Elsevier Editorial System(tm) for Surface and Coatings Technology
Manuscript Draft

Manuscript Number: SURFCOAT-D-15-00521R1

Title: Contact damage resistance of TiN-coated hardmetals: Beneficial effects associated with substrate grinding

Article Type: Full Length Article

Keywords: Substrate grinding; Contact damage resistance; Coated hardmetal; Surface integrity

Corresponding Author: Ms. Jing Yang,

Corresponding Author's Institution: UPC-ETSEIB

First Author: Jing Yang

Order of Authors: Jing Yang; Fernando García Marro; Trifon Trifonov; Magnus Odén; Mats Peter Johansson-Jõesaar; Luis Llanes

Abstract: Contact loading is a common service condition for coated hardmetal tools and components. Substrate grinding represents a key step within the manufacturing chain of these coated systems. Within this context, the influence of surface integrity changes caused by abrasive grinding of the hardmetal substrate, prior to coating, is evaluated with respect to contact damage resistance. Three different substrate surface finish conditions are studied: ground (G), mirror-like polished (P) and ground plus heat-treated (GTT). Tests are conducted by means of spherical indentation under increasing monotonic load and the contact damage resistance is assessed. Substrate grinding enhances resistance against both crack nucleation at the coating surface and subsequent propagation into the hardmetal substrate. Hence, crack emergence and damage evolution is effectively delayed for the coated G condition, as compared to the reference P one. The observed system response is discussed on the basis of the beneficial effects associated with compressive residual stresses remnant at the sub-surface level after grinding, ion-etching, and coating. The influence of the stress state is further corroborated by a lower resistance against damage for the coated GTT specimens. Finally, it is proposed and preliminary validated that substrate grinding also enhances damage tolerance of the coated system when exposed to contact loads.

March 13, 2015

Professor Alan Matthews , Editor-in-Chief
*Surface and Coatings Technology*
Dept. of Engineering Materials, University of Sheffield,
Sir Robert Hadfield Building, Mappin Street,
Sheffield, S1 3JD, England, UK

**RE: " Contact damage resistance of TiN-coated hardmetals: Beneficial effects associated with substrate grinding ",**
**by J. Yang, F. García Marro, T. Trifonov, M. Odén,**

**M.P. Johansson-Jõesaar and L. Llanes**

Dear Professor Matthews,

Please find attached electronic files corresponding to our contribution on contact damage resistance of TiN-coated hardmetals: beneficial effects associated with substrate grinding, which we (all authors do agree to the submission of the manuscript) offer for publication in *Surface and Coatings Technology.*

I hope it is found satisfactory.

Sincerely yours,

Jing Yang

*Response to Reviewers

Jing Yang
Ph. # + 34-934011083
email: jing.yang1@upc.edu / jinya@ifm.liu.se

Barcelona, May 18, 2015

Professor Samir Aouadi
Editor, *Surface and Coatings Technology*
Department of Materials Science and Engineering
University of North Texas
Denton, TX 76203-5017
USA
E-mail: Samir.Aouadi@unt.edu

**REF: Ms Nº: SURFCOAT-D-15-00521, *"Contact damage resistance of TiN-coated hardmetals: Beneficial effects associated with substrate grinding", by J.Yang, F. García Marro, T. Trifonov, M. Odén, M.P. Johansson-Jõesaar and L. Llanes***

Dear Professor Aouadi,

Thank you for your email of April 16 informing us about your positive consideration of the referred manuscript, upon the completion of minor revisions, for its publication in Surface and Coatings Technology. From the reports appended in your email, it seems that the four reviewers have read the paper very carefully. Please thank them for their care and criticism. As requested, we have considered all their comments, and the following is a list of our responses (and modifications):

***(R1) Reviewer #1's comments:***

***(R1)*** *This is an interesting paper on the effect of surface finishing of a particular substrate in the behaviour of PVD coatings. This is a very critical topic in the field of industrial applications of hard PVD coatings, where this kind of studies is not frequent and most of the workshop knowledge is far from being of scientific quality.*

*It is worth to be noticed that four of the six authors already published similar results in the 2nd CIRP Conference on Surface Integrity, CSI 2014; Nottingham; United Kingdom (Procedia CIRP, Volume 13, 2014, Pages 257-263).*
**(Authors) R1** is right about connection between results published in our contribution to the 2nd CIRP Conference on Surface Integrity (reference [27] in the submitted manuscript) and those presented in this paper. However, the two papers are distinctly different and cover completely different research topics and contain different data. The former focuses on "ground hardmetals" and "flexural strength/fractography", whereas the current one deals with "coated hardmetals" and "contact damage". Hence, we find R1's comment regarding "similar results" inappropriate.

*The paper is well organised, the experimental method is correct and the conclusions are sound and justified.*
*Some minor points should be clarified before publication:*
***(R1)**- A 13% of binder is higher than the average. Is there a reason for choosing this particular grade of hardmetal?*
**(Authors)** Depending on cutting/milling conditions, binder content choice for coated hardmetal tools usually vary within the range of 3-15$\%_{wt}$. In the case of interrupted cutting (e.g. milling), contact damage resistance would be expected to play a more relevant role than for continuous operation (e.g. turning); thus, relatively higher binder content levels (tougher grades) may be more appropriated. Within this context, a hardmetal grade with 13$\%_{wt}$ binder content was chosen for the study. Accordingly, one sentence has been added to indicate this issue in the revised manuscript.

***(R1)** Information about the size of the WC grains should be suitable.*
**(Authors)** Corresponding microstructural information has been provided in the revised version.

***(R1)** It is not clear why the effect of the Co binder in the residual stress can be neglected.*
**(Authors)** Co binder effects on residual stresses are not neglected, and our writing may be blamed for such misunderstanding. Residual stresses induced by grinding are "macrostresses" evaluated in the WC phase and assumed to be representative of the whole WC-Co composite. They are different from the "microstresses" (different for each individual phase) that arise due to the difference in thermal expansion between the binder and the carbide as the material cools from liquid- or solid-phase sintering. In cemented carbides with WC as the carbide, the WC is taken as a reliable reference phase because it remains stoichiometric and does not take solute in solution. On the other hand, the metal binder does take W and C into solution during sintering, so that the starting binder powder cannot be used as a stress-free reference. Furthermore, the diffraction peaks from the cobalt-base binder phase are weak and broad due to its relatively low content that yield nonreliable stress measurements. The suitability of the protocol implemented for residual stress assessment is supported by the fact that it is the one reported in relevant literature dealing with it, e.g. Refs [23-25] of manuscript. Revised version included modified text aiming to explain this issue better.

***(R2) Reviewer #2's comments:***

***(R2)** 1. The coating composition has significant effects on contact stress of hardmetal, research in this area are proposed to be discussed.*
**(Authors)** R2 is completely right about the relevance of coating composition on the contact damage response of coated hardmetals. Indeed, it has been the main topic addressed by UPC's research group in recent papers (References [40], [43] and [44]), and interested readers may get further information on this issue there. However, the variable experimentally studied in this investigation was "substrate surface finish", while the

coatings' features were kept unchanged. Accordingly, effects of coating composition is outside the scope of this paper.

***(R2)*** *2.Figure 9 is not suitable to be used to explain relevant content in the paper.*
**(Authors)** – We disagree with R2 on this issue. Although assessment of substrate grinding effects on damage tolerance of coated systems exposed to contact loads was not the main objective of the work, Figure 9 is helpful for as a starting point to understand and discern the different interactions among propagating contact-induced cracks, remnant compressive stress fields, surface texture features (nicely highlighted in Figure 9), and pre-existing grinding-induced microcracks. In this regard, it must be emphasized that we have not attempted to yield higher relevance of this issue than the one stated, recalling further research for deeper understanding. The fact that all other reviewers did not do any specific comment on Figure 9 will support our viewpoint.

***(R2)*** *3. The content discussed in the results and discussion part is very rich, but logic and systematicness are not strong, propose to adjust.*
**(Authors)** – Although R2's comment seems "simple", it is excessively generic and rather unclear. Indeed, it will be somehow opposite to R1's assessment of the manuscript as "well organised with sound and justified conclusions". We would have appreciated specific suggestions from R2, as we find current paper's structure the optimal one for presenting and discussing our experimental findings. Nevertheless, a final and detailed English grammar revision has been conducted aiming for an improved final version.

***(R2)*** *4. Effect of heat treatment on mechanical properties of hardmetal and coatings are proposed to be adequate discussed.*
**(Authors)** The effect of heat treatment on the mechanical properties of hardmetals has been described and discussed in the manuscript in several sections. For instance, within "2.1 Materials and substrate surface finish conditions: High temperature annealing …. as GTT."; or in page 9: "The coated GTT specimens display virtually no residual stress." Furthermore, there is a relevant sections in the discussion presented in sections 3.2, 3.3 and 3.4, regarding relief of grinding-induced stresses and its influence on contact response of the coated specimens.

Concerning heat treatment effects on the mechanical properties of the coating, it must be highlighted that referred annealing treatment was carried out on the ground substrate prior to coating deposition, i.e. the coatings are not heat treated.

***(R3) Reviewer #3's comments:***

***(R3)*** *Review of « Contact damage resistance of TiN-coated hardmetals : beneficial effects associated with substrate grinding » by J Yang et al. 2015*
*This paper comes after the publication of two articles published by the same author in «Procedia CIRP» in 2014 and in «Surface and coatings technology» in 2015. Some sentences, in particular in the «experimental procedure», are fully repeated. The authors should correct this.*
**(Authors)** Similar to the response given to **R1** above, **R3** is right about connection between results published in our previous contributions published in Procedia CIRP [2014] and SCT [2015] (references [27] and [21] in the submitted manuscript respectively) and those presented in this paper. However, once again, it should be clarified (and emphasized) that the three studies address different issues within the shared research framework. Focus is on "flexural strength/fractography" in the 2014's paper, on "scratch resistance" in the 2015's one, and on "contact (spherical indentation) damage" in the submitted contribution. Both 2015's studies share findings associated with surface integrity characterization of coated hardmetals, but such information must be included in the two papers. Nevertheless, we fully agree with **R3** that identical sentences in the experimental procedure must be corrected. We apologize for this mistake and appreciate the thoroughness of the reviewer. In the new version this has been corrected.

***(R3)*** *In this paper, the behaviour under spherical contact of TiN coated hard metal is studied. Three kinds of samples, having different surface finished substrate covered with the same film have been examined. The authors clearly show that ground substrate enhance the contact damage resistance, which is the main goal of the paper. They explained this effect by the presence of compressive residual stress. However, some revision are needed.*
*Before the paper being published the authors should answer the following questions:*

***(R3)*** *1. The authors should justify why only the WC phase is studied in terms of residual stresses. Why residual stresses is not expected in the Co-phase.*
**(Authors)** Such query has been addressed above (see Authors' response to final comment from **R1**).

***(R3)*** *2. The values of residual stresses are different from the paper of 2014 while the one of the roughness are the same. Why?*
**(Authors)** The residual stress values given in the 2014's paper correspond to those measured in the "just ground and uncoated" substrate. On the other hand, the values reported in the submitted contribution refer to those measured in the ground substrate, but AFTER ion-etching and coating deposition process. Hence, they are indeed different, and these differences are discussed in the text (final paragraph in **Section 3.1**).

***(R3)*** *3. What is the effect of the BIT preparation and particularly of the Bakelite coating on the integrity of the TiN coating ?*
**(Authors)** – BIT preparation technique effect is indicated within the second paragraph of **Section 3.3**: "In general, crack extension develops faster, in terms of applied load and particularly once the crack has already penetrated into the substrate, in indented BIT samples than in specimens without any artificial interface." Such "faster damage evolution" in indented BIT specimens could be rationalized on the basis of a finite element modelling / experimental investigation conducted by Helbawi and coworkers about difference in subsurface damage in indented alumina specimens with and without bonding layer [Int J Mechanical Sciences 43 (2001) 1107]. These authors found that in BIT specimens, stress distribution is more shifted to and concentrated at the surface as compared to integral samples. Accordingly, we have included this information (and new reference) in the revised version.

As for the effect of the Bakelite coating, we do not quite understand R3's comment, as our implemented protocol does not involve any Bakelite coating. We use Bakelite just to mount and fix the two separate coated specimens facing each other. It is possible that R3's comment referred to bonding glue's coating. If this were the case, corresponding effect would be included in the BIT preparation technique effects described above.

***(R3)*** *4. Generally with spherical indenter, contact pressure versus deformation (a/R) curves are plotted. Why it is not the case here? It seems that all the experimental information is available to plot it.*
**(Authors) R3** is right about the possibility of plotting contact pressure versus deformation curves. Indeed, experimental data for plotting it are given in **Figure 3**. However, we found that relative (slight) differences among studied conditions (G, P and GTT) were shown clearer by **Figure 3** than by plotting pressure-deformation curves.

***(R3)*** *5. The Ra value for the uncoated polished sample seems to be strange $0.01 \pm 0.01$. Is this correct?*
**(Authors)** Detailed Ra value for the polished samples is $0.010 \pm 0.003$ µm. However, data in **Table 1** (common for all the surface finish conditions studied) is listed with 0.01 µm resolution.

***(R3)*** *6. The authors claim that cracks were more difficult to distinguish for G samples compared to P samples due to rougher surfaces. However, the Ra value is lower for G than for P samples. So, this explanation is no valid.*
**(Authors) R3** is completely right, and we should apologize for inappropriate use of word "rougher". The main reason for crack detection being more difficult in G condition is due to

its specific groove-like surface texture inherited from grinding (and not changed by ion-etching or coating deposition). Such different surface texture aspect for G and P specimens is clearly discerned in the surface view of coated specimens shown in **Figure 9**. Cracks are then hidden by the larger surface undulations for G surface finish. Accordingly, the text in the manuscript has been modified (highlighted in the text).

***(R3)*** *7. "Qualitative similar" page 11 is not correct.*
**(Authors)** Text in the manuscript has been modified by adding "ly" to "Qualitative"

***(R3)*** *8. "There, it" at the end of page 12.*
**(Authors)** Text in the manuscript has been modified by adding "," after "There".

***(R3)*** *9. In figure 9, the author should underline the residual imprint of spherical contact.*
**(Authors)** Residual imprints of spherical contact have been underlined in Figure 9.

***(R3)*** *10. Finally, is contact fatigue a perspective of this work?*
**(Authors)** Yes, it is an ongoing work, and we hope to obtain some publishable results in the near future.

***(R4)*** **Reviewer #4's comments:**

***(R4)*** *You need to specify hardmetal grain size.*
**(Authors)** Corresponding microstructural information has been provided in the revised version.

***(R4)*** *Does the hardmetal polishing regime produce relief of the WC grains and/or chemical attack of the cobalt?*
**(Authors)** No. The polishing protocol used (described in Section 2.1) includes systematic and sequential material removal steps, such that relative differences in surface relief of both constitutive phases as well as possible localized attack of any of them are completely avoided. The surface preparation method used is based on extensive investigation conducted by UPC's research group on hardmetals (bulk specimens) for many years. Such work includes fracture and fatigue research where extreme surface integrity (mirror-like finish, no residual stresses and no subsurface damage) is mandatory for understanding microstructural influence on the mechanical response of cemented carbides.

***(R4)*** *Given that crack "pop in" does not occur as it would for a classically brittle material such as float glass, is Hertzian indentation a serious contender for KIC evaluation in this type of material? Include in Discussion.*
**(Authors)** Reviewer is right about possible consideration of Hertzian indentation as an alternative method for fracture toughness evaluation. Indeed, such issue has been addressed and discussed by the authors in Ref. [22] of the submitted manuscript. It corresponds to a paper just published by us on testing method (and microstructure) effects on the fracture toughness of cemented carbides (Int. J. Refract. Met. Hard Mater. 49 (2015) 153-160). As it is detailed there, for the hardmetal used in this investigation, spherical indentation results in overestimated fracture toughness values, as compared to those measured using either Chevron-notched three-point bending or Palmqvist indentation. Nevertheless, discrepancies may be reduced for softer/tougher grades, as far as cracks may still be induced under Hertzian testing. Additional information on the influence of testing method on the evaluation of fracture toughness has not been included in the revised version (space limitation as well as somehow "out of place" discussion), although the above article is indicated as reference for hardness and toughness values of the hardmetal substrate studied.

***(R4)*** *Is there a possibility of R-curve behaviour, even with a fine grain hardmetal, given that the growing crack is constrained less than in a standardised plane strain KIC test such as SENB, SEVNB?*

**(Authors)** We understand that an intrinsic R-curve behavior will always exist for any cemented carbide, independent of carbide grain size, on the basis of the development of a multiligament (ductile bridges) zone behind the crack tip. On the other hand, extension and shape of such R-curve is surely dependent on microstructure (longer and less steep for coarser and high-binder content microstructures) and "constraining" (plane stress versus plane strain conditions). As far as cracking phenomenon is localized at the surface (i.e. ring cracks growing into the substrate), a less-constrained scenario should be expected. However, we would expect that beneficial effects associated with grinding (main idea proposed and validated in the paper) would not be directly affected by R-curve issues. This may not be the case if microstructural assembly of substrate changes, an interesting field to explore in future research.

We hope the modified version will now be completely suitable for publication. Once again, thank you for your time and cooperation.

Sincerely yours,

Jing Yang

***Highlights (for review)**

- Substrate grinding effectively delay both crack emergence and damage evolution.
- The grinding beneficial effect is related to the compressive residual stresses.
- An additional positive effect of grinding in terms of damage tolerance is proposed.

# Contact damage resistance of TiN-coated hardmetals: Beneficial effects associated with substrate grinding

J. Yang*[1,2], F. García Marro[1,3], T. Trifonov[3], M. Odén[2], M.P. Johansson-Jõesaar[2,4] and L. Llanes[1,3]
[1] CIEFMA - Departament de Ciència dels Materials i Engenyeria Metal□lúrgica, ETSEIB, Universitat Politècnica de Catalunya, Barcelona 08028, Spain
[2] Nanostructured Materials, Department of Physics, Chemistry, and Biology (IFM), Linköping University, Linköping 58183, Sweden
[3] CRnE – Center for Research on Nanoengineering, Universitat Politècnica de Catalunya, Barcelona 08028, Spain
[4] SECO Tools AB, Fagersta 73782, Sweden
* Corresponding author: jing.yang1@upc.edu / jinya@ifm.liu.se

## Abstract

Contact loading is a common service condition for coated hardmetal tools and components. Substrate grinding represents a key step within the manufacturing chain of these coated systems. Within this context, the influence of surface integrity changes caused by abrasive grinding of the hardmetal substrate, prior to coating, is evaluated with respect to contact damage resistance. Three different substrate surface finish conditions are studied: ground (G), mirror-like polished (P) and ground plus heat-treated (GTT). Tests are conducted by means of spherical indentation under increasing monotonic load and the contact damage resistance is assessed. Substrate grinding enhances resistance against both crack nucleation at the coating surface and subsequent propagation into the hardmetal substrate. Hence, crack emergence and damage evolution is effectively delayed for the coated G condition, as compared to the reference P one. The observed system response is discussed on the basis of the beneficial effects associated with compressive residual stresses remnant at the subsurface level after grinding, ion-etching and coating. The influence of the stress state is further corroborated by the lower contact damage resistance exhibited by the coated GTT specimens. Finally, differences observed on the interaction between indentation-induced damage and failure mode under flexural testing points in the direction that substrate grinding also enhances damage tolerance of the coated system when exposed to contact loads.

**Keywords:** Substrate grinding; Contact damage resistance; Coated hardmetal; Surface integrity

## 1. Introduction

Hardmetals belong to a class of composite materials, in which hard particles, tungsten carbide (WC), are bound together by a soft and ductile metallic binder, cobalt (Co). Such a particular microstructure assembly yields an extraordinary combination of mechanical and tribological properties. As a consequence, these cemented carbides are positioned at the forefront in a wide range of applications, mainly as machining/forming tools and wear-resistant components [1]. In many of these applications the hardmetals are coated by a thin ceramic film. The main advantages of depositing such coatings are better protection against mechanical and thermal loads, superior wear resistance, and chemical stability, i.e. the surface can withstand a higher tool speed and less lubricant usage [2-5].

Manufacturing of hardmetals often involves grinding, and in the case of cutting tool inserts also edge preparation, etching and coating. The quality of the shaped components is influenced by how the surface integrity evolves through the different process steps. In this regard, substrate grinding and coating deposition represent key steps, as they are critical for defining the final performance and relative tool manufacturing cost [3,6,7].

Considering the complex service conditions to which coated hardmetal tools and components are subjected (abrasive and adhesive wear, impact, contact loading, etc.), extensive research has been conducted in order to investigate the mechanical response of these coated systems. Within this context, existing literature concentrates on the influence of either chemical nature or layer-architecture of the film on hardness, scratch resistance, friction coefficient, as well as wear and impact behavior [3,8-11]. Only a few investigations address the influence of surface topography or subsurface integrity resulting from changes induced at different manufacturing stages, particularly regarding those implemented prior to coating deposition, i.e. grinding, lapping, polishing, blasting, and peening [7,12-19]. On the other hand, a favorable effect of substrate grinding on lifetime of coated hardmetal tools (i.e. enhanced tribomechanical performance) is well established [3,7,13,17,19-21]. Therefore, knowledge and understanding of these surface finish effects become relevant for effective design of coated hardmetals in machining applications involving extreme service conditions, e.g. interrupted cutting and difficult-to-machine materials [7,12].

Following the above ideas, it is the aim of this study to investigate the contact damage behavior of a fine-grained hardmetal coated with an arc evaporated TiN film, with three different substrate surface finish conditions prior to the coating deposition: ground (G), mirror-like polished (P), and ground followed by high temperature annealing (GTT). Controlled damage is induced by means of spherical indentation under increasing monotonic load, and contact damage resistance is assessed on the basis of crack nucleation at the coating surface as well as its subsequent extension into the hardmetal substrate. Mechanical testing is complemented by residual stress evaluation using X-ray diffraction and detailed scanning electron microscopy inspection of the damage zone in a cross section view.

## 2. Experimental Procedure

### 2.1 Materials and substrate surface finish conditions

The substrate studied was a WC-Co hardmetal grade with a carbide mean grain size of 0.7 μm and binder content of 13 wt.%. Hardmetals with such high Co content are frequently used for application with intermittent loading conditions such as cutting tools for milling due to their improved toughness. The cemented carbide under consideration here has a Vickers hardness and fracture toughness of 14.8 GPa and 11.2 MPa√m respectively [22].

Three different surface finish conditions prior to coating deposition were investigated. Two of them corresponded to abrasive material removal processes: ground (G) and polished (P). G surface finish was attained by means of a commercial diamond abrasive wheel, using coolant to minimize heat generation. The mirror shine P surface was achieved by a sequence of polishing steps using diamond-containing disks, diamond suspensions (final grit size 3 μm), and finishing with a suspension of 45 nm colloidal silica particles.

It is well established that grinding of hardmetals introduces mechanical and thermal alterations at the surface and subsurface levels [23-27]. These changes include deformation, microcracking, residual stresses and possibly phase transformation of the binder phase. However, knowledge about how each alteration type contributes to the global effects from grinding remains unclear. This is of particular interest since their individual contribution may be either beneficial or detrimental. In an attempt to provide such information, a residual stress-free ground substrate was studied as the third surface finish condition. High temperature annealing of hardmetals has been validated as a successful protocol for relieving residual stresses, independent of nature (tensile/compressive) or source (mechanical abrasion [27-29] or electrical discharge machining [30-32]). Hence, ground specimens were heat treated at 920 °C for 1 h in vacuum, and the resulting surface finish condition is here referred to as GTT.

The coating deposition was conducted using an MZR323 reactive cathodic arc evaporation system. All substrates, corresponding to the three surface finish conditions, were mounted on a rotating mounting drum facing pure Ti cathodes and kept at the same height during the deposition. About 3 μm thick TiN coating was reactively grown in a $N_2$ atmosphere at a pressure of 2 Pa, using a substrate bias of -50 V, and maintaining the substrate temperature at 450 °C. Prior to deposition, the substrates were ultrasonically cleaned in an alkali solution and alcohol followed by sputter cleaning with ~500 eV Ar-ions. The base pressure of the deposition system was $2.0 \times 10^{-3}$ Pa. Hardness of the deposited coatings was about 28 GPa independent of substrate surface finish. It was obtained using a nanoindenter (MTS XP system), equipped with a calibrated Berkovich diamond tip [21]. 16 indents (4 X 4 array) were performed until they reached the maximum load limitation 650 mN of the equipment. Hardness values were calculated using the Oliver-Pharr method [33].

### 2.2 Surface integrity assessment

Surface integrity characterization for each surface pretreatment condition was conducted in terms of roughness, residual stresses, and damage discerned at the surface and subsurface levels. Surface roughness was measured by using a stylus profilometer (Surftest SV512, Mitutoyo). Arithmetic deviation from the mean line through the complete profile ($R_a$) and maximum profile depth ($R_y$) were recorded for the G and P surface finish conditions at different stages of the coating process, i.e. uncoated, ion-etched and ion-etched plus coated substrate. Roughness parameter values were averaged over five measurements per sample.

Surface residual stresses in the hardmetal substrate and TiN coating were determined by means of X ray diffractometry and employing the $\sin^2\psi$ method using a Panalytical Empyrean four-circle diffractometer using a Cu-$K_\alpha$ radiation [21,27]. The biaxial residual surface stress induced by grinding was measured in just the WC phase. However, this stress represents a good approximation of the deviatoric macrostress in the surface, i.e. the grinding stresses [14,23,25]. The diffraction peaks from the cobalt-base binder phase are weak and broad due to its relatively low content that yield nonreliable stress measurements. In this regard, it should be noted that, according to literature [34-36], residual stress values in the range from -100 to -500 MPa would be expected in the WC phase, neglecting any machining-induced strain/stress effects.

Finally, the surface integrity was inspected by means of field emission scanning electron microscopy (FESEM), using a JEOL JSM-7001F equipment. Cross-sectional samples for subsurface studies were prepared by focused ion beam (FIB) milling, using a Zeiss Neon 40 system.

### 2.3 Evaluation of contact damage resistance

In order to study contact damage phenomena in hard ceramics, it is common practice to use spherical indentation (Hertzian contact) [37,38]. Recently it has also been applied to introduce controlled damage in cemented carbides [39] as well as in coated hardmetals and tool steels [40-44]. The main reason for the popularity of this technique is the fact that a spherical indenter delivers concentrated stresses over a small area of the specimen surface, such that typical "blunt" service-like conditions are simulated and damage evolution associated with increasing load can be examined.

In this study, contact damage on the hardmetals was induced by applying a monotonic load through a hardmetal spherical indenter (with curvature radius of 1.25 mm) using a servohydraulic testing machine (Instron 8511). The load was imposed by means of a trapezoidal wave-form, at a loading rate of 30 $Ns^{-1}$ and applying the full test force during 20 s. Applied load ranged from 625 to 2500 N. At least three indentations were made at each load. The main goal of these tests was to obtain irreversible damage, particularly circular surface cracks. Residual depth of indentation imprints was evaluated by surface topography analysis using confocal laser scanning microscopy (CLSM, Lext OLS3100 Olympus). This technique was also employed for discerning surface damage produced by the Hertzian contacts by means of Nomarski interference contrast. This experimental protocol was designed to determine the critical load for circular crack formation under monotonic loading.

Subsurface evolution of the indentation damage with increasing load is essential for documenting crack extension phenomena from the coating surface into the hardmetal substrate. In this regard, two inspection approaches were followed. The first one was conducted by implementing the bonding interface technique (BIT), i.e. by employing "clamped-interface" specimens. BIT samples were produced following a procedure similar to that commonly employed in ceramics by Lawn's group (e.g. Refs. [45,46]), although here extended to coated systems [40,41]. It is schematically outlined in **Figure 1**, and may be described as follows: (1) a TiN-coated hardmetal specimen (with a given substrate surface finish) is transversally cut to obtain two rectangular pieces; (2) the two parts are attached tightly and put into a mould of bakelite, with the coated sides facing each other, and the ensuing surface, perpendicular to the substrate – coating / coating – substrate interfaces, is ground and polished; (3) the attained mould is then broken mechanically and, once more, the two halves are put into another mould of bakelite with the newly polished surfaces clamped face to face; (4) the coated surface is indented symmetrically across the surface trace on the interface; and finally, (5) the two parts are taken out mechanically again and indentation half-surfaces and cross sections are finally examined using FESEM. Here it must be highlighted the extreme care required in the BIT sample preparation in this study, particularly regarding alignment of the two halves and polishing stages, because the heterogeneous character of the thin coating – hard substrate systems.

The second approach was based on direct examination of cross-sections FIB-milled at specific cracked locations, partially circumventing the residual imprints. Before ion milling, a thin protective platinum layer was deposited on the areas of interest. U-shaped trenches with one cross-sectional surface perpendicular to crack path and to the specimen surface were produced by FIB with a final milling using an ion beam current of 500 pA. FESEM inspection was done on FIB-polished cross-sections.

As it will be shown later, for G specimens contact-induced crack penetration exhibits a diffuse cracking network at specific subsurface locations, depending on the groove-like surface texture. This localized and distributed crack pattern may affect the damage tolerance of these materials, as compared to the P condition. Although it is beyond the scope of this study to investigate the influence of substrate surface finish condition on damage tolerance, a simple additional test was proposed and carried out for assessment of this issue. It consisted in testing to failure coated hardmetal specimens previously indented, and documenting the interaction between the extrinsically induced damage (i.e. indentation imprint) and the failure mode. In doing so, the highest indentation load investigated (i.e. 2500 N) was used, as it yields quite different damage scenarios regarding crack penetration into the substrate. Failure was induced under four-point bending, with inner and outer spans of 20 and 40 mm respectively, on rectangular bars of 45×4×4 mm dimensions. Two specimens were evaluated for G and P surface finish conditions. The interaction between the Hertzian indentation imprint and the failure mode was inspected using CLSM.

## 3. Results and Discussion

### 3.1 Surface integrity characterization: roughness, subsurface damage and residual stresses

Machining of WC-Co cemented carbides is extremely dependent on abrasive processes, and grinding is a primary choice. Material removal during grinding takes place by the action of abrasive grits (diamond particles with sharp edges, embedded in a softer bonding agent within grinding wheels) acting as thousands of abrasive cutting points simultaneously and millions continually [47]. As a result, both the hard (carbide) and the soft (binder) phases are affected by the grinding action, and it is discerned at both surface and subsurface levels.

Roughness features for all the surface finish conditions are studied as a function of the coating process chain and related to the apparent surface texture. Mean and standard deviation values for roughness parameters $R_a$ and $R_y$ for the surface finish conditions G and P vs. the uncoated substrate, after ion-etch and after ion-etch plus deposition are given in **Table 1**. Grinding clearly affects the roughness values, these being one order of magnitude higher than those resulting from polishing. On the other hand, after ion-etching, the relative differences decrease as this substrate pre-treatment actually increases the roughness of the mirror polished surface. Furthermore, the coating deposition increases the roughness for all surface finish conditions compared to their uncoated and ion-etched states. The increased roughness is especially evident for the P condition, reaching a value similar to that of the coated G specimen. The high surface roughness determined in the coated conditions is assumed to result from the presence of protruding coating surface asperities (macroparticles) in the TiN-coatings. These micrometer-sized heterogeneities are typical for coatings grown by cathodic arc evaporation and they may negatively impact coating quality and surface finish [48-51]. Finally, grinding effects at the surface level are evidenced in terms of not only roughness but also texture. It is intimately associated with the relative movement of the grinding wheel with respect to the hardmetal substrate, leaving as a result unidirectional groove-like features.

The influence of grinding on surface integrity at the subsurface level was assessed through cross-sectional views attained by means of FIB milling (**Figure 2**). Grinding-induced damage in terms of carbide microcracking, down to depths of approximately 0.5 μm, is evidenced (**Figure 2**). Similar subsurface damage features are not discerned in the coated substrates with P finish condition. This grinding-induced damage scenario is in good agreement with that reported by Hegeman et al. [23], and is the result of the applied stresses exerted by the diamond abrasive grains during machining. On the other hand, the relatively soft metal binder is smeared out over the surface with the pulverized WC grains, and may be either partly removed from the surface together with WC grain fragments or redistributed at the subsurface level. Furthermore, cross-sectional inspection reveals dense and uniform TiN coatings with fine-grained columns along the growth direction. As expected, GTT samples are found to have the same surface topography and surface/subsurface damage as the ground substrates.

The residual stresses assessed in the WC phase, close to the interface between the substrate and the coating, are given in **Table 2** for all surface conditions studied together with the stress level of the coating. The residual stress level for the TiN coating was found to be independent of substrate surface finish. The compressive stresses at the substrate of coated G specimens are one order of magnitude higher than those assessed for the coated P substrates. However, a direct comparison of these values with those obtained on the hardmetal substrate prepared with identical grinding conditions reveals that the residual stress levels are reduced by a factor of two during the coating process [27]. This difference is ascribed to the combined effect from removal of highly stressed material during the ion etching and stress annihilation by thermal annealing during ion cleaning and coating deposition [7,17,19]. The coated GTT specimens display virtually no residual stress.

### 3.2 Spherical indentation and surface damage

Hertzian tests were aimed to induce irreversible deformation, even at the smallest applied load. Accordingly, plastic yielding was observed for all tested specimens throughout the used load range. Residual depths associated with indentation imprints are shown in **Figure 3** for the three conditions studied. It is clear that irreversible deformation gets more pronounced as indentation load is increased. Within the experimental scatter, clear differences as a function of substrate surface finish are not observed. However, a trend towards smaller residual depths is discerned for the coated G condition, particularly at the higher applied indentation load.

The evolution of surface damage induced by spherical contact under increasing indentation load was assessed by means of CLSM. Besides residual surface traces associated with irreversible deformation, the first damage feature corresponded to the appearance of short and disconnected ring cracks, circumventing the indent at the surface of the coating (**Figure 4)**. Due to the surface texture inherited from grinding (and not changed by ion-etching or coating deposition) cracks were more difficult to distinguish in the coated G and GTT specimens compared to the P treated variant. The critical load level was defined on the basis of first observed damage, i.e. no damage was observed for any of the three indents made at the immediately lower load level. Within this context, the coated G condition exhibited the highest resistance against crack nucleation (1250 N), followed by the coated P variant (1100 N) and finally the coated GTT condition (1000 N). As applied loads get higher, damage evolution and mechanisms involved are rather independent of surface condition: superposition of crack arcs into a quasi-full fissure ring and discrete appearance of partial multicracks circumventing the original single ring cracks. A small qualitative difference between ground and polished surface condition is that crack segments are less continuous in coated G and GTT specimens. The wavy surface texture associated with grinding-induced grooves affects the local propagation of ring cracks at the contact periphery. The crack propagates in a zigzag-like manner associated with peaks and valleys at the micrometer length scale, which is not the case for the smoother P surface condition. In contrast to crack nucleation, no relative difference in the load level is discerned for the evolution of a specific damage feature with respect to surface finish condition. Furthermore, local cracking related to grinding grooves and coating outgrowths are evidenced in all cases.

### 3.3 Subsurface indentation damage

The damage resulting from contact loading was assessed at the subsurface level by direct examination of either half-surface's cross-section of indented BIT specimens or FIB-milled cross-sections at specific cracked locations (partially circumventing the residual imprints). The evolution of Hertzian-induced damage with increasing applied load was qualitatively similar for all the surface conditions, independent of the inspection technique used. A detailed inspection by FESEM show that damage evidenced at the coating surface (e.g. **Figure 4**) advances through the thin film down to the interface (**Figure 5a**). The cohesive failure through the coating is quite straight and likely conforming to columnar boundaries, i.e. the cracking of the coating is directly related to the microstructural texture exhibited by the film. As the load is increased, through-thickness fissures penetrate into the substrate along the metallic binder surrounding the ceramic particles (**Figures 5b**).

A clear substrate surface finish effect is discerned by considering the load level at which the referred crack penetration, from the coating into the substrate, takes place. In this regard, examination of both BIT and FIB-milled cross-sections indicate a delayed crack extension for the coated G specimen, as compared to the coated P and GTT ones. The differences in cracking scenario are extreme in the inspected BIT specimens indented using the highest tested load (i.e. 2500 N), as illustrated in **Figure 6**. Here, crack penetration within the substrate is less than 1 μm for the coated G sample. On the contrary, deep crack penetration (> 20 μm) has already occured for the P condition at a load 1500 N, whereas clear crack extension into the substrate (> 7 μm) was observed for the coated GTT sample at a load level as low as 1000 N. In general, crack extension develops faster, in terms of applied load and particularly once the crack has already penetrated into the substrate, in indented BIT samples than in specimens without any artificial interface. This finding may be rationalized on the basis of the investigation conducted by Helbawi and coworkers on the differences in subsurface damage in indented alumina specimens with and without bonding layer [52]. In their study, it is reported that the stress distribution shifts and concentates more to the surface in BIT specimens compared to integral ones. Such qualitatively behavior differences depending on the specimens geometry used was not observed for the coated G specimen (with a high compressive residual stresses at the surface), as it exclusively shows shallow penetration (< 1 μm) for all the test conditions studied.

The above findings points out that G substrate surface finish exhibit higher crack penetration resistance than the P and GTT ones. However, it should be noted that the coated G specimens may exhibit different cracking extension pattern at specific subsurface locations, depending on surface texture features. As it is illustrated in **Figure 7a**, the presence of groove-like features (i.e. peaks/valleys) may promote the interaction of penetrating contact-induced cracks with the pre-existing grinding-induced fissures. It then yields a diffusing crack network restricted within a thin (~ 1-2 μm) subsurface layer. On the other hand, just shallow cracking is discerned at regular and smooth locations (**Figure 7b**).

### 3.4 Substrate surface finish effects on contact damage resistance

All the damage events identified using different inspection approaches, together with the load associated with their emergence in the different coated specimens studied, are given in **Figure 8**. They are presented as a contact damage map as a function of applied load, under spherical indentation conditions.

Considering the coated P condition as reference, it may be discerned that emergence of specific damage events is delayed (in terms of applied load) as substrate is just ground, before ion-etching and coating. However, if the ground substrate is thermal annealed before coating (GTT condition), damage takes place earlier than for the coated P samples. Relative beneficial effects associated with grinding are more relevant in terms of resistance to crack penetration, from the coating into the substrate, than resistance to crack nucleation under contact loading. It points out that compressive residual stresses existing at the substrate subsurface (about ~1 GPa) are more effectively shielding through-thickness (coating) cracks than preventing their nucleation at the coating surface. Such positive influence may be rationalized on the basis of two different action-effect correlations. First, as a compressive residual stress field is superimposed to the far-field applied stress during contact loading, the driving force for crack extension diminishes because effective stress intensity factor at the crack tip is reduced. Second, compressive residual stresses act to close pre-existing fissures introduced during grinding before coating, yielding as a result an effective recovery of the faulted mechanical integrity at the subsurface level. The relevance of these effects is experimentally supported by direct comparison of the contact damage response of the coated G and GTT conditions, as clearly evidenced in **Figure 8**. There, it may be seen that once the compressive residual stresses are relived through heat treatment (GTT condition), the improved crack penetration resistance exhibited by the G specimen is completely lost.

Finally, the interaction among propagating contact-induced cracks, a remnant compressive stress field, surface texture features and pre-existing grinding-induced microcracks at the subsurface deserves an additional analysis. As it is observed in **Figure 7a**, damage scenario resulting from the referred interaction seems to be less localized (and thus critical) than those observed for the P condition. In general terms, it may be described as distributed and oriented, departing from the straight and longitudinal crack path exhibited by the through-thickness fissures nucleated at the surface. Indeed, it somehow resembles the damage scenario resulting after contact loading of structural ceramics with heterogeneous microstructures, which has been validated as an optimal microstructure tailoring strategy for improving damage tolerance of these materials [37]. Based on this assumption, a simple damage tolerance investigation was made for the G and P coated systems. It consisted of bend testing to failure coated specimens previously indented. **Figure 9** shows surface CLSM micrographs of failures from Hertzian indentation sites in bars broken for these two coated systems. As expected, in both cases ruptures are associated with indentation-induced damage. However, interaction between extrinsic damage and failure path are different. While rupture in the coated P system is characterized by surface traces of the fracture going along previously identified surface ring (**Figure 9a**), fracture for the coated G condition traverses the inner contact orthogonally (i.e. following grinding-induced grooves) (**Figure 9b**). As extrinsically induced damage is maximum (regarding depth and localization) in the contour periphery, these findings would point out a beneficial effect of grinding-induced changes (i.e. distributed damage, surface texture and residual stresses) with respect to effective damage tolerance of the coated hardmetals studied. Nevertheless, the findings presented here are limited, and further research is recalled in this field if grinding effects on damage tolerance of coated hardmetals want to be documented and understood throughly.

## 4. Conclusions

The influence of surface topography and subsurface integrity, resulting from abrasive grinding of the hardmetal substrate, on the contact damage resistance of a TiN-coated 13 wt.% Co fine-grained hardmetal has been studied. The experimental study involved introduction of controlled damage under monotonic spherical indentation and assessment of contact damage resistance in terms of crack prevention (nucleation) as well as crack containment (extension). The main results and conclusions are summarized as follows:

1) Substrate grinding enhances contact damage resistance in terms of both critical load for crack emergence and subsequent damage evolution. This beneficial effect is particularly relevant regarding extension of surface cracks into the hardmetal substrate, corresponding crack penetration being rather shallow for the coated ground condition.

2) The grinding-induced compressive residual stresses are pointed out as the main reason for the enhanced contact damage resistance, as discerned from the direct comparison between the responses observed for the coated G and GTT conditions. Such remnant stress state overcomes the potential deleterious effect expected from surface texture (peak/valley stress raisers) or pre-existing grinding-induced damage, the latter given by microcracks confined but widely distributed within a thin subsurface layer (about 1 μm in depth).

3) The interaction among resulting surface integrity (especially the above referred distributed damage) and the cracks introduced by the external contact loads indicates an additional positive effect in terms of damage tolerance.

**Acknowledgements**

Funding for this investigation was partly supplied by the Spanish MINECO (Grant No. MAT 2012-34602). One of the authors (J.Y.) acknowledges funding received through Erasmus Mundus joint European Doctoral Programme DocMASE. The authors sincerely thank J. Esteve (Universitat de Barcelona) for experimental assistance in heat treatment activities. Research work was conducted within a cooperative effort among SECO Tools AB, Linköping University and Universitat Politècnica de Catalunya.

## References


[1] Prakash L. Fundamentals and general applications of hardmetals. In: Sarin VK, Mari D, Llanes L (Vol. eds.), *Comprehensive Hard Materials*. Elsevier 2014; Vol. 1, Ch. 1.02, p. 29-90.

[2] Bewilogua K, Bräuer G, Dietz A, Gäbler J, Goch G, Karpuschewski B, et al. Surface technology for automotive engineering. CIRP Annals Manufacturing Technology 2009;58:608-27.

[3] Bouzakis KD, Michailidis N, Skordaris G, Bouzakis E, Biermann D, M'Saoubi R. Cutting with coated tools: coating technologies, characterization methods and performance optimization. CIRP Annals - Manufacturing Technology. 2012;61:703-3.

[4] Fang ZZ, Koopman MC, Wang H. Cemented tungsten carbide hardmetal – An introduction. In: Sarin VK, Mari D, Llanes L (Vol. eds.), *Comprehensive Hard Materials.* Elsevier 2014; Vol. 1, Ch. 1.04, p. 123-37.

[5] Mitterer C. PVD and CVD hard coatings. In: Sarin VK, Llanes L, Mari D (Vol. eds.), *Comprehensive Hard Materials.* Elsevier 2014; Vol. 2, Ch. 2.16, p. 449-67.

[6] Byrne G, Dornfeld D, Denkena B. Advancing cutting technology. CIRP Annals - Manufacturing Technology. 2003;52:483-507.

[7] Breidenstein B, Denkena B. Significance of residual stress in PVD-coated carbide cutting tools. CIRP Annals - Manufacturing Technology. 2013;62:67-70.

[8] Bouzakis K-D, Vidakis N, David K. The concept of an advanced impact tester supported by evaluation software for the fatigue strength characterization of hard layered media. Thin Solid Films 1999;355–356:322–9.

[9] Fox V, Jones A, Renevier NM, Teer DG. Hard lubricating coatings for cutting and forming tools and mechanical components. Surface and Coatings Technology. 2000;125:347-53.

[10] Mitterer C, Holler F, Reitberger D, Badisch E, Stoiber M, Lugmair C, et al. Industrial applications of PACVD hard coatings. Surface and Coatings Technology. 2003;163–164:716-22.

[11] Podgornik B, Hogmark S, Pezdirnik J. Comparison between different test methods for evaluation of galling properties of surface engineered tool surfaces. Wear. 2004;257:843-51.

[12] Bromark M, Larsson M, Hedenqvist P, Olsson M, Hogmark S. Influence of substrate surface topography on the critical normal force in scratch adhesion testing of TiN-coated steels. Surface and Coatings Technology. 1992;52:195-203.

[13] Knotek O, Löffler F, Krämer G. Substrate- and interface-related influences on the performance of arc-physical-vapour-deposition-coated cemented carbides in interrupted-cut machining. Surface and Coatings Technology. 1992;54-55:476-81.

[14] Tönshoff HK, Karpuschewski B, Mohlfeld A, Seegers H. Influence of subsurface properties on the adhesion strength of sputtered hard coatings. Surface and Coatings Technology. 1999;116–119:524-9.

[15] Tönshoff HK, Seegers H. X-Ray diffraction characterization of pre-treated cemented carbides for optimizing adhesion strength of sputtered hard coatings. Surface and Coatings Technology. 2001;142–144:1100-4.

[16] Carlsson P, Olsson M. PVD coatings for sheet metal forming processes — a tribological evaluation. Surface and Coatings Technology. 2006;200:4654-63.

[17] Denkena B, Breidenstein B. Influence of the residual stress state on cohesive damage of PVD-coated carbide cutting tools. Advanced Engineering Materials. 2008;10:613-6.

[18] Heinrichs J, Jacobson S. Laboratory test simulation of aluminium cold forming – influence from PVD tool coatings on the tendency to galling. Surface and Coatings Technology. 2010;204:3606-13

[19] Denkena B, Breidenstein B. Residual stress distribution in PVD-coated carbide cutting tools: Origin of cohesive damage. Tribology in Industry. 2012;34:158-65.

[20] Denkena B, Spengler C. Influence of different grinding processes on surface and subsurface characteristics of carbide tools. Advances in Abrasive Technology Vi. 2004;257-258:195-200.

[21] Yang J, Roa JJ, Odén M, Johansson-Jõesaar MP, Esteve J, Llanes L. Substrate surface finish effects on scratch resistance and failure mechanisms of TiN-coated hardmetals. Surface and Coatings Technology 2015; 265: 174-84.

[22] Sheikh S, M´Saoubi R, Flasar P, Schwind M, Persson T, Yang J, Llanes L. Fracture toughness of cemented carbides: Testing method and microstructural effects, International Journal of Refractory Metals and Hard Materials 2015; 49:153-60.

[23] Hegeman JBJW, De Hosson JTM, de With G. Grinding of WC–Co hardmetals. Wear. 2001;248:187-96.

[24] Jiang D, Anné G, Vleugels J, Vanmeensel K, Eeraerts W, Liu, W, et al. Residual stresses in hardmetals caused by grinding and EDM machining and their influence on the flexural strength. In: Kneringer G, Rödhammer P, Wildner H. (Eds.). *Powder Metallurgical High Performance Materials, 16th International Plansee Seminar* (Reutte, Austria). Plansee 2005; Vol. 2, p. 1075-85.

[25] Merkleina M, Andreasa K, Engela U. Influence of machining process on residual stresses in the surface of cemented carbides. Procedia Engineering. 2011;19:252-7.

[26] Jawahir IS, Brinksmeier E, M'Saoubi R, Aspinwall DK, Outeiro JC, Meyer D, et al. Surface integrity in material removal processes: Recent advances. CIRP Annals - Manufacturing Technology. 2011;60:603-26.

[27] Yang J, Odén M, Johansson-Jõesaar MP, Llanes L. Grinding effects on surface integrity and mechanical strength of WC-Co cemented carbides. Procedia CIRP. 2014;13:257-63.

[28] Exner HE. The influence of sample preparation on Palmqvist's method for toughness testing of cemented carbides. Transactions of the Metallurgical Society of AIME 1969; 245: 677-83.

[29] Torres Y, Casellas D, Anglada M, Llanes L. Fracture toughness evaluation of hardmetals: influence of testing procedure. International Journal of Refractory Metals and Hard Materials 2001; 19: 27-34.

[30] Llanes L, Casas B, Idañez E, Marsal M, Anglada M. Surface integrity effects on the fracture resistance of electrical-discharge-machined WC-Co cemented carbides. Journal of the American Ceramic Society 2004; 87: 1687–93.

[31] Casas B, Torres Y, Llanes L. Fracture and fatigue behavior of electrical-discharge machined cemented carbides. International Journal of Refractory Metals and Hard Materials 2006; 24: 162–7.

[32] Llanes L, Casas B, Torres Y, Salán N, Mestra A. Fatigue performance improvement of electrical discharge machined hardmetals by means of combined thermal annealing and surface modification routes. International Journal of Refractory Metals and Hard Materials 2013; 36: 60-5.

[33] Oliver WC, Pharr GM. An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments. Journal of Materials Research. 1992;7:1564-83.

[34] Mari D, Krawitz AD, Richardson JW, Benoit W. Residual stress in WC-Co measured by neutron diffraction. Materials Science and Engineering A 1996; 209: 197-205.

[35] Larsson C, Odén M. X-ray diffraction determination of residual stresses in functionally graded WC–Co composites. International Journal of Refractory Metals and Hard Materials 2004; 22: 177-84.

[36] Livescu V, Clausen B, Paggett JW, Krawitz AD, Drake EF, Bourke MAM. Measurement and modeling of room temperature co-deformation in WC–10 wt.% Co. Materials Science and Engineering A 2005; 399: 134-40.

[37] Lawn BR. Indentation of ceramics with spheres: a century after Hertz. Journal of the American Ceramic Society 1998;81: 1977-94.

[38] Jiménez-Piqué E, Anglada M, Llanes L. Resistance to contact deformation and damage of hard ceramics. In: Sarin VK, Llanes L, Mari D (Vol. eds.), *Comprehensive Hard Materials.* Elsevier 2014; Vol. 2, Ch. 2.13, p. 367-81.

[39] Góez A, Coureaux D, Ingebrand A, Reig B, Tarrés E, Mestra A, et al. Contact damage and residual strength in hardmetals. International Journal of Refractory Metals and Hard Materials 2012; 30: 121-7.

[40] Tarrés E, Ramírez G, Gaillard Y, Jiménez-Piqué E, Llanes L. Contact fatigue behavior of PVD-coated hardmetals. International Journal of Refractory Metals and Hard Materials 2009;27:323-31.

[41] Llanes L, Tarrés E, Ramírez G, Botero CA, Jiménez-Piqué E. Fatigue susceptibility under contact loading of hardmetals coated with ceramic films. Procedia Engineering 2010;2:299-308.

[42] Ramírez G, Mestra A, Casas B, Valls I, Martínez R, Bueno R, et al. Influence of substrate microstructure on the contact fatigue strength of coated cold-work tool steels. Surface and Coatings Technology 2012; 206: 3069-81.

[43] Yang J, Botero CA, Cornu N, Ramírez G, Mestra A, Llanes L. Mechanical response under contact loads of AlCrN-coated tool materials. IOP Conf. Series: Materials Science and Engineering 2013;48:012003.

[44] Ramírez G, Jiménez-Piqué E, Mestra A, Vilaseca M, Casellas D, Llanes L, A comparative study of the contact fatigue behavior and associated damage micromechanisms of TiN and WC:H-coated cold-work tool steel, Tribology International 2015;88:263-270.

[45] Guiberteau F, Padture NP, Lawn BR. Effect of grain size on Hertzian contact damage in alumina. Journal of the American Ceramic Society 1994;77:1825-31.

[46] Cai H, Stevens Kalceff MA, Lawn BR. Deformation and fracture of mica-containing glass-ceramics in Hertzian contacts, Journal of Materials Research 1994; 9: 762-70.

[47] Malking S, Guo C. Grinding technology: Theory and application of machining with abrasives. Second Edition, Industrial Press Inc. New York 2008.

[48] Matthews A, Lefkow AR. Problems in the physical vapour deposition of titanium nitride. Thin Solid Films. 1985;126:283-91.

[49] Münz WD, Lewis DB, Creasey S, Hurkmans T, Trinh T, Ijzendorn W. Defects in TiN and TiAIN coatings grown by combined cathodic arc/unbalanced magnetron technology. Vacuum. 1995;46:323-30.

[50] Anders A. Approaches to rid cathodic arc plasmas of macro- and nanoparticles: a review. Surface and Coatings Technology. 1999;120–121:319-30.

[51] Harlin P, Carlsson P, Bexell U, Olsson M. Influence of surface roughness of PVD coatings on tribological performance in sliding contacts. Surface and Coatings Technology. 2006;201:4253-9.

[52] Helbawi H, Zhang L, Zarudi I. Difference in subsurface damage in indented specimens with and without bonding layer. International Journal of Mechanical Sciences. 2001;43:1107-21.

# Table captions

**Table 1.** Nomenclature and roughness parameters ($R_a$ and $R_y$) associated with substrate surface conditions and the coating process steps: uncoated; ion-etched; and coated.

**Table 2.** Residual stresses measured (for the WC phase) on the substrate surface of coated systems for the G, P and GTT conditions. The intrinsic residual stresses level for the coating is also listed for comparison purpose.

**Table 1**

| Condition | Substrate surface finish | $R_a$ (µm) | | | $R_y$ (µm) | | |
|---|---|---|---|---|---|---|---|
| | | Uncoated | Ion-etched | Coated | Uncoated | Ion-etched | Coated |
| **G** | Ground | 0.19±0.07 | 0.16±0.02 | 0.25±0.05 | 1.05±0.35 | 1.03±0.12 | 1.72±0.30 |
| **P** | Polished | 0.01±0.01 | 0.09±0.01 | 0.27±0.05 | 0.11±0.04 | 0.59±0.06 | 1.54±0.20 |

**Table 2**

| Condition | Residual stresses (MPa) |
|---|---|
| G+Coat | -1071±24 |
| P+Coat | -59±15 |
| GTT+Coat | -118±14 |
| Coating | -3299±140 |

## Figure captions

**Fig. 1.** Schematic show of the sample preparation procedure of bonding interface technique (BIT) for analysis of sub-surface damage induced under Hertzian contact stresses in cross sections. Note that the size ratio between the coating and the substrate is exaggerated.

**Fig. 2.** Cross-section view of the coated system, corresponding to the ground substrate surface finish. Note that FIB milling was made perpendicular to the grinding marks, and grinding-induced damage is pointed out with arrows.

**Fig. 3.** Residual depth of the indentation imprint for the G, P and GTT conditions corresponding to each indentation load level: 625 N, 1000 N, 1250 N, 1500 N, 1870 N and 2500 N. The dashed lines are linear fittings for the three conditions.

**Fig. 4.** Circular cracks in the surface of the P conditioned sample, generated with a sphere of 1.25mm curvature radius and applied load level of 2500 N.

**Fig. 5.** The evolution of crack penetration for P conditioned sample corresponding to the load levels: (a) 1500 N and (b) 1870 N, respectively. The bottom two images are the enlarged views of the upward regions indicated by the dashed squares, respectively.

**Fig. 6.** Cross-section view of crack penetration feature in the inspected BIT specimens for the (a) G, (b) P and (c) GTT conditions at the 2500 N load level.

**Fig. 7.** Two different crack path views for the G conditioned sample at the load level 1500 N: (a) crack propagates into a substrate area containing pre-existing grinding-induced fissures; and (b) crack penetrates into a clean sub-surface where the surface texture irregularities are absent. The bottom two images are the enlarged views of the upward regions indicated by the dashed squares, respectively.

**Fig. 8.** Damage map showing the surface top view and the cross-section views obtained by BIT and FIB techniques, for the three substrate surface finish conditions (G, P and GTT) and different load levels studied. The meaning of the symbols inside the map is indicated by the right schematic drawing: blank – no crack/no penetration; half filled – partial cracking/shallow penetration (<3 μm); full filled – multi cracking/pronounced penetration (>5 μm).

**Fig. 9.** Surface CLSM micrographs of failures sites from Hertzian indentation at 2500 N in bars broken for the investigated coated systems: (a) coated P condition - failure origin at surface ring crack; and (b) coated G condition - fracture path going through the inner contact orthogonally (i.e. following grinding-induced grooves). The residual imprints of spherical contact are underlined by dashed lines.

Figure 1

Click here to download high resolution image

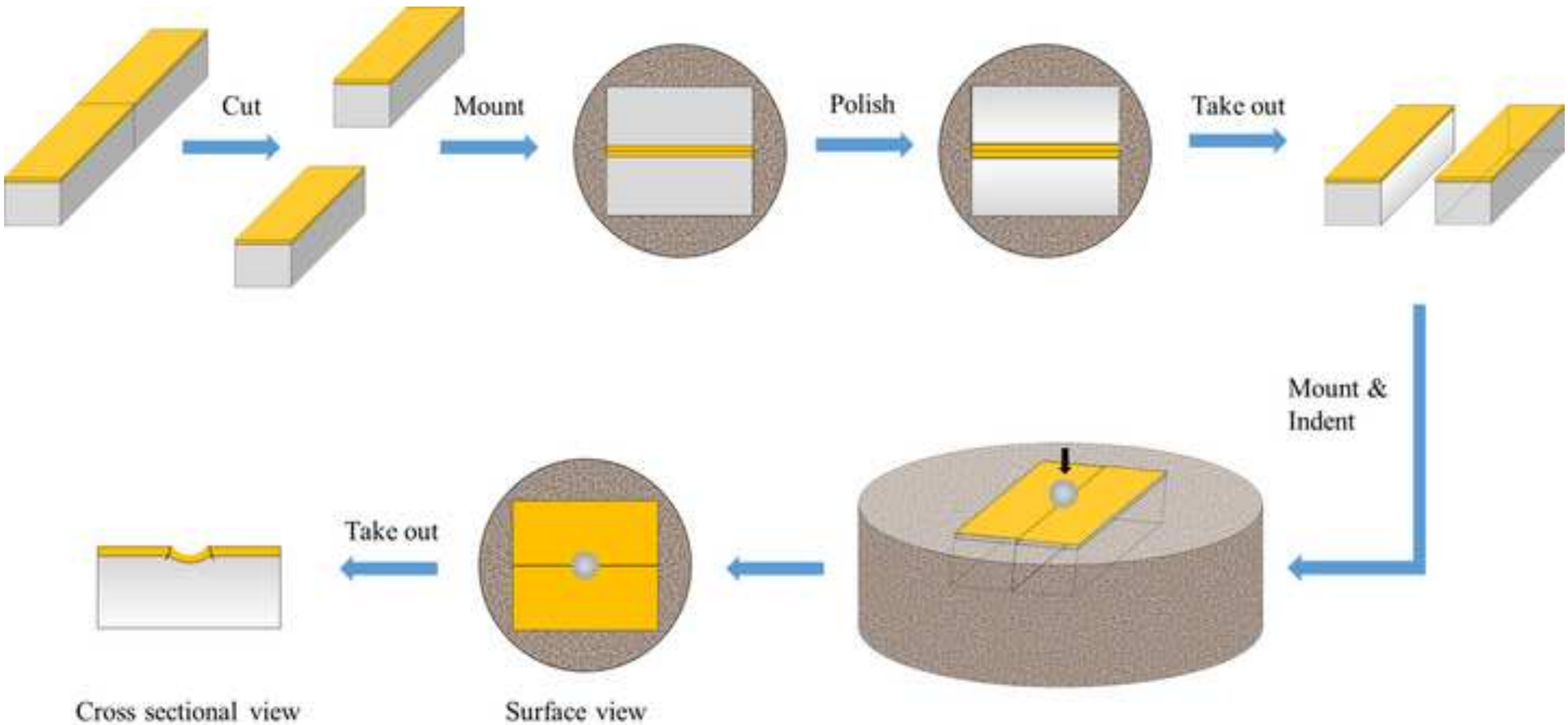

Figure 2
Click here to download high resolution image

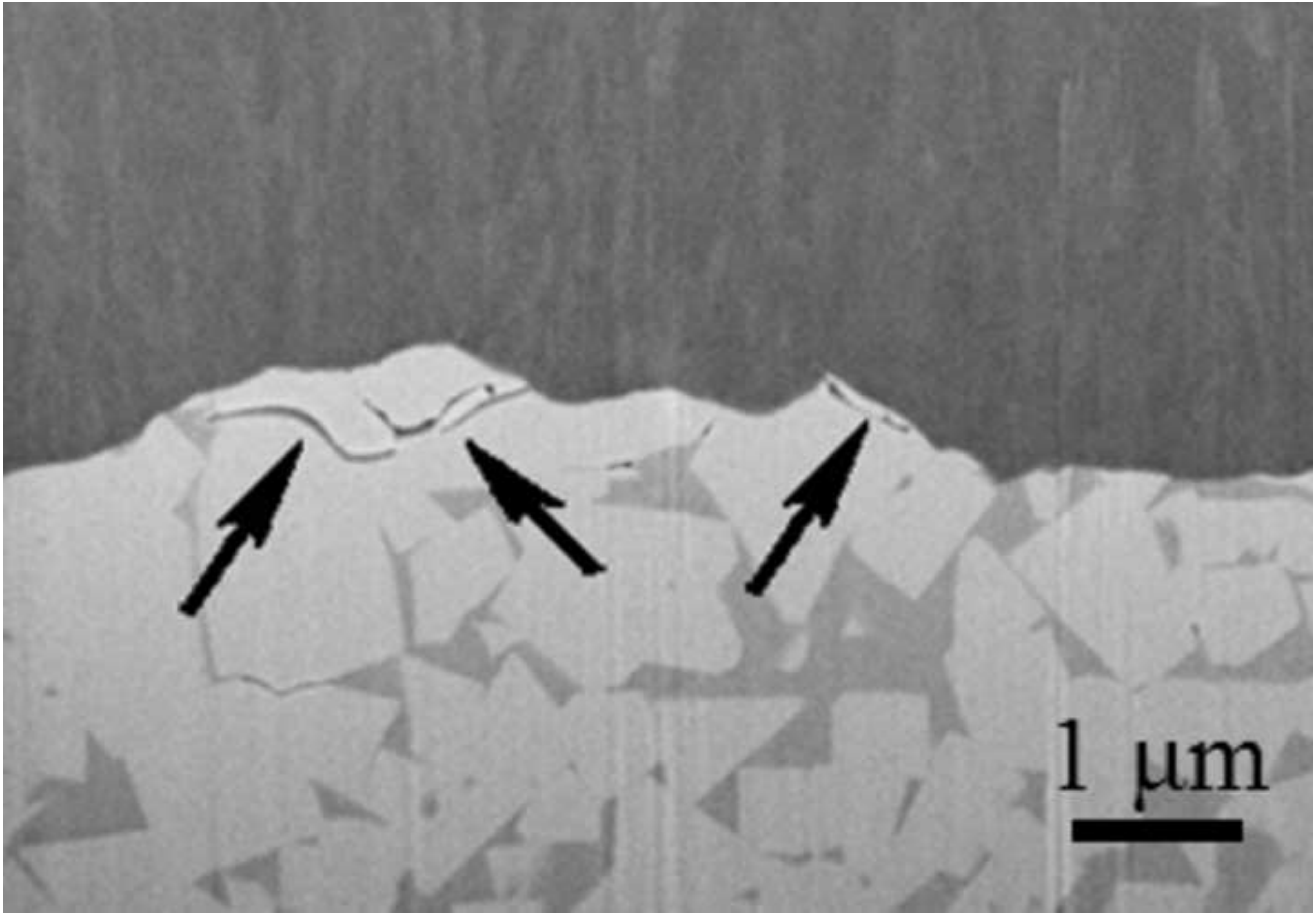

Figure 3
Click here to download high resolution image

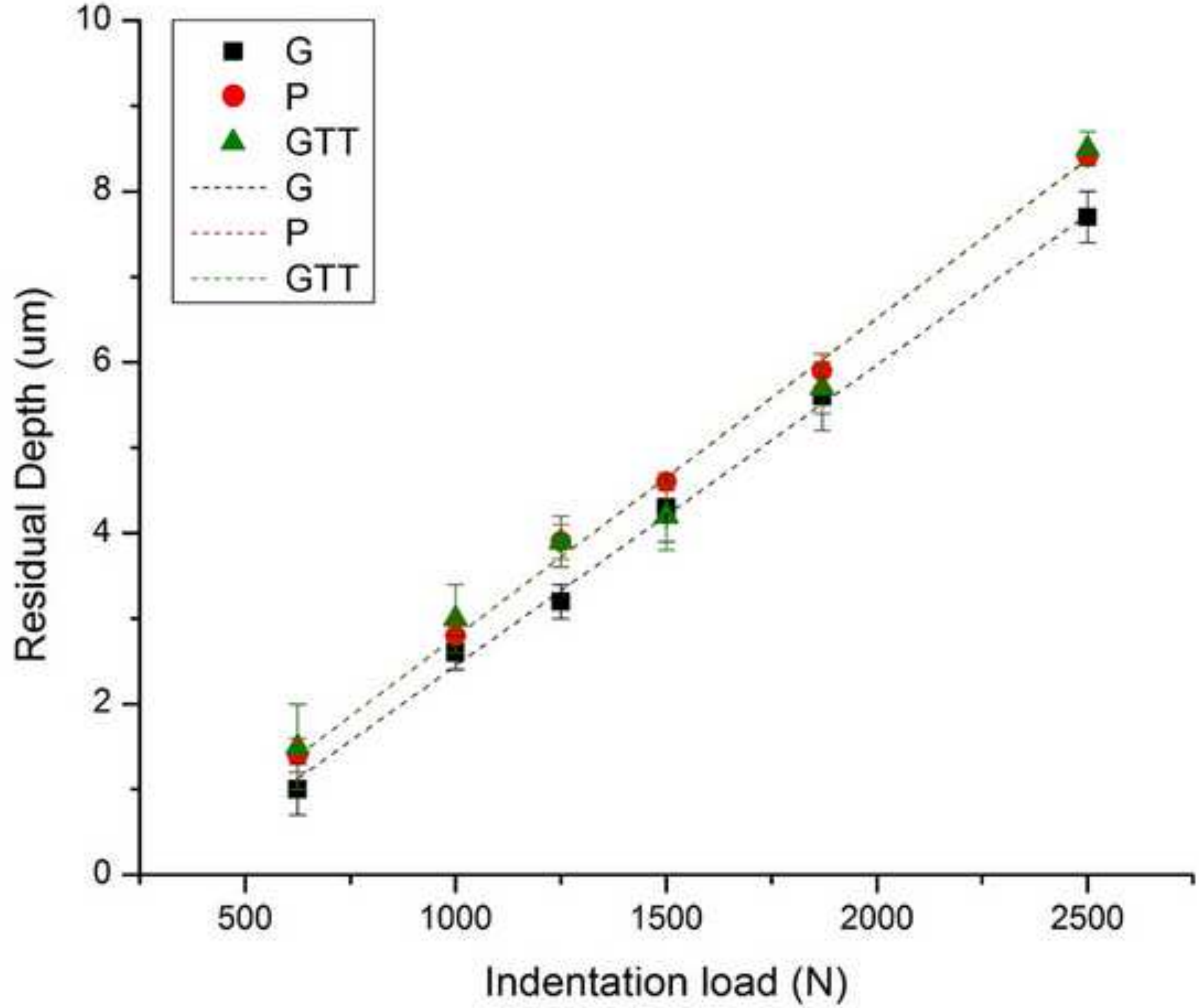

Figure 4
Click here to download high resolution image

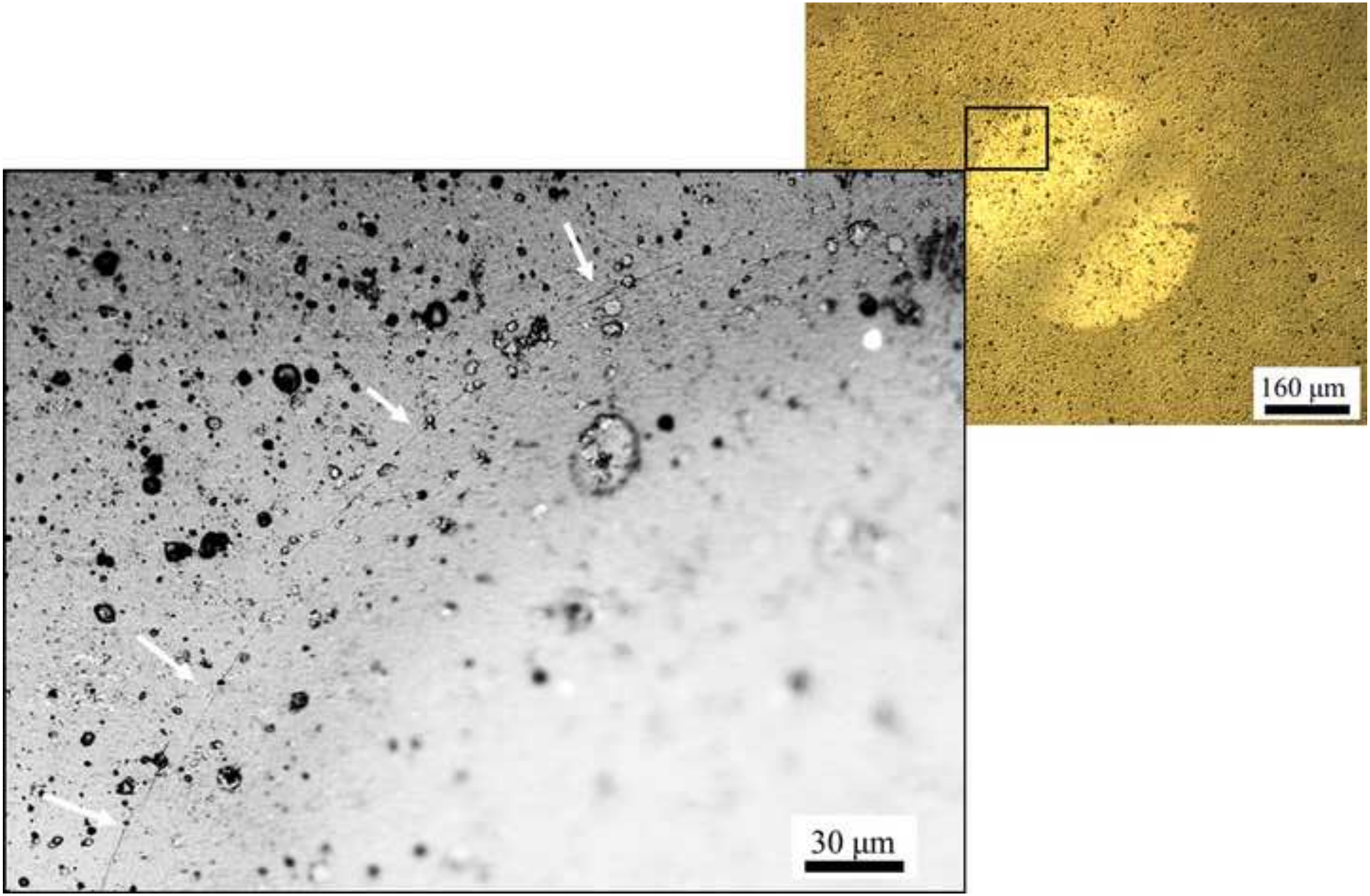

Figure 5a

Click here to download high resolution image

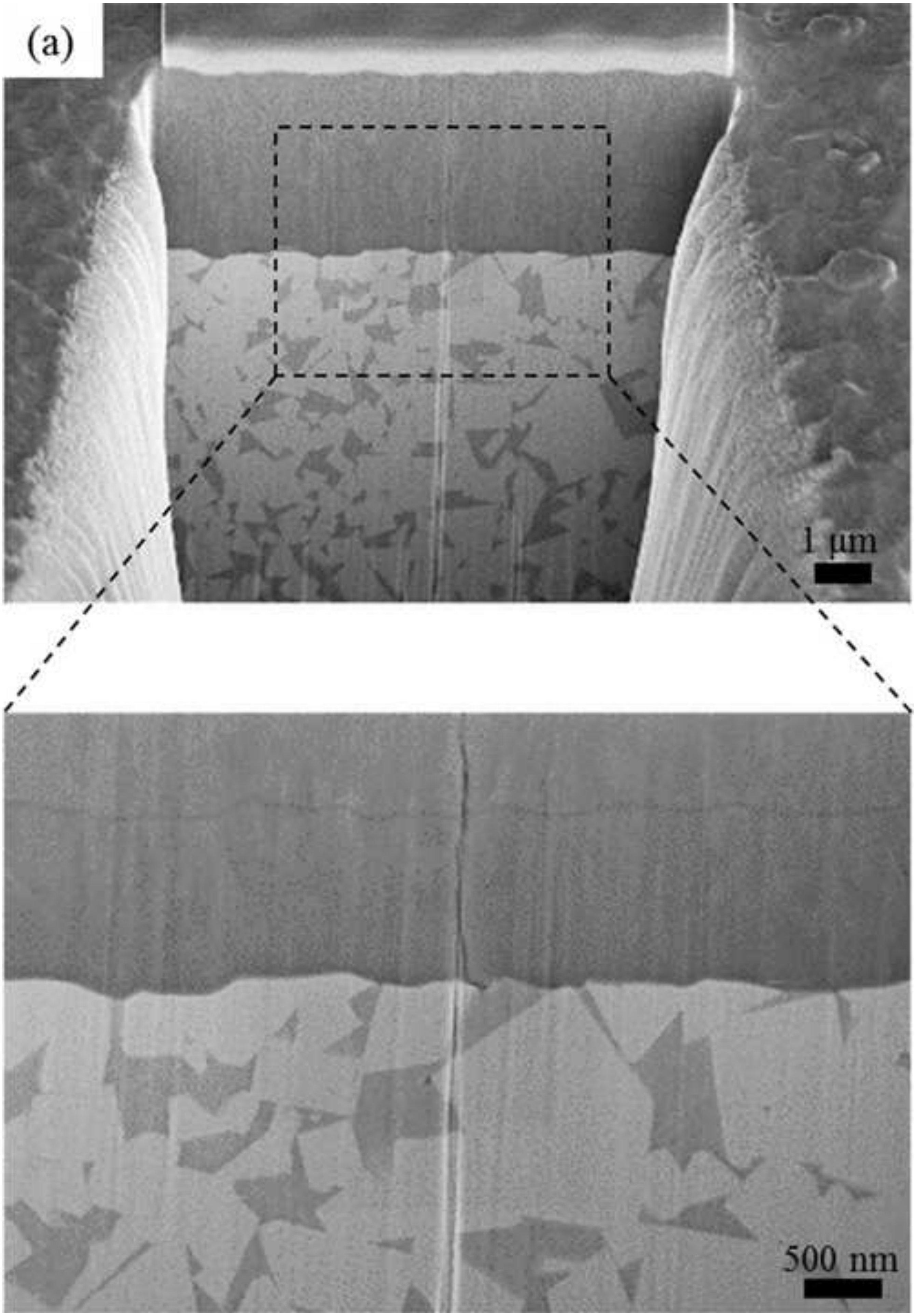

Figure 5b
Click here to download high resolution image

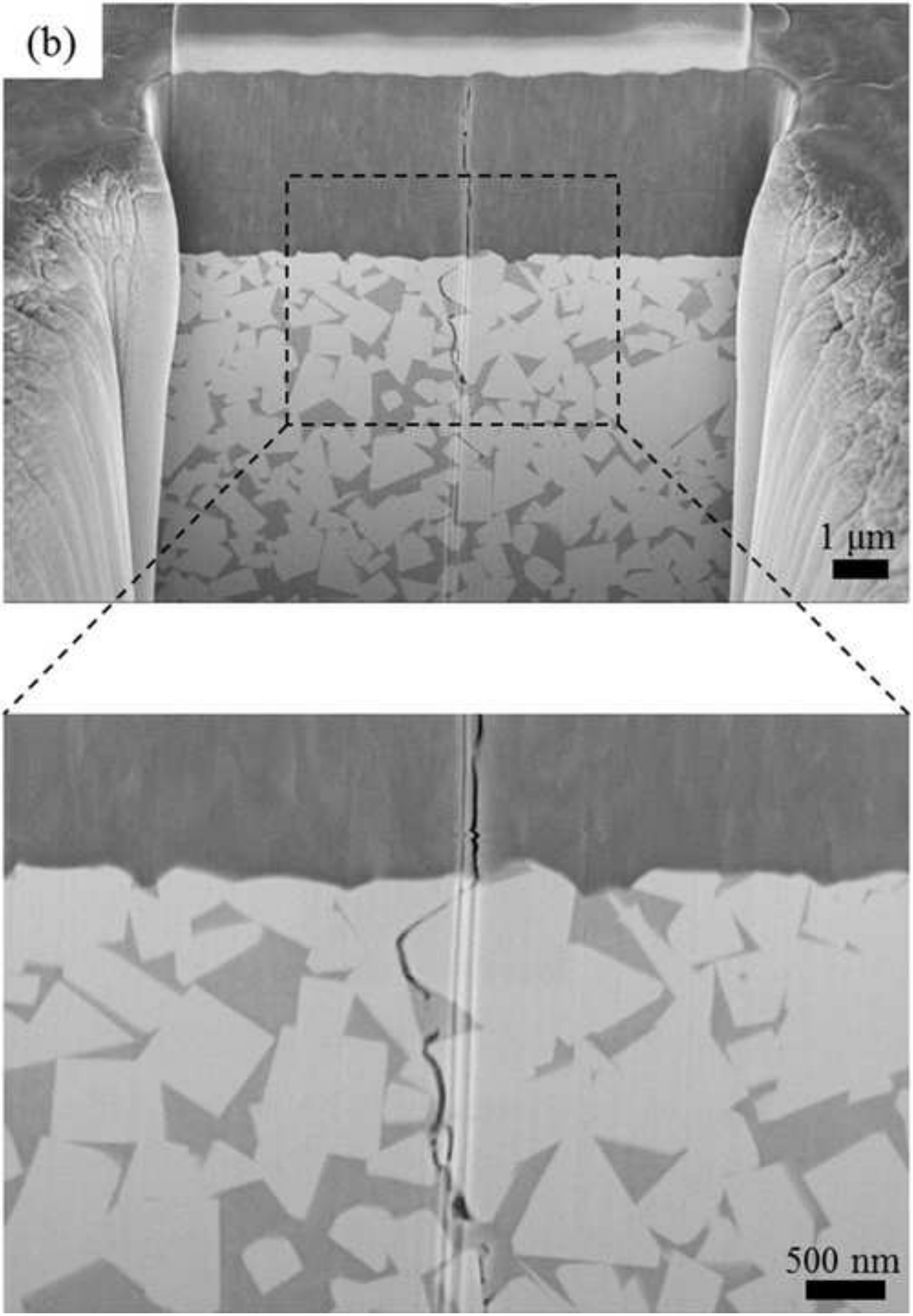

Figure 6a
Click here to download high resolution image

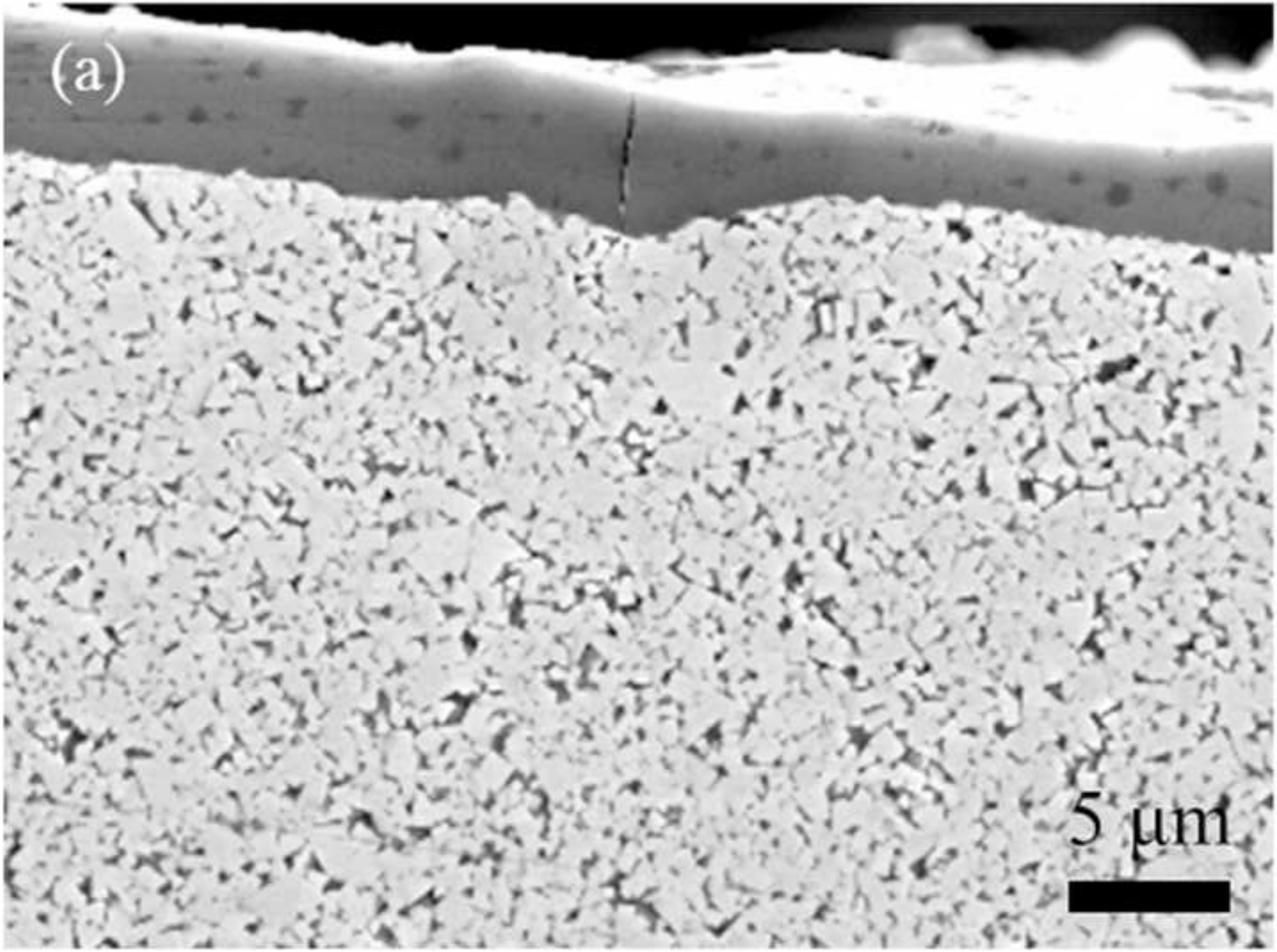

Figure 6b
Click here to download high resolution image

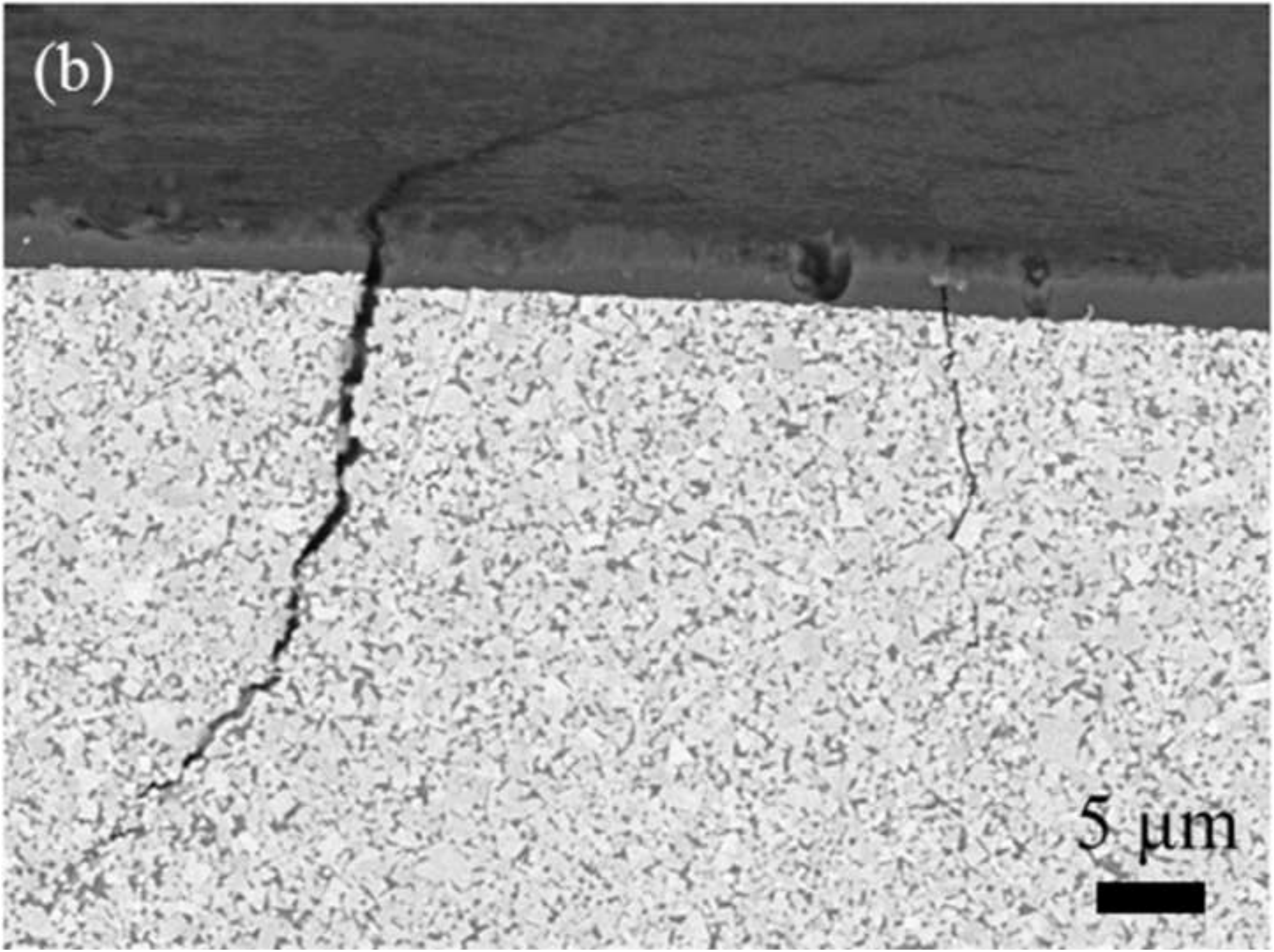

Figure 6c
Click here to download high resolution image

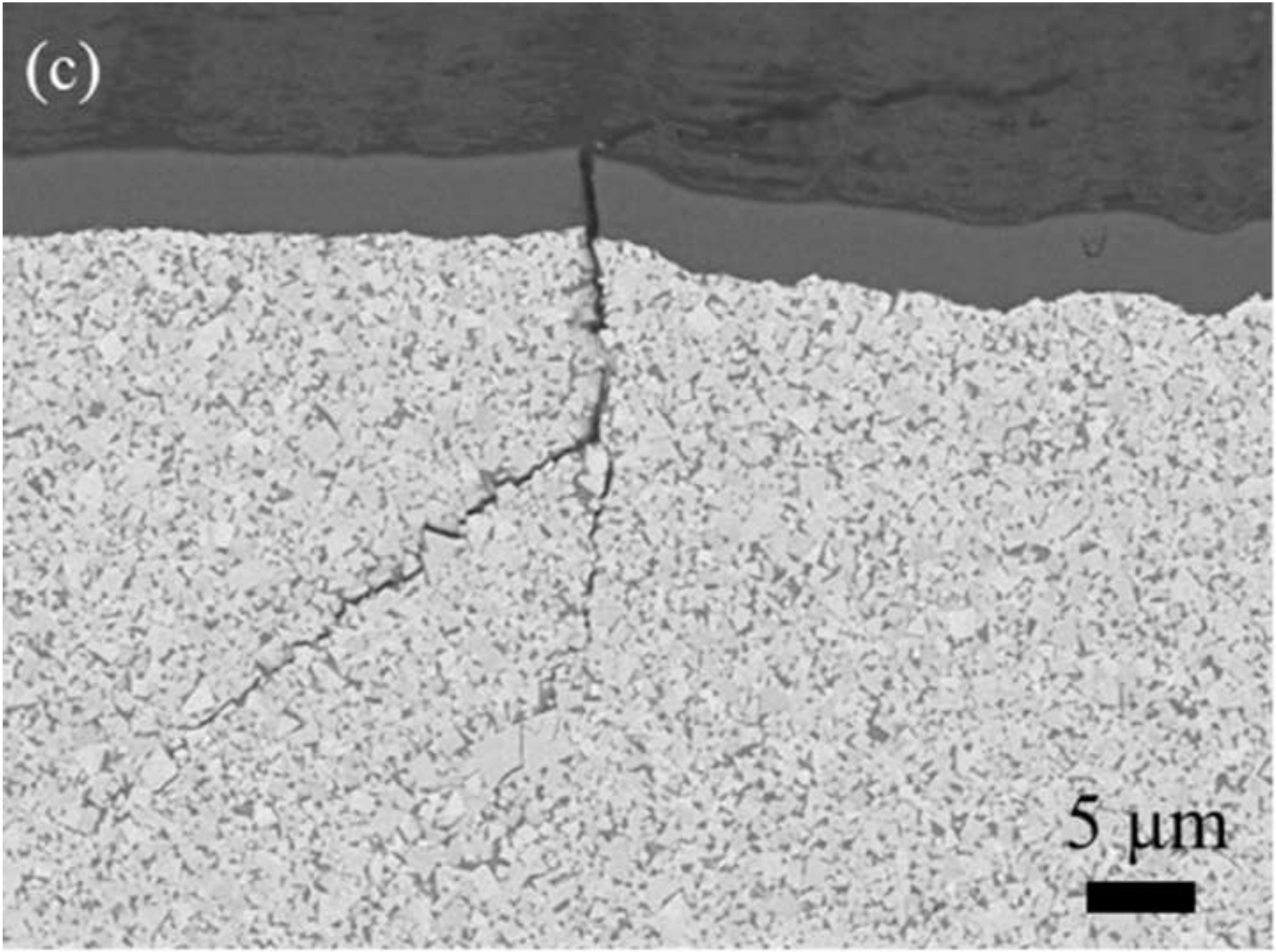

Figure 7a
Click here to download high resolution image

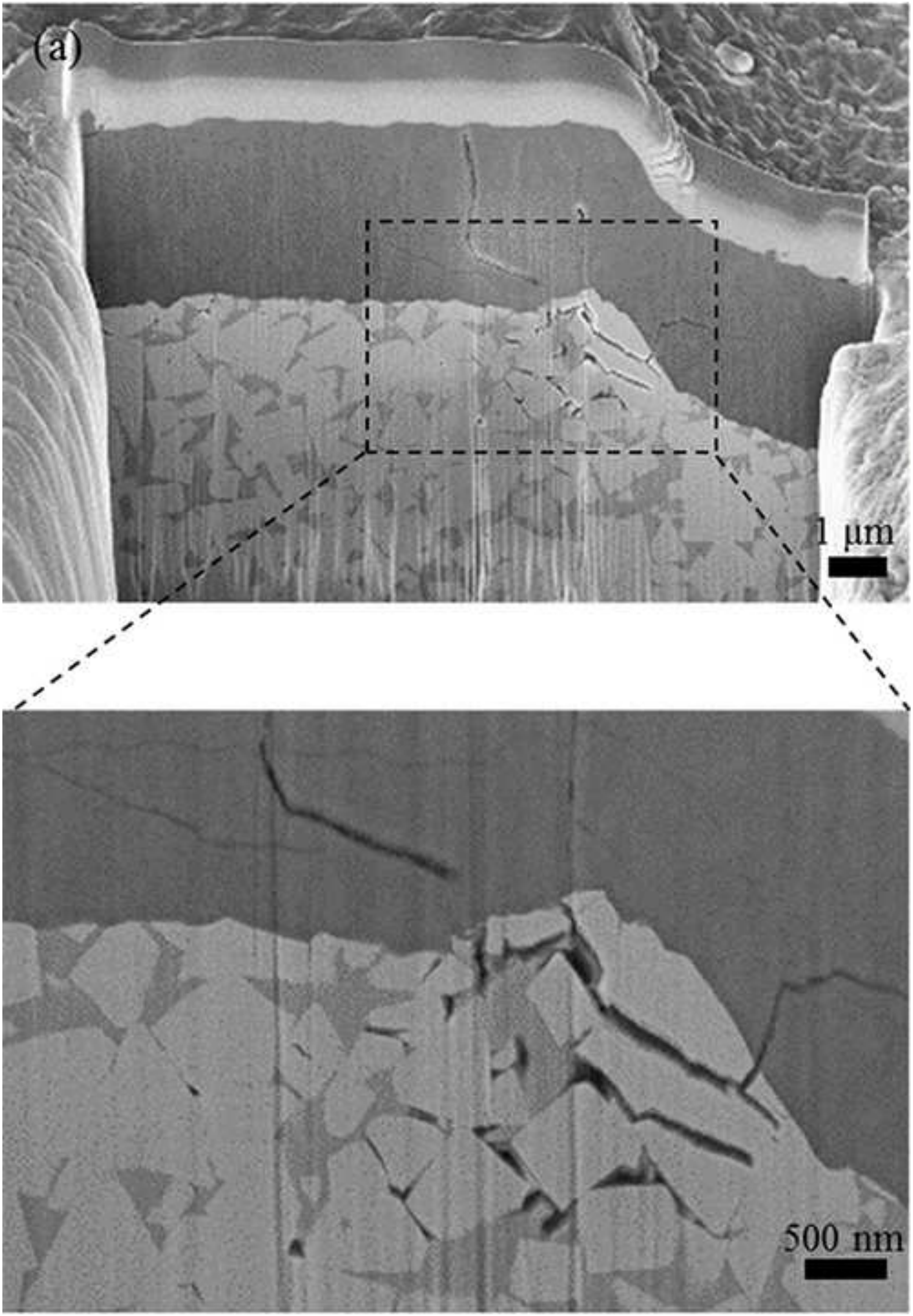

Figure 7b
Click here to download high resolution image

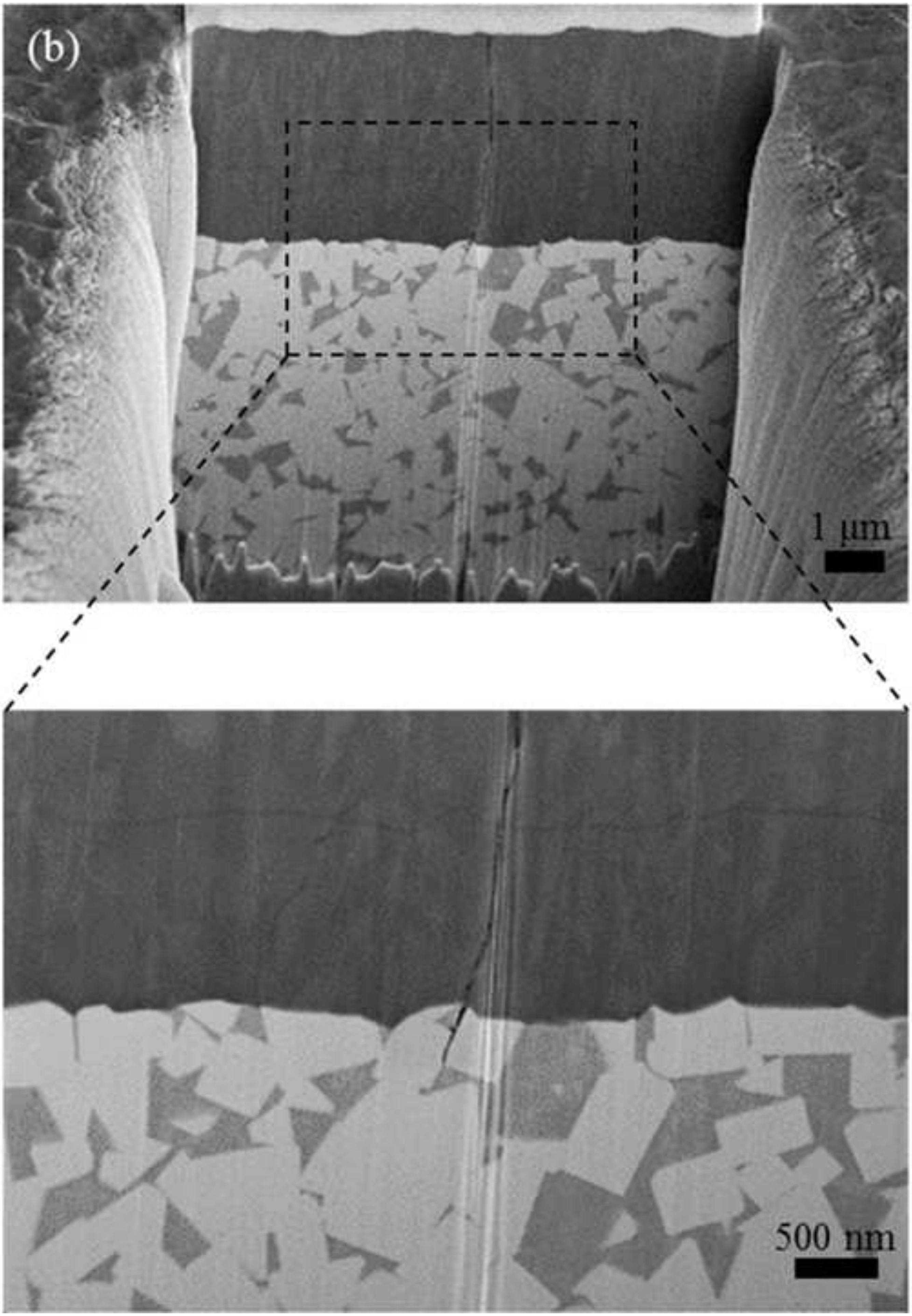

Figure 8
Click here to download high resolution image

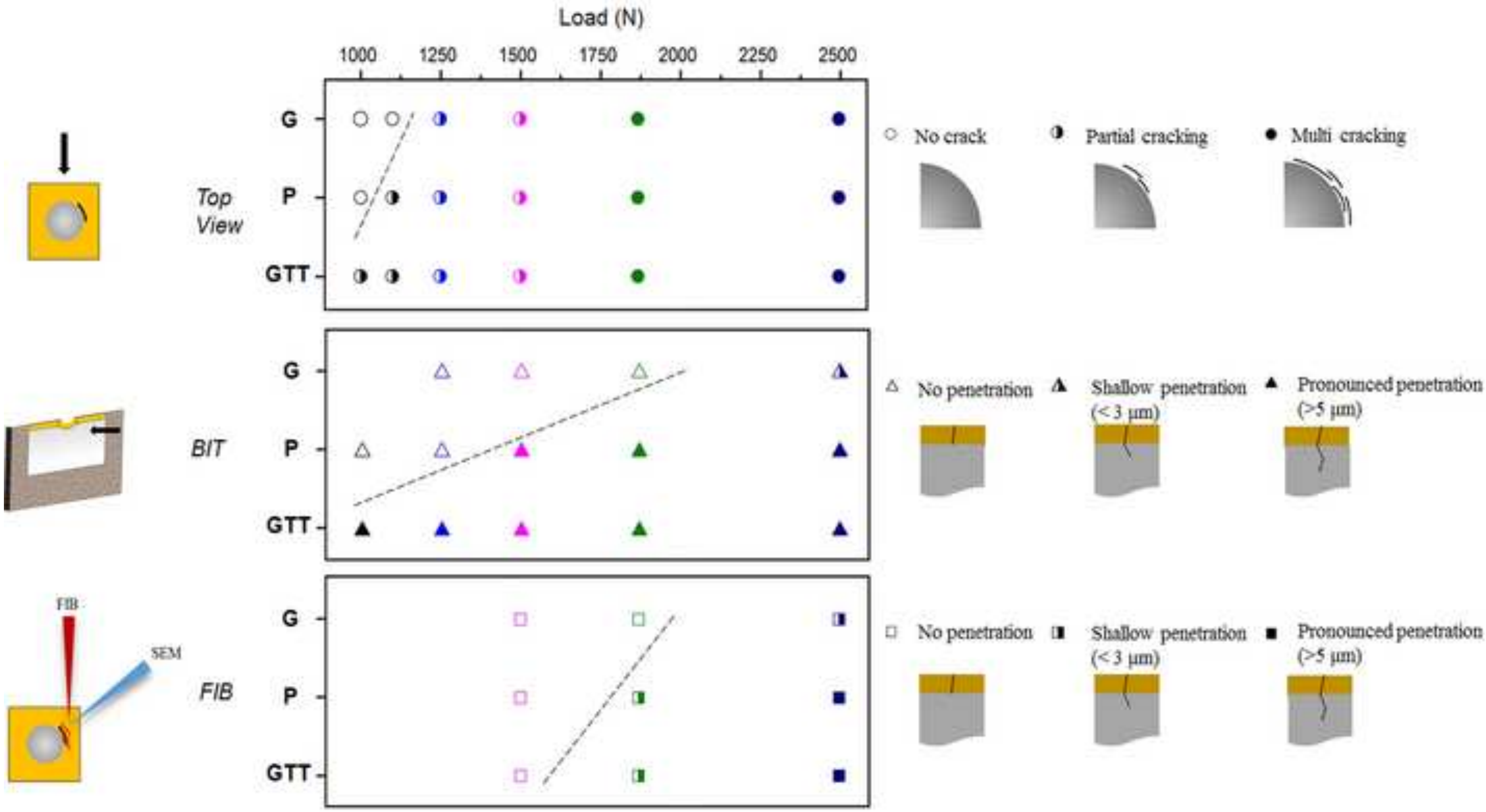

Figure 9a
Click here to download high resolution image

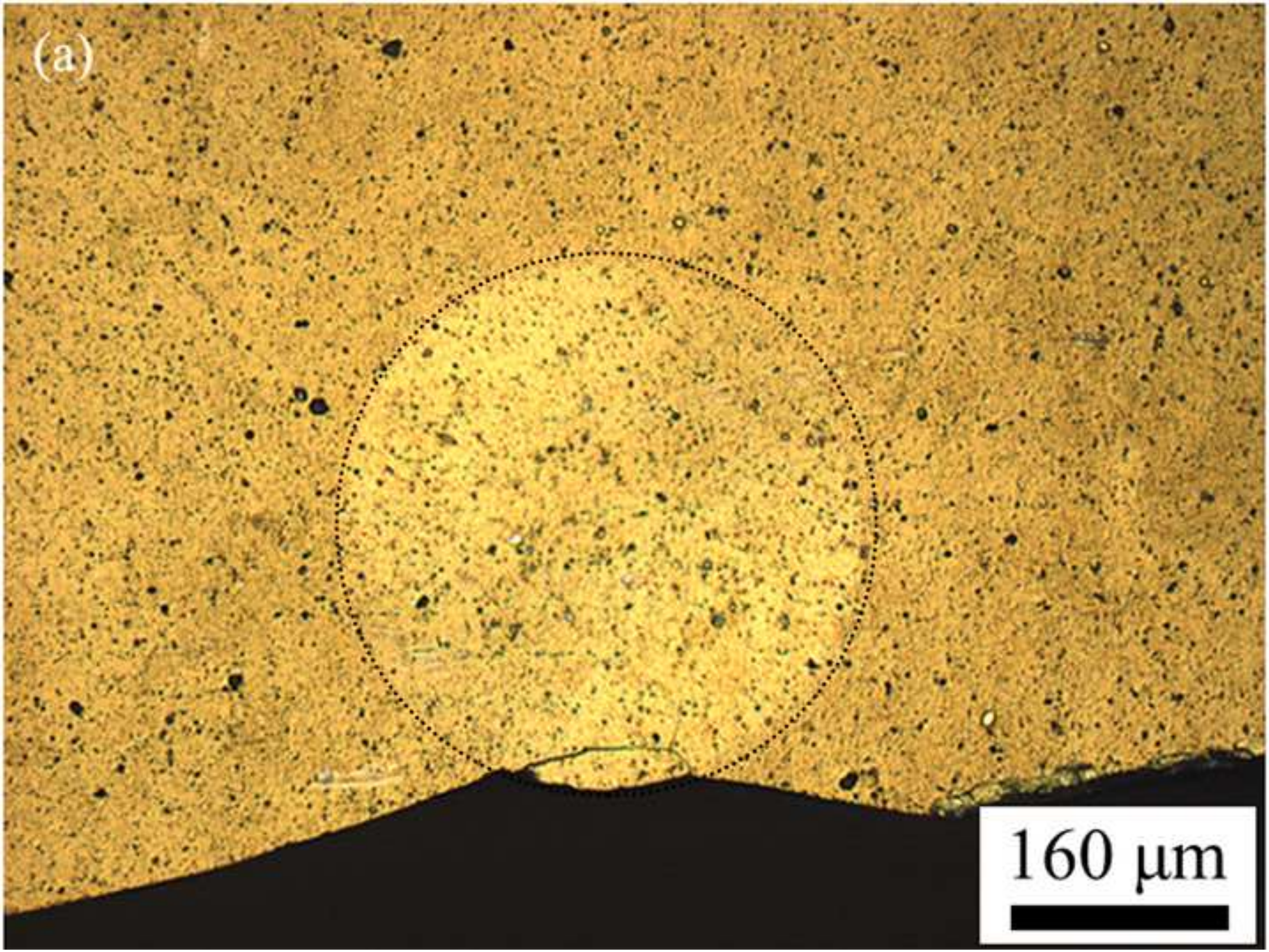

Figure 9b
Click here to download high resolution image