INTRODUCTION

Cemented carbides, also called hardmetals, are a group of powder metallurgy (PM) liquid-phase-sintered materials consisting of brittle refractory carbides of the transition metals (e.g., WC, TiC, TaC) embedded in a metallic matrix. The success of this ceramic–metal composite resides in the combination of the hardness and wear resistance of the ceramic particles with the toughness of the metallic phase. It makes cemented carbides the materials of choice in several engineering and tooling applications, such as metal cutting, mining, rock drilling, metal forming, structural components, and wear-resistant parts.

The exceptional toughness level exhibited by cemented carbides is related to highly effective toughening by constrained deformation of the ductile phase. Ductile-phase (cobalt ligaments) reinforcement of a brittle matrix (tungsten carbide network) is a prime example of toughening mechanisms that act in the crack wake to screen the crack tip from the far-field driving force. From a fracture viewpoint, such toughening has proven to be a successful microstructural design strategy in brittle-like materials because it implies the existence of a crack-growth-resistance-curve (R-curve) behavior that imparts damage tolerance, and thus improved in-service reliability to the corresponding structural components. Thus, the first step of crack propagation in cemented carbides is the formation of a crack from a microstructural heterogeneity that subsequently advances continuously in the brittle phase and circumvents the ductile regions leaving bridging ligaments across the crack faces.

The fracture and fatigue phenomena in WC-cobalt cemented carbides (hardmetals) have been subjects extensively investigated in the last 30 years. From these studies, it is well established that the metallic binder phase plays a key role as the toughening and fatigue-susceptible agent in these materials, as its effective ductility is critical for defining crack-growth resistance and cyclic-induced degradation. However, experimental proof of the role of toughening and fatigue micromechanisms has usually been presented on the basis of post-failure fractographic examination. In this work, the fracture and fatigue behavior of WC-cobalt is investigated and a 3D characterization of crack–microstructure interaction during stable crack growth in hardmetals is carried out in order to gain a better understanding of the failure processes in cemented carbides under monotonic and cyclic loads. In doing so, focused ion beam/field emission scanning electron microscopy (FIB/FESEM), 3D tomography, and imaging reconstruction are combined with systematic mechanical and indentation protocols for assessing crack-extension behavior of cemented carbides. Experimental findings clearly highlight existing differences regarding failure mechanisms operative under monotonic and cyclic loads, and provide new and interesting insights for understanding them.

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a multiligament zone (~2–4 ligaments) is developed in the direction of crack propagation. As the crack propagates, the binder ligaments elongate and microcavities are formed inside the ligament to maintain volume constancy. Using finite element calculations it was demonstrated that binder sites exposed to a high local plastic strain and stress triaxiality are preferred zones for microvoid nucleation. First, a void will be followed by others along the plane linking the adjacent cracks in the carbide phase and finally the ligament fractures by void growth and coalescence. Fracture along the carbide–binder interface proceeds in a similar manner to that in the binder, i.e., by the nucleation, growth, and coalescence of microcavities. However, shallow and closely spaced microvoids are evidenced when the crack runs parallel and close to the binder–carbide interface. Fischmeister et al. attributed the formation of such a fine dimple structure to the fulfillment of high-stress triaxiality conditions in the binder zone close to the carbide interface.

Cemented carbides, despite the plasticity developed by the binder ligaments, exhibit brittle-like behavior. This fracture mode, such as in other brittle materials, is governed by unstable propagation of flaws that may be inherent to processing, or induced in the forming process or under service conditions. Thus, strength and reliability of hardmetals are dependent on the size, geometry, and distribution of existing flaws. Following this idea, linear elastic fracture mechanics (LEFM) is recognized as an acceptable theoretical basis to describe and rationalize the fracture behavior of cemented carbides. On the other hand, a statistical tool is required to rationalize the rupture strength of this brittle material, as it is dependent on the size and geometry of the flaws. To this end, Weibull statistics are commonly employed to evaluate not only rupture strength, but also the reliability of the material.

Meanwhile, as is the case for other brittle-like composite systems (e.g., ceramic and intermetallic-base materials) where crack-tip shielding mechanisms prevail, the susceptibility of hardmetals to mechanical degradation under cyclic loading is well established. Schleinkofer et al. documented a strong strength degradation of cemented carbides under cyclic loads that predominantly occur in the ductile binder phase. They established that subcritical crack growth is the controlling stage for fatigue failure in cemented carbides, not crack nucleation. On the other hand, taking into account this second assumption and that fracture rupture behavior of cemented carbides has been extensively rationalized within the LEFM framework, Torres et al. proposed the fatigue-crack-growth threshold as the effective toughness under cyclic loading. Experimental validation for such an approach was presented for a series of WC-cobalt hardmetal grades. Moreover, the results cited a strong influence of microstructure on the fatigue sensitivity of hardmetals, depending on the compromising role of the metallic binder, as both a toughening and fatigue-susceptible agent. These investigations also point out a similarity to the Paris-Erdogan law for fatigue-crack-growth kinetics reported for structural ceramics and intermetallics. Binder fatigue degradation, also observed in the experimental applied stress–fatigue life (S–N) data published by Sailer et al., has been rationalized on the basis of fatigue-induced accumulation of the FCC to HCP phase transformation within the cobalt binder. However, most of the experimental support validating the above-referred failure scenarios are either indirect, through clear evidences of different fractographic features associated with fracture and fatigue, or rather limited (but direct), by means of transmission electron microscopy (TEM) of slices (local and small areas) in regions around crack tips.

Over the past few years, new advanced characterization techniques have been successfully implemented for microstructure, surface modification and deformation/damage characterization in various areas of materials research. Among them, the focused ion beam (FIB) technique has shown to be extremely versatile for overcoming many of the experimental limitations/difficulties ascribed to more conventional approaches, such as scanning electron microscopy (SEM) and/or TEM, atomic force microscopy, and X-ray diffraction. Fruitful examples of its implementation for quantifying microstructure, as well as evaluating tribological and mechanical phenomena in cemented carbides, are cited by Cairney et al., Beste et al., Mingard et al., and Gant et al.

It is the purpose of this study to assess the fracture and fatigue behavior of a WC-cobalt cemented carbide and to use the FIB technique for bringing new insights into the toughening and fatigue micromechanisms operative in hardmetals when subjected to monotonic (toughness) and cyclic (fatigue) loading conditions. In doing so, the main focus will be to document crack-microstructure interactions at regions close to the tip of cracks arrested after stable growth, and to discuss results on the basis of existing knowledge on fracture and fatigue phenomena for these materials.

**EXPERIMENTAL ASPECTS**

The material studied is a medium-grained WC-cobalt cemented carbide supplied by Sandvik Hyperion. The key microstructural parameters (binder content (w/o), mean grain size (dWC), carbide contiguity (CWC), and binder mean free path (λ_binder)) are listed...
Fracture and Fatigue Behavior of Cemented Carbides: 3D Focused Ion Beam Tomography of Crack-Microstructure Interactions

TABLE I. MICROSTRUCTURAL PARAMETERS

<table>
<thead>
<tr>
<th>Binder (w/o)</th>
<th>c_{WC} (µm)</th>
<th>C_{WC}</th>
<th>\lambda_{binder} (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>11% Co</td>
<td>1.12 ±0.71</td>
<td>0.38 ±0.07</td>
<td>0.42 ±0.28</td>
</tr>
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</table>

in Table I. Mean grain size was measured following the linear intercept method by means of FESEM, using a JEOL-7001F unit. Carbide contiguity and binder mean free path were deduced from best-fit equations, attained after compilation and analysis of data published in the literature,\(^3\,^5\) on the basis of empirical relationships given by Roebuck and Almond,\(^30\) but extending them to include the influence of carbide size.\(^12\,^13\)

Mechanical characterization includes hardness (HV30), flexural strength (σ_f), fracture toughness (K_{IC}), and fatigue-crack-growth (FCG) parameters. Hardness was measured using a Vickers diamond pyramid indenter under a load of 294N. In all the other cases, testing was conducted using a four-point bending fully articulated test jig with inner and outer spans of 20 mm and 40 mm, respectively. Flexural strength tests were performed on an Instron 8511 servo-hydraulic machine at room temperature. At least 15 specimens (45 mm × 4 mm × 3 mm) were tested per grade. The mean grain size was measured following the linear intercept method by means of FESEM, using a Zeiss Neon 40. Carbide contiguity and binder free path were deduced, 2 mm apart, in the center of the prospective tensile surface of each flexure specimen by applying a load of 490N using a Vickers diamond pyramid. Care was taken to orient one set of the corresponding Palmqvist cracks of each indentation flaw parallel to the cross section of the specimen where the prospective rupture would occur. Indentation residual stresses for all the controlled flaws were relieved by subjecting the specimens to tensile cyclic bending (load ratio of 0.1 and frequency 10 Hz) in order to induce stable crack growth. Finally, fracture of the cracked specimens was induced in flexure, under either monotonic or cyclic loads. All the specimens ruptured at one of the controlled flaws, Stably grown and arrested cracks remained for the other three indentations. FIB serial sectioning was then carried out in regions close to the tips of these surviving cracks. A schematic of the process is shown in Figure 1.

In order to document crack-microstructure interaction during stable crack extension, combined FIB/FESEM (Zeiss Neon 40) was used. Before ion milling, a thin protective platinum layer was deposited on small areas of interest, corresponding to regions close to tips of arrested cracks, along surface crack paths. U-shaped trenches with one cross-sectional surface (perpendicular to the crack path and to the specimen surface) were produced by FIB (Figure 2(a)).
agreement with values estimated from a direct implementation of the basic LEFM equation relating strength, toughness, and critical flaw size (Table II), by considering defects as surface semicircular flaws. This supports the use of LEFM for rationalizing the fracture behavior of the hardmetal grade studied.

**Fatigue Behavior**

FCG rates are plotted against the range and the maximum applied stress intensity factor, \(\Delta K\) (Figure 4(a)) and \(K_{\text{max}}\) (Figure 4(b)), respectively, for the two load ratios studied. As previously reported for WC-cobalt cemented carbides,\(^{19,23}\) the hardmetal grade under consideration exhibits: (i) a large-power dependence of FCG rates on \(\Delta K\), and (ii) subcritical crack growth at \(\Delta K\) values much lower than the fracture toughness. Also, a pronounced load-ratio effect is observed in the dependence of FCG on \(\Delta K\). However, as

<table>
<thead>
<tr>
<th>HV30 (GPa)</th>
<th>Flexural Strength (MPa)</th>
<th>Weibull Modulus (MPa (\sqrt{\text{m}}))</th>
<th>(K_{\text{c}}) (MPa (\sqrt{\text{m}}))</th>
<th>Estimated (2a_{\text{cr}}) ((\mu)m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>12.8 ±0.2</td>
<td>3,101 ±102</td>
<td>36</td>
<td>13.9 ±0.3</td>
<td>31 ±2</td>
</tr>
</tbody>
</table>

**RESULTS AND DISCUSSION**

**Hardness, Strength, and Toughness**

Hardness, flexural strength, Weibull modulus, and fracture toughness of the material are listed in Table II. The dispersion in flexural strength is relatively small and, accordingly, the corresponding Weibull analysis yields high values, indicative of high reliability from a structural viewpoint (Figure 3(a)). Fractographic examination reveals that critical defects are abnormal coarse grains and carbide agglomerates (Figure 3(b)) with an equivalent diameter \((2a_{\text{cr}})\) ~20–30 \(\mu\)m. It is in agreement with values estimated from a direct implementation of the basic LEFM equation relating strength, toughness, and critical flaw size (Table II), by considering defects as surface semicircular flaws. This supports the use of LEFM for rationalizing the fracture behavior of the hardmetal grade studied.

**Fatigue Behavior**

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observed for other brittle materials, R effects are largely reduced when plotting FCG against $K_{\text{max}}$. This is an indication of the predominance of static failure over cyclic failure modes.\textsuperscript{16,23}

The FCG–fatigue life relationship proposed and validated by Torres et al. for WC-cobalt cemented carbides\textsuperscript{19} has been extended for the hardmetal studied. In doing so, a classical approach on the basis of fatigue limit, within an infinite-life framework, and FCG threshold ($K_{\text{th}}$) is implemented by defining the latter as the effective toughness under fatigue for a given critical flaw size. Thus, the fatigue limit ($\sigma_f$) is deduced from the stress-intensity factor threshold of a small non-propagating crack emanating from a defect of critical size ($2a_{\text{cr}}$), according to a relationship of the form:

$$\sigma_f = Y \frac{K_{\text{th}}}{\sqrt{a_{\text{cr}}}} \quad (1)$$

Hence, the fundamental LEFM correlation between strength, stress-intensity factor, and defect size also applies to natural defects in cemented carbides. This assertion may be made considering that: (i) the size of the critical flaws is larger than both $d_{\text{WC}}$ and $\sqrt{\text{binder}}$; (ii) plasticity is confined to process zone ahead of the crack tip; and (iii) process zones governing fracture (multiligament zones behind the crack tip) extends over a relatively short distance (~five ligaments).\textsuperscript{19,34} Thus, fatigue-limit values can be estimated from the relation given by equation (2), under the assumption that flaws controlling strength have the same size, geometry, and distribution under monotonic and cyclic loading.

$$\sigma_f = \sigma_{\text{f, th}} \frac{K_{\text{f, th}}}{K_{\text{IC}}} \quad (2)$$

In an attempt to validate the estimated fatigue limit, an experimental study was conducted using 15 samples, following an up-and-down (staircase) loading regime for fatigue testing (Figure 5(a)), and defining “infinite fatigue life” at $10^6$ cycles. Predicted and experimentally determined fatigue limits are listed in Table III. The FCG threshold–fatigue limit correlation is validated by the excellent agreement attained between them. Furthermore, the fractographic examination conducted on failed specimens reveals that size, geom-
etry, and the nature of the initial critical defects are similar under both monotonic- and cyclic-loading conditions (Figure 5(b)). However, under the application of cyclic loads, the defects grow until they reach a critical size and fracture is unstable.

**Crack–Microstructure Interactions**

After failure, the fracture surfaces were examined using FESEM. Clear differences are evidenced when comparing fractographic features corresponding to stable crack growth (Figure 6(a)) and unstable crack growth (Figure 6(b)). While in the former, “step-like” fatigue damage features are discerned within the metal binder, in the latter well-defined dimples are evident, suggesting a ductile fracture mechanism. Fracture under cyclic loading in the cobalt binder appears to follow a faceted, crystallographic fracture mode, as can be appreciated by the sharp angular facets localized within broken binder regions. With the purpose of documenting and understanding the crack-growth processes under monotonic and cyclic loads, a tomographic reconstruction of the process zone at the crack tip has been performed by means of the FIB/FESEM technique.

**Stable Crack Growth Under Monotonic Loading**

Figure 7 shows crack-microstructure details along the path of an arrested crack, after stable growth under monotonic loading. From these preliminary findings, many of the conclusions drawn by Sigl, Exner and Fischmeister are validated, namely, (1) continu-

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**TABLE III: FATIGUE-Crack-Growth Threshold and Predicted and Experimentally Determined Fatigue-Limit Values in Terms of Maximum Applied Stress**

<table>
<thead>
<tr>
<th>FCG Threshold (MPa m(^{1/2}))</th>
<th>Predicted Fatigue Limit (MPa)</th>
<th>Experimental Fatigue Limit (MPa)</th>
</tr>
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<tbody>
<tr>
<td>7.6 ±0.2</td>
<td>1,696 ±80</td>
<td>1,632 ±130</td>
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**Figure 5.** (a) Up-and-down (staircase) fatigue test to determine the mean fatigue limit for the WC-nickel cemented carbide, and (b) example of critical defect growth under cyclic loading

**Figure 6.** FESEM images corresponding to (a) stable crack growth under cyclic loads (R = 0.1) and (b) unstable crack growth under monotonic loading for WC-cobalt hardmetal. Fatigue facets in (a) and ductile dimples in (b) are evident in the metallic phase
ous cracks enclose cobalt regions (ligaments) that elongate during crack opening by void formation with localized plastic deformation at the bridges between the voids, (2) ligaments finally fail by void coalescence, (3) the mean depth of the plastic zone is always smaller than the mean-free path in the binder, and (4) partition of crack paths between binder and carbide does not correspond to the volume fraction but significantly favors the ductile binder. Additionally, local blunting effects as crack fronts end within the binder (as invoked by McVeigh and Liu,35 although at a higher-length scale) and the role of carbide corners as stress risers are clearly discerned.

In conclusion, the multiligament zone may be unambiguously established as the primary foundation for rationalizing R-curve behavior and exceptional fracture toughness of cemented carbides. Within this framework, a description and understanding of microstructural effects on fracture toughness, R-curve characteristics, strength variability, and damage tolerance for hardmetals are also validated. In Figure 8 the 3D reconstruction of the microstructure containing the crack propagated under monotonic loading is shown. The light blue corresponds to the WC carbides, the dark blue corresponds to the cobalt binder, and the red area corresponds to the crack path. An examination of the reconstruction permits a clear visualization and understanding of the fracture process (microv air nucleation and growth). A clear example of the process is presented in Figure 9.
Unstable Crack Growth Under Cyclic Loading

Crack–microstructure interaction during stable crack growth under cyclic loading is shown in Figure 10. Different from the failure scenario discerned under monotonic loading, fatigue micromechanisms in cemented carbides are less defined and understood. Taking this into consideration, images attained through FIB “slice and view” shed further light for proposing and/or validating specific failure micromechanisms: (1) subcritical fatigue-crack growth is more predominantly located in the ductile binder phase than under monotonic loading;9 (2) fatigue-crack extension follows crystallographic-like paths (steps); and (3) crack-arrest phenomena within binder ligaments appear to be normal, particularly in regions far from microstructure-related stress risers. Although the crystallographic nature of the steps is evident, their intrinsic origin as related to shear bands, stacking faults and/or twins resulting from FCC-to-HCP phase transformation is not clear. The fact that step-like markings on fatigue-fracture surfaces have been observed not only in cobalt-base hardmetals but also in nickel- and cobalt/nickel-base grades,36,37 makes clear that fatigue susceptibility of cemented carbides goes beyond changes in deformation mode induced by the FCC-to-HCP transformation, well established in WC-cobalt grades.38 As in monotonic loading, the 3D reconstructed images of the whole volume and the individual phases is presented in Figure 11. It is interesting to note that under cyclic loading the crack prop-

Figure 10. Stable crack growth under cyclic loading: FESEM micrographs showing crack–microstructure interactions. A crystallographic-like fracture path within the binder is evidenced in these images, corresponding to serial sections obtained by FIB/FESEM tomography

Figure 11. 3D reconstruction of process zone for stable crack growth under monotonic loading. Light and dark blue correspond to the WC skeleton and the cobalt phase, respectively; red represents the cracked region. A volume of 10 μm (X axis) x 10 μm (Y axis) x 8 μm (Z axis) was reconstructed

Figure 12. Crack propagation within a binder pool under the application of cyclic loads
agates through the ductile metallic phase and bridging ligaments are not formed. Figure 12 shows the fatigue failure process in a binder pool.

CONCLUSIONS

The fracture and fatigue behavior of a WC-cobalt cemented carbide has been investigated. A detailed study of crack–microstructure interactions during stable crack growth under monotonic and cyclic loads has also been conducted by the complementary use of mechanical and indentation testing protocols and FIB/FESEM tomography. From the results the following conclusions may be drawn:

1. The fracture behavior of the WC-cobalt cemented carbide has been successfully rationalized using LEFM, on the basis of a reliable assessment of fracture toughness as well as the size and geometry of critical flaws.

2. As previously reported for cemented carbides, the WC-cobalt grade studied exhibited a large-power dependence of FCG rates on ∆K, subcritical crack growth at ∆K values lower than the fracture toughness, and pronounced load-ratio effects in FCG–∆K curves. R effects are largely reduced when plotting FCG against K\text{max}. This indicates the predominance of static over dynamic failure modes.

3. Fatigue behavior (“infinite fatigue life”) can be rationalized by a fatigue mechanics approach considering the initiation of subcritical crack growth as the control parameter in fatigue failure (K\text{th} is the effective toughness under cyclic loads).

4. The key role played by the metallic binder phase as the toughening and fatigue susceptible constituent in cemented carbides is validated. Unequivocal proof of the multiligament zone as the foundation for understanding toughness and R-curve behavior in hardmetals is provided. Fatigue susceptibility of these materials is associated with inhibition of such toughening mechanism under cyclic loading. In this case, stable crack growth follows a crystallographic path through the metallic binder, distinct from the typical ductile failure mode found under monotonic loading.

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