2, the Berkovich indenter was perfectly centred and in the picture is possible to see the start of the crack for the splitting, unfortunately the load was not large enough and the pillar did not fully split. Even thought, pop-ins appeared, and the first one was used for the fracture toughness determination. In 3-WC2-P3, 3-WC2-B3 and 3-WC2-B4 pillars, a higher load was applied but only in 3-WC2-P3 the pillar was not fractured. This pillar showed a pop-in in a lower load, that could be because the applied load was at too much distance from the centre of the pillar.
Fracture at small scale of WC grains of hardmetals

b) 

\[
\begin{array}{c}
\text{Load on Sample (mN)} \\
\end{array}
\]

\[
\begin{array}{c}
\text{Displacement (nm)} \\
\end{array}
\]

27.31

\[
\begin{array}{c}
\text{Load on Sample (mN)} \\
\end{array}
\]

\[
\begin{array}{c}
\text{Displacement (nm)} \\
\end{array}
\]

c) 

d)
Figure 4.1.1.1. Results for pillar splitting of pillars: (a) 3-WC2-P1, (b) 3-WC2-P2, (c) 3-WC2-P3, (c) 3-WC2-B1, (d) 3-WC2-B2, (e) 3-WC2-B3 and (f) 3-WC2-B4.

Dimensions of pillars, applied loads for each pillar and the determined fracture toughness for the ones that have been possible to calculate are in Table 4.1.1.1.
Fracture at small scale of WC grains of hardmetals

Table 4.1.1.1.

*Dimensions, conditions applied and fracture toughness determination for indentation pillars of 3 μm diamter.*

<table>
<thead>
<tr>
<th>Pillar</th>
<th>Diameter (μm)</th>
<th>Height (μm)</th>
<th>Indenter</th>
<th>Coefficient (γ)</th>
<th>Maximum load (g)</th>
<th>Critical Splitting load (mN)</th>
<th>Kic (Mpa/√m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3-WC2-P1</td>
<td>3,2</td>
<td>4,0</td>
<td>Cube-corner</td>
<td>0,8</td>
<td>1</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>3-WC2-P2</td>
<td>3,1</td>
<td>3,3</td>
<td>Berkovich</td>
<td>0,4</td>
<td>4</td>
<td>27,3</td>
<td>6,3</td>
</tr>
<tr>
<td>3-WC2-P3</td>
<td>2,8</td>
<td>3,7</td>
<td>Cube-corner</td>
<td>0,8</td>
<td>3</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>3-WC2-B1</td>
<td>3,0</td>
<td>3,3</td>
<td>Cube-corner</td>
<td>0,8</td>
<td>1</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>3-WC2-B2</td>
<td>3,1</td>
<td>3,4</td>
<td>Cube-corner</td>
<td>0,8</td>
<td>2</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>3-WC2-B3</td>
<td>3,0</td>
<td>3,5</td>
<td>Cube-corner</td>
<td>0,8</td>
<td>3</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>3-WC2-B4</td>
<td>2,9</td>
<td>3,3</td>
<td>Cube-corner</td>
<td>0,8</td>
<td>3</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

4.1.2. Pillars with 5μm of diameter

Berkovich indenter showed better results in previous tests with a better distribution of the load, as seen in Figure 4.1.1.1.(a), so that, in this test a Berkovich indenter was used. Moreover, pillars with greater diameter were milled in order to have more precision of the indenter.

In the Figure 4.1.2.1 the results of the splitting of pillars of 5μm of diameter are presented. In this case, the pillars didn't broke as the theory but showed better results. All pillars splitted and showed pop-ins. Some of the pillars were indented too far from the centre of the pillar and show pop-ins at lower loads, and because of that, will show lower values of fracture toughness.
Fracture at small scale of WC grains of hardmetals

Figure 4.1.2.1. Results for pillar splitting of pillars: (a) S-WCR2-P1, (b) S-WCR2-P2, (c) S-WCR2-P3, (d) S-WCR3-P4, (e) S-WCR3-P5 and (f) S-WC3-B1, (g) S-WC2-B2 and (h) S-WCR3-B3.
Table 4.1.2.1.

Dimensions, conditions applied and fracture toughness determination for indentation pillars of 3 μm diameter.

<table>
<thead>
<tr>
<th>Pillar</th>
<th>Diameter</th>
<th>Height</th>
<th>Indenter</th>
<th>Coefficient</th>
<th>Maximum load (g)</th>
<th>Critical Splitting load (mN)</th>
<th>Kic (Mpa/√m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5-WCR2-P1</td>
<td>4,3</td>
<td>4,5</td>
<td>Berkovich</td>
<td>0,4</td>
<td>7</td>
<td>33,9</td>
<td>4,23</td>
</tr>
<tr>
<td>5-WCR2-P2</td>
<td>4,4</td>
<td>4,3</td>
<td>Berkovich</td>
<td>0,4</td>
<td>7</td>
<td>42,1</td>
<td>5,19</td>
</tr>
<tr>
<td>5-WCR2-P3</td>
<td>5,3</td>
<td>6,0</td>
<td>Berkovich</td>
<td>0,4</td>
<td>7</td>
<td>53,2</td>
<td>4,94</td>
</tr>
<tr>
<td>5-WCR3-P4</td>
<td>5,4</td>
<td>6,4</td>
<td>Berkovich</td>
<td>0,4</td>
<td>7</td>
<td>37,7</td>
<td>3,41</td>
</tr>
<tr>
<td>5-WCR3-P5</td>
<td>5,7</td>
<td>6,6</td>
<td>Berkovich</td>
<td>0,4</td>
<td>7</td>
<td>10,1</td>
<td>0,85</td>
</tr>
<tr>
<td>5-WC3-B1</td>
<td>5,5</td>
<td>5,8</td>
<td>Berkovich</td>
<td>0,4</td>
<td>8,5</td>
<td>36,7</td>
<td>3,27</td>
</tr>
<tr>
<td>5-WC2-B2</td>
<td>5,0</td>
<td>6,1</td>
<td>Berkovich</td>
<td>0,4</td>
<td>8</td>
<td>14,7</td>
<td>1,47</td>
</tr>
<tr>
<td>5-WCR3-B3</td>
<td>5,1</td>
<td>5,5</td>
<td>Berkovich</td>
<td>0,4</td>
<td>8</td>
<td>29,3</td>
<td>2,90</td>
</tr>
</tbody>
</table>
4.2. Micro-cantilever bending

In Figure 4.1.2.1 there is the result of the EBSD analysis in the samples. Grains where the pillars were made are indicated with a circle.

Figure 4.1.2.1. EBSD analysis made with Chanel 5, showing locations where the pillars were made.

In Figure 4.1.2.2 the results of the cantilever and notch milling are presented. The milling of the notch and its size is a determining factor that will change the determined fracture toughness.
Figure 4.1.2. Images of the cantilevers milled and each notch: (a) P1, (b) P2, (c) P3, (d) B1 and (e) B2.
The cantilever bending test was done as described in the methodology chapter and with this condition the load-displacement curves of Figure 4.1.2.3 were obtained. In the cantilever P3 the notch was milled in a higher distance from the junction part and another crack was created between the junction and the notch during the bending test. In the cantilever B2 the notch was not correctly milled and the propagation of the crack was not parallel. Also, in the cantilever B1 the indenter was not correctly. The results of the fracture toughness

Other cantilevers present a crack propagation with lines parallel to horizontal plane and notch.
Figure 4.1.2.3. Images of the broken prismatic cantilevers and the results of the bending tests. (a) P1, (b) P2, (c) P3, (d) B1 and (e) B2.

In Table 4.1.2.1 the determined values for fracture toughness are presented. Because of the evaluation of the visual fracture and crack propagation explained before, the results of the cantilevers P3, B2 and B1 are not entirely correct. The anisotropy of WC grains is not captured by cantilevers bending.
Table 4.1.2.1. Dimensions of cantilevers tested and values for fracture toughness.

<table>
<thead>
<tr>
<th>Cantilever</th>
<th>D (μm)</th>
<th>b (μm)</th>
<th>w (μm)</th>
<th>a (nm)</th>
<th>l</th>
<th>y</th>
<th>P</th>
<th>σ</th>
<th>a/b</th>
<th>F(a/b)</th>
<th>Kic (Mpa/√m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>P1</td>
<td>15,1</td>
<td>4,0</td>
<td>5,8</td>
<td>1,5</td>
<td>82,4</td>
<td>3,0</td>
<td>3,4</td>
<td>1,6</td>
<td>0,4</td>
<td>1,5</td>
<td>5,5</td>
</tr>
<tr>
<td>P2</td>
<td>15,5</td>
<td>3,9</td>
<td>5,9</td>
<td>1,1</td>
<td>81,2</td>
<td>3,0</td>
<td>4,3</td>
<td>2,1</td>
<td>0,3</td>
<td>1,5</td>
<td>5,9</td>
</tr>
<tr>
<td>P3</td>
<td>15,5</td>
<td>3,9</td>
<td>5,9</td>
<td>0,8</td>
<td>80,2</td>
<td>3,0</td>
<td>4,6</td>
<td>2,3</td>
<td>0,2</td>
<td>1,5</td>
<td>5,5</td>
</tr>
<tr>
<td>B1</td>
<td>16,15</td>
<td>3,9</td>
<td>5,8</td>
<td>1,0</td>
<td>80,4</td>
<td>3,0</td>
<td>3,8</td>
<td>1,9</td>
<td>0,3</td>
<td>1,5</td>
<td>5,0</td>
</tr>
<tr>
<td>B2</td>
<td>15,45</td>
<td>4,7</td>
<td>6,1</td>
<td>1,0</td>
<td>128</td>
<td>3,4</td>
<td>6,7</td>
<td>2,3</td>
<td>0,2</td>
<td>1,5</td>
<td>6,4</td>
</tr>
</tbody>
</table>

In Figure 4.1.2.4 values of fracture toughness are compared for different techniques. The anisotropy of WC grains is graphically showed. The values found in this project are lower than the ones obtained by cube-corner.
Figure 4.1.2.4. Fracture toughness values obtained in this bachelor project compared between different techniques.
5. Environmental impact analysis

In this bachelor's project no samples have been processed or prepared. All samples were given by Sandvik. The environmental impact of this project is only related to the electrical energy that was consumed using the machines and computer.
6. Conclusions

The results of this project draw the following conclusions:

- For the preparation of pillar samples a 3 step technique of 3nA, 2nA and 500pA was found to be the most accurate to mill pillars with ratio height/diameter higher than 1, with a duration of less than 30 minutes per sample. And for the preparation of cantilevers the right milling currents (15nA and 2nA) were found to mill cantilevers in less than 3 hours, but a correct way to mill perfect notches was not found.

- The values found were not entirely correct because of the lower precision of the nanoindenter. The nanovision scan technique gives better results because of the better precision. Even thought, more reliable results would be found doing the tests in-situ.

- The values for fracture toughness of WC grains obtained by pillar splitting and bending cantilever techniques are lower than the values for cube-corner fracture toughness evaluation technique. Also, the values determined by cantilever bending are higher than by pillar splitting. Even though, the anisotropy of WC grains is confirmed.
7. **Budget and financial analysis**

The cost of the project is calculated in different parts: equipment, engineering support and engineering by the designer. Following tables show in detail the concept, hours and cost. Finally, in the last table, the total cost is presented.

**Table 4.1.2.1.** Summary of the equipment employed in this project.

<table>
<thead>
<tr>
<th>Equipment</th>
<th>Quantity (h)</th>
<th>Cost/ud.</th>
<th>Cost</th>
</tr>
</thead>
<tbody>
<tr>
<td>SEM-FIB</td>
<td>176 h</td>
<td>106,7 €/h</td>
<td>18779,20 €</td>
</tr>
<tr>
<td>Nanoindenter</td>
<td>41,5 h</td>
<td>60 €/h</td>
<td>2490 €</td>
</tr>
<tr>
<td>EBSD</td>
<td>2 h</td>
<td>18 €/h</td>
<td>36 €</td>
</tr>
<tr>
<td><strong>SUBTOTAL:</strong></td>
<td></td>
<td></td>
<td><strong>21305,2 €</strong></td>
</tr>
</tbody>
</table>

**Table 4.1.2.2.** Summary of the cost of the support engineering in this project.

<table>
<thead>
<tr>
<th>Concept</th>
<th>Support</th>
<th>Quantity (h)</th>
<th>Cost/ud.</th>
<th>Cost</th>
</tr>
</thead>
<tbody>
<tr>
<td>SEM-FIB training</td>
<td>Support technician</td>
<td>12 h</td>
<td>53,52 €/h</td>
<td>642,24 €</td>
</tr>
<tr>
<td>Nanoindenter training</td>
<td>Supervisor</td>
<td>14 h</td>
<td>50 €/h</td>
<td>700 €</td>
</tr>
<tr>
<td>Project supervising</td>
<td>Supervisor</td>
<td>65 h</td>
<td>50 €/h</td>
<td>3250 €</td>
</tr>
<tr>
<td><strong>SUBTOTAL:</strong></td>
<td></td>
<td></td>
<td></td>
<td><strong>4592,24 €</strong></td>
</tr>
</tbody>
</table>
Table 4.1.2.3. Summary of the cost of the engineering by the designer.

<table>
<thead>
<tr>
<th>Concept</th>
<th>Quantity (h)</th>
<th>Cost/ud.</th>
<th>Cost</th>
</tr>
</thead>
<tbody>
<tr>
<td>Search of information</td>
<td>100 h</td>
<td>24,8 €/h</td>
<td>2480 €</td>
</tr>
<tr>
<td>Training</td>
<td>26 h</td>
<td>24,8 €/h</td>
<td>644,8 €</td>
</tr>
<tr>
<td>FIB milling</td>
<td>162 h</td>
<td>24,8 €/h</td>
<td>4017,6 €</td>
</tr>
<tr>
<td>Nanoindententer tests</td>
<td>29,5 h</td>
<td>24,8 €/h</td>
<td>731,6 €</td>
</tr>
<tr>
<td>Results analysis</td>
<td>50 h</td>
<td>24,8 €/h</td>
<td>1240 €</td>
</tr>
<tr>
<td>Memory development</td>
<td>140 h</td>
<td>24,8 €/h</td>
<td>3472 €</td>
</tr>
<tr>
<td><strong>SUBTOTAL:</strong></td>
<td></td>
<td></td>
<td><strong>12586 €</strong></td>
</tr>
</tbody>
</table>

Table 4.1.2.4. Summary table of the total cost of the project.

<table>
<thead>
<tr>
<th>Concept</th>
<th>Cost</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total cost of equipment used</td>
<td>21305,2 €</td>
</tr>
<tr>
<td>Total cost of support engineering</td>
<td>4592,24 €</td>
</tr>
<tr>
<td>Total cost of the engineering</td>
<td>12586 €</td>
</tr>
<tr>
<td><strong>TOTAL:</strong></td>
<td>38483,44 €</td>
</tr>
<tr>
<td>IVA (21%):</td>
<td>8082,52 €</td>
</tr>
<tr>
<td><strong>TOTAL + IVA</strong></td>
<td>46564,96 €</td>
</tr>
</tbody>
</table>
8. Bibliography


Fracture at small scale of WC grains of hardmetals