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Influence of the notch root radius on the fracture toughness of brittle metals: nanostructure tungsten alloy, a case study

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Abstract
Tungsten and tungsten-based alloys are considered as candidate materials for next generation nuclear fusion reactors. Unfortunately, its use for structural applications is compromised due to its inherent brittleness. In order to improve this crucial feature, it is necessary to accurately measure its fracture toughness as a real property, independent of geometrical parameters and the method used to introduce the induced crack. This is the objective of this work, where the notch tip radius influence on the fracture toughness of a brittle nanostructured tungsten alloy is analysed in depth.

Three point bending tests (TPB) were performed at room temperature on four types of notch geometries in which the notch root radius was gradually reduced. Notches were performed by four different methods: classical diamond disk, diamond wire, razor blade and ultra-short pulsed laser. Results obtained showed that single edge notched beam (SENB) specimens overestimate fracture toughness values and introduce some deformation on the notch root grains. However, razor blade (SEVNB), gives very good results with low dispersion, but is only suitable for coarse grain materials since size effect appears when the grain size decreases. Therefore, in a case of nanostructured materials as in this case, notch root radius is still too big (several times the grain size), requiring a new method to be implemented. This was solved that by producing very sharp single edge laser-notched beam (SELNB) specimens, 5-20 nm root radius, by ultra-short pulsed laser ablation. This method was previously used on ceramics but no evidence of its use was found on metals.

Introduction
Tungsten materials and alloys are considered relevant candidate materials for the fabrication of components for future fusion reactors. Due to its excellent thermo-physical properties (high melting point, high thermal conductivity, low thermal expansion, and low sputtering rates) they are suitable as plasma-facing materials (PFMs) but also for structural applications. Unfortunately, when considering as structural material, tungsten has some drawbacks; it is inherently brittle, even at operation temperature range where it should be ductile [1]. This brittleness makes essential to find or develop experimental mechanical testing methods where the poor fracture toughness of this materials can be determined. Not only that, the selected experimental method must supply a real determination of the fracture toughness as a material property, but not only as material parameter depending on the technique employed for the determination. As it will be shown later, the introduction of very sharp notches (similar to cracks) on specimens is essential for an accurate testing.

In metals, the most reliable way to initiate and grow controlled and reproducible cracks is through cyclic fatigue from straight notched specimens [2]. This method prevents from damage to the material and produces very small crack root radius, since a *real* crack is introduced. Despite this, it has certain restrictions when it refers to extremely brittle materials, as is the case of tungsten and its alloys. The main problem encountered is that the fatigue threshold stress is very close to the fracture threshold stress and define and control this limit level is very dependent on the specimen preparation. As a result, the probability of introducing reproducible sharp notches close to a crack without a high loss of material is reduced [3][4] and a more functional and reproducible alternative is required.

Over the years, numerous authors have studied alternative techniques to measure fracture toughness in ceramics and hard materials. For brittle metals, however, these methods are not as well established. A very usual and common method for the determination of fracture toughness in brittle materials is Indentation Microfracture (IM) [5]. Nevertheless, based on previous experience, the application of this method is not possible; tungsten is not brittle enough for crack growth from indentation corners, even at high loads (figure 1). The obtained result is often just chipping due to annular subsuperficial cracks from the plastic deformation around the indentation. So that, this method is not applicable to these materials

Another typical alternative applied on conductive materials is notching with Electro-Discharge Machining (EDM). Unfortunately, this process had to be discarded *a priori* because it induced quite extends thermal damage and modified significantly the microstructure **Error! Reference source not found.**.

In this study, four experimental methods (that gradually approach a crack-like notch) were compared in order to examine the influence of the notch root radius on the measured fracture toughness. Such influence can be a consequence of the interaction of a distorted stress field with material flaws in front of the notch [7].

Experimental Methods

A tungsten alloy was prepared following a powder metallurgy route of Mechanical Alloying (MA) and subsequent Hot Isostatic Pressing (HIP) describe elsewhere [8]. From this process a cylinder with dimensions 30 mm diameter and 50 mm length was obtained. This cylinder was then cut by refrigerated EDM to obtain bend bar specimens with nominal dimensions 1.6 x 1.6 x 25 mm^3. Microstructure of the alloy was examined in a Field Emission Scanning Electron Microscope (FE-SEM) from Zeiss (Germany) after the suitable polishing and etching with the solution (10 g KOH, 10 g $K_3Fe(CN)_6$ and 100 ml distilled water) during 7 s. From the microstructure (figure 2) and fracture surface analysis, bimodal distribution of grain size was observed with coarse grains around 2 μm and fine grains smaller than 0.1 μm.

To determine fracture toughness, Three-Point Bend (TPB) geometry was used due to the simplicity of the experimental device and the limitations of available material. Four types of methods were used to introduce notches in the TPB bar specimens.

The first method performed was the Single Edge Notched Beam (SENB) [9]. With it, notches were introduced in the specimens with a refrigerated diamond disk of 400 μm thickness at 1800 rpm. This is an easy, quick and low-cost method that has been very common during years in brittle metal and ceramics.

Other SENB specimens were produced by using a diamond wire. In this second method, the cutting speed was lower than in previous case, but the process was also simple and quick. The resulting notch radius was slightly reduced as a consequence of the 130 μm wire diameter used. A slightly smaller thread wire diameter can be employed, nevertheless, the feed rate will be greatly reduced as well as the lifespan of the wire; which will made the process too expensive and sluggish. In this case, the feed speed was controlled by carefully adjusting the force of the wire over the specimen. Additionally, special care must be taken in applying excessive pressure to the wire otherwise it will sag and produce a not straight notch. This effect should be avoided, since it can result in measurement inaccuracies of the fracture toughness.

The third method utilized a sharp industrial razor blade of 150 μm thick which was regularly impregnated in diamond paste of 1 μm grain size. Here, the razor blade wears the material because of the friction to create singe edge V-notched beam specimens [10]. To achieve these V-notches, a reciprocating cutting machine (that operates like a tribometer) was specially designed (figure 3). In this machine, the specimen is set in a clamp on a ground plate while the razor blade lays into a guide-way above. This upper part can move up and down to apply a controlled normal force against the mounted specimen. Whereas, the lower part moves linearly backwards and forwards with the adequate frequency selected by the operator. To obtain the best results, i.e. less time of preparation and the sharpest notch, several factors have to be considered:

- Material microstructure (e.g. porosity) and physical properties as elastic modulus, hardness and toughness vary for each material and strongly influence the rest of factors.

- An excessive reciprocating frequency on the linear movement may provoke a widening of the notch due to the vibrations of the machine or even the breakage of the material, whereas a deficient frequency increases process time.
- The normal force, together with frequency, is the most important factor. An overload leads to the breakage of the material or the razor blade, in contrast deficient force leads to an increase of time.
- The sharpness of the razor blade decreases over time, worn-out razor blades need to be changed several times during the operation, usually every 15-20 min.
- The diamond paste used as abrasive must be replenished several times throughout operation. At the first steps, diamond paste of 1 μm grain size was used. However, in the last change of razor blade it was proved that sharpest notch root radius were obtained when reducing diamond paste grain size and increasing its concentration.
- A proper cleaning between changes of razor blade is highly important to remove leftovers of material that may blunt the notch root radius or even stop the progress. Special care should be taken before the last step of the machining. Once the process is fully completed, specimens should be cleaned in an ultrasonic bath to remove any diamond grit still embedded in the notch tip.

The optimization of all these parameters for each material is a time-consuming process. In addition, once the best conditions are selected, the notching process may last one or two days.

Finally, the fourth method used was a femptosecond laser ablation to machine ultra-sharp notches on Three-Point Bending (TPB) specimens, so that the method was called Single Edge Laser-Notch Beam (SELNB) [11][12]. Prior to implementation, laser conditions were optimized to obtain the sharpest notch root radius together with minimum thermal damage. In optimal working conditions, as used in this case, thermal damage to the material, special mind when is a good conductor, is minimal or even non-existent. This is because the femptosecond laser is working in non-linear optical condition and the action of the laser transforms the material directly into plasma by an ablation process. So a transfer of heat energy as the solid is minimal.

Although the selection of the proper parameter of the pulsed laser needs some time, the process is less tedious than for previous method. In addition, once the parameters optimization is set, notching process is quite fast and the method is valid for similar materials. This fact is proved because we have already implemented this method for different W-based alloys with consistent results [13]**Error! Reference source not found.**.

After the notching process, TPB tests were performed on the notched bend bar specimens at room temperature. Tests were accomplished on a universal testing machine (Instron 3369, UK) using displacement control with a rate of 100 μm/min and a load span of 8.5 mm. Load and load point displacement were continuously monitored using a load cell and a liner variable differential transformer induction transducer, respectively. Four samples were tested for each type of notch to check dispersion of results. The corresponding fracture toughness values four each type of specimen were calculated by applying the stress intensity factor formula [15].

Results

Notches of SENB specimens performed with diamond disk and diamond wire, were far from being crack-like since root radius were 250 and 70-80 μm, respectively (figure 4). For the diamond disk case, as a consequence of the tool friction and pressure over the specimen during the cutting process, grains just below the notch root radius were seriously deformed (figure 5). This fact was quite surprising for a brittle material as this W alloy, although it can be also observed with smaller extension in the case of diamond wire (figure 6). Calculated fracture toughness values from these blunt SENB specimens were nearly the same, 6.1 ± 0.6 MPa·$m^{1/2}$ for diamond disk and 6.0 ± 0.7 MPa·$m^{1/2}$ for diamond wire.

In the case of SEVNB specimens produced with the razor blade, we obtained successful sharp V-notches with 5-7 μm notch root radius (figure 7). The obtained fracture toughness value significantly decreased by 13% to 5.2 ± 0.3 MPa·$m^{1/2}$. During the examination of the fracture surfaces, damage evidences as in the case of SENB specimens were not found. Adjacent grains look perfectly cut by the tool, however, notch root radius was still bigger than the alloy grain size (figure 8).

With the last method implemented, SELNB, we have achieved extraordinarily small notch root radius around 5-20 nm (figure 9), that can be considered similar to a real natural or induced crack. The obtained fracture toughness value was even reduced to 4.4 ± 0.1 MPa·m$^{1/2}$. We have considered this value the theoretical limit of the fracture toughness for this material according to the relation exposed by Damani *et al.* [7]. This relation said that if the notch root radius is close to infinitesimal (nanometrical) values, experimental fracture toughness is approximately equal to theoretical. Some authors reported for this method applied in ceramics an affected zone with microcraks and pores in the vicinity of the laser notch [16]. They added then the length of the affected area to the initial value measured for the calculation of fracture toughness. This postulate, however, could not be correct since the influence of the damage zone can increase the fracture toughness as we have previously shown. Here, as the laser ablation was perfectly optimized after a strong analysis of the cutting parameters [11][12] evidences of this affected zone were not found (figure 10). Additionally, the higher thermal conductivity of tungsten in comparison to ceramics could be a determining factor to avoid that. Further evidence of material without thermal affected zone, is that the initial notch length measured on the side and the final from fracture surfaces have the same values.

The analysis of the fracture toughness values revealed not only a decrease in the obtained results, but in the dispersion as well. This reduction of results scattering that goes to 10%, 6% and 2% of the values for SENB, SEVNB and SELNB, respectively, matches with the notch root radius progressive sharpening. It is attributed t the fact that the SELNB method is less invasive and harmful for the material, therefore results become more repetitive.

Discussion

Diamond disk and diamond wire are simple and economic notching tools to produce SENB specimens. However, significant drawbacks as large notch root radius and damage of the adjacent grains lead to an overestimation of the fracture toughness values close to 40% (figure 11) and high dispersion of results. These reasons make SENB methods only suitable for rough fracture toughness evaluation, but not for a fine-tuning of the material.

In the case of the razor blade, its efficiency is well established and verified among ceramics [3][10][17][18][19][20][21], but few authors have applied the method on metals [22][23], especially in tungsten materials and alloys. The success of the method in ceramics is attributed to the fact that they are more abrasive than the metallic blade and exhibit higher hardness. Consequently, during notching, the blade wore away with the abrasive diamond paste causing progressive sharpening of the blade, which generates a notch tip radius around a few microns.

In metals, however, materials in contact have a wear resistance of the same order of magnitude. This fact cause an increase wearing of the blade and consequently of the notch root radius [23]. In this method, precise control of the razor blade is difficult, but we achieved low dispersion of fracture toughness results and a slightly decreased overestimation in the results around 20% (figure 11). Furthermore, the use of this method for notching metals such as W materials and alloys becomes time consuming and tedious.

Nevertheless, for coarse grain ceramics, SEVNB method can be used to determine a fracture toughness value quite close to the real, in agreement with the SEPB data [11][18]. But in the case of nanostructured materials as is the case, the SEVNB method overestimates values of fracture toughness, since the critical notch root radius should be of the same size as the relevant microstructural features [7][20].

So that, the introduction of the notch by ultra-short laser ablation (SELNB), exhibits obvious advantages such as high accuracy and speed, good reproducibility and precision for reliable fracture toughness testing as in the case of structural ceramics studied [11][12][24]. The produced notches are to all intents like a real crack.

Conclusions

The effect of the notch root radius of a nanostructure tungsten alloy was analysed on its fracture toughness results. To that end, notches with different tools (diamond disk, diamond wire, razor blade and ultra-short pulse laser) were introduced on bend bar specimens and tested at room temperature by TPB tests. Results obtained reveal a very high dependence of the notch root radius on the fracture toughness values. For that reason, obtained values normally used to determine fracture toughness, cannot be considered as a real property of the material, if not only a parameter depending on the experimental test method. As a result of the exhaustive analysis, the following stamens can be settled:

- SENB specimens are adequate methods just for rough evaluations, since they overestimate fracture toughness results around 40%. This is due to the rough geometry of the notch tip radius but it is also caused by the damage introduced in the rear part of the notch tip (severe plastic deformation induced by the cutting process).
- SEVNB specimens provide closer results, 20% higher, but notch root radius is still bigger than grain size.
- SELNB specimens obtained from laser notch ablation are considered to provide the real fracture toughness values. Notch root radius has the same order of size than a real crack and bellow alloy nanostructure.

Although the SELNB method involves higher costs, it provides an alternative for an accurate measure of the fracture toughness. As a result, this proposed method produces high quality reproducible notches with no damage on the rear of the notch root radius and little dispersion of results. Despite this, knowledge of the material microstructure is essential to evaluate the more efficient method. Materials with coarse microstructure do not require the use of more expensive advance techniques as laser ablation, since a method as the razor blade is enough to provide real fracture toughness values.

Acknowledgements

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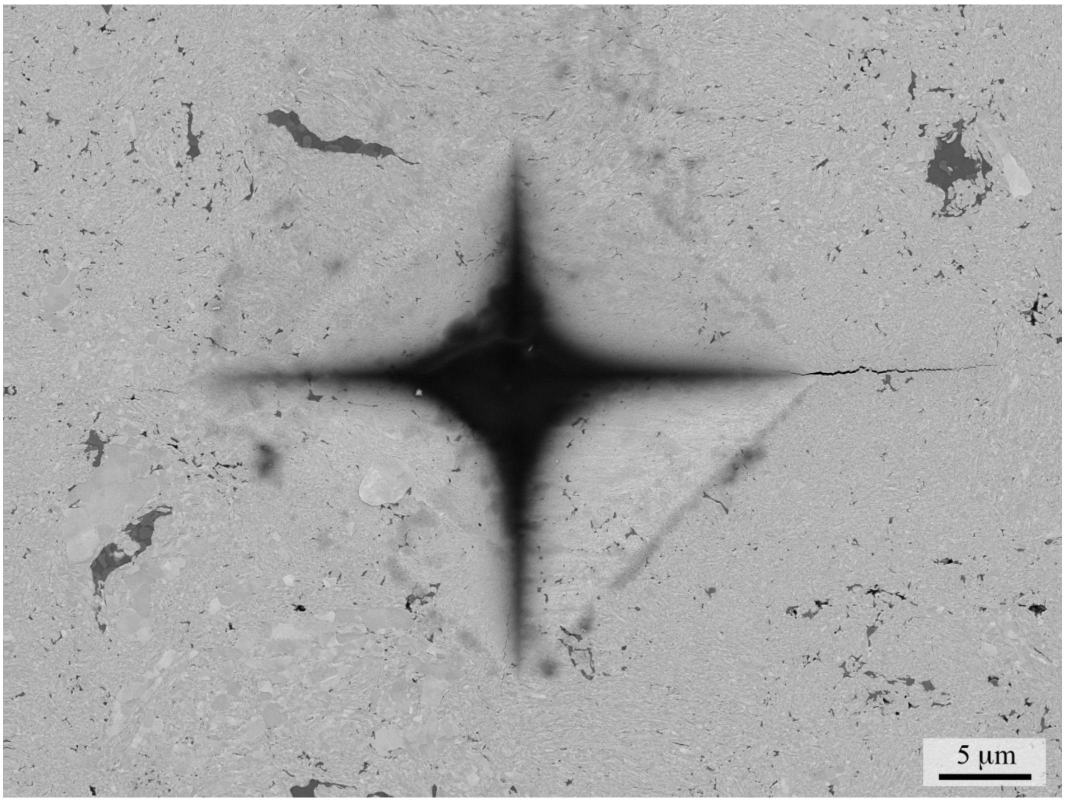

Fig. 1 Vickers microindentation with a load of 9.8 N. IM cannot be applied since cracks only grow from some corners and have different length.

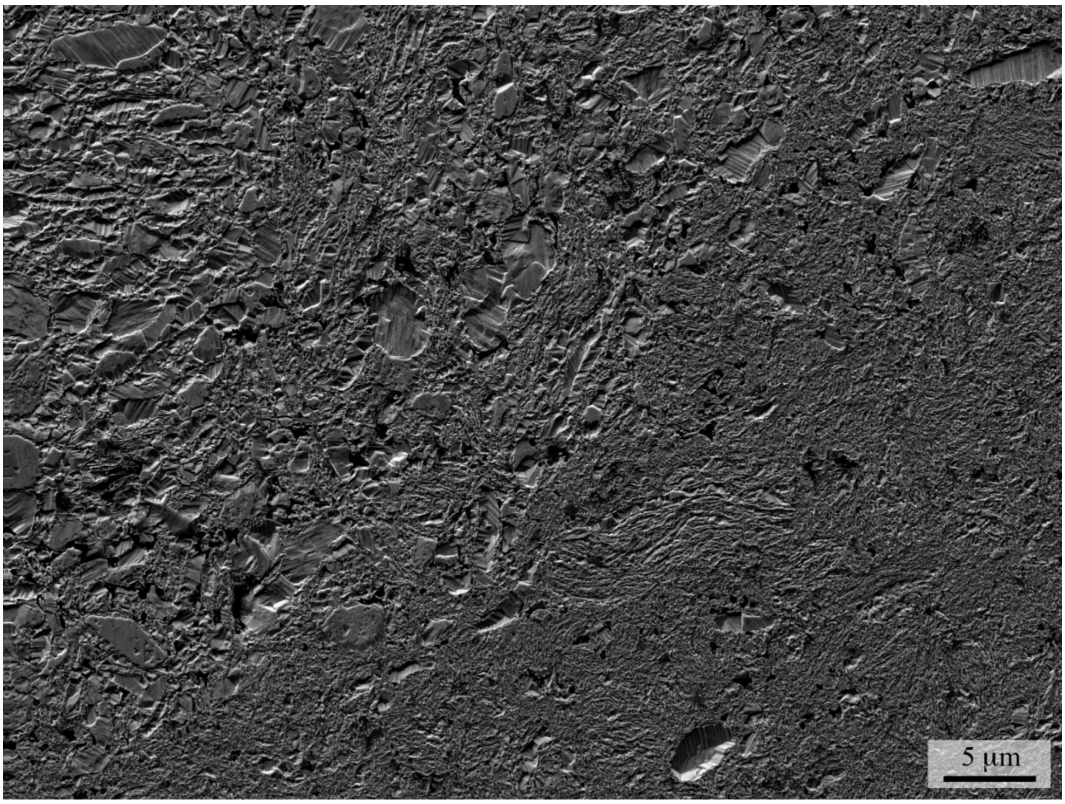

Fig. 2 Microstructure of the nanostructure W-alloy

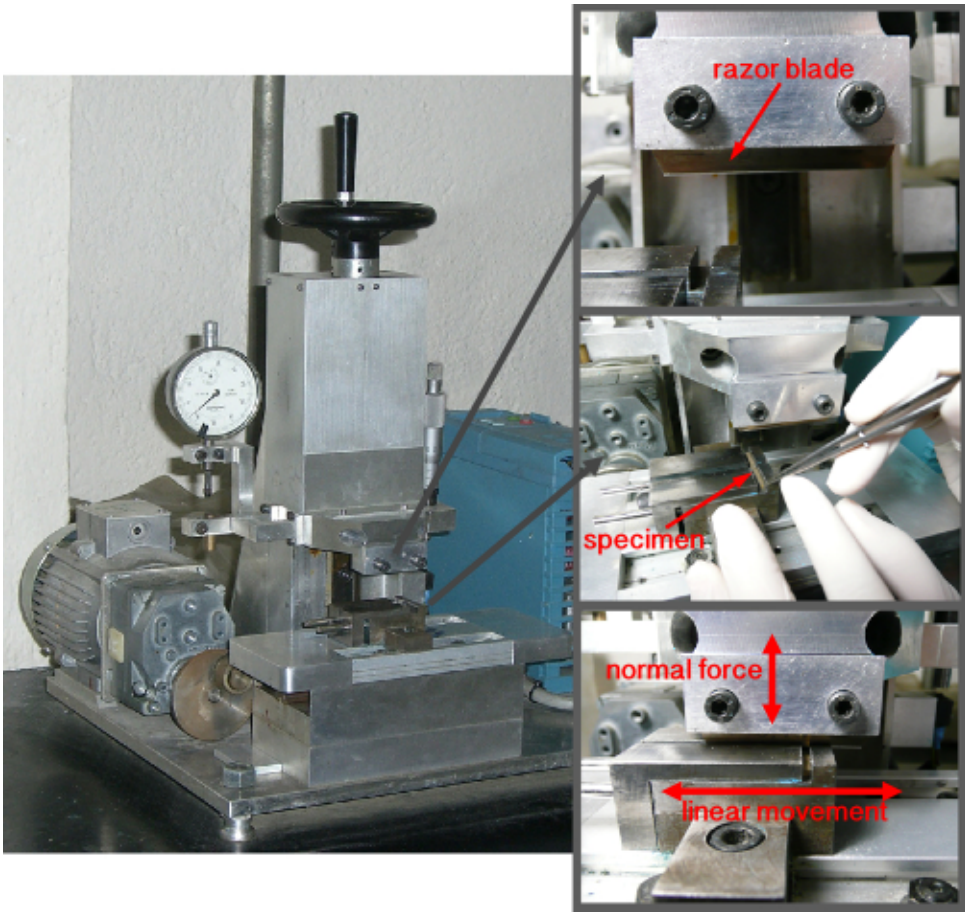

Fig. 3 Razor blade machine to create SEVNB specimens. Details of the position of the razor blade and the specimen are shown on the right side.

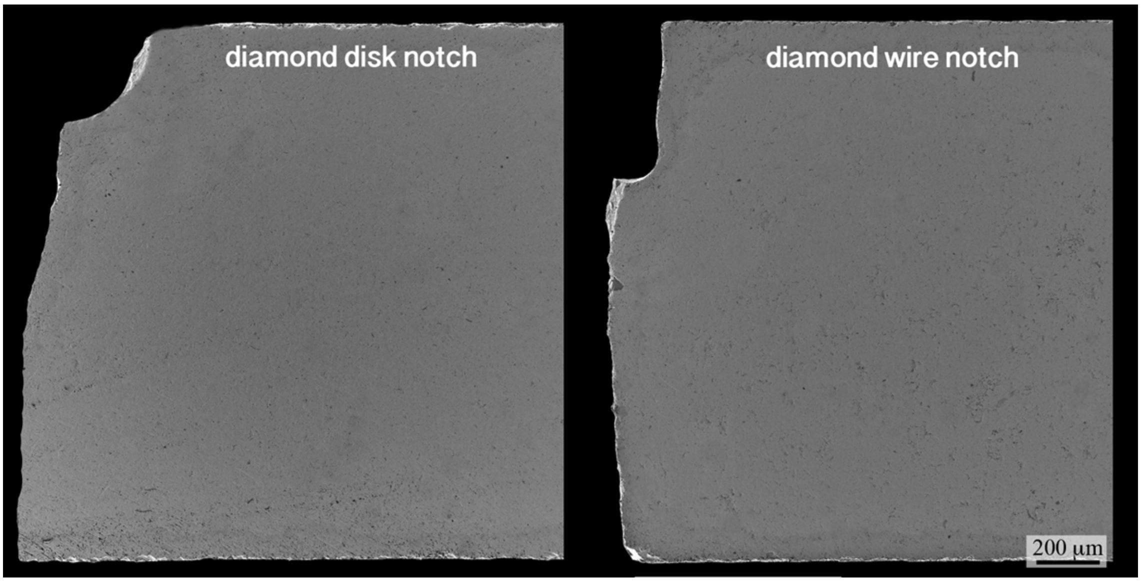

Fig. 4 Sections of the SENB specimens after TPB tests of notches performed with diamond disk and diamond wire.

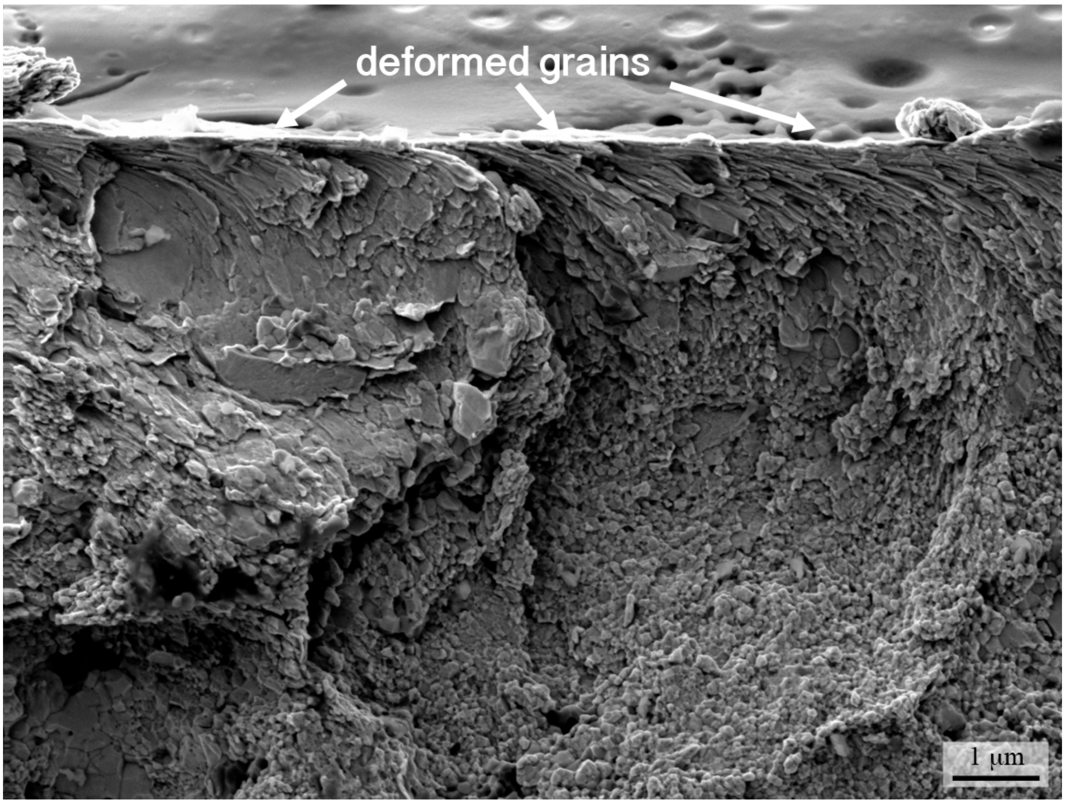

Fig. 5 FE-SEM fracture surface of the SENB specimen performed with diamond disk after TPB tests. Evidence of deformation around the notch root radius is observed.

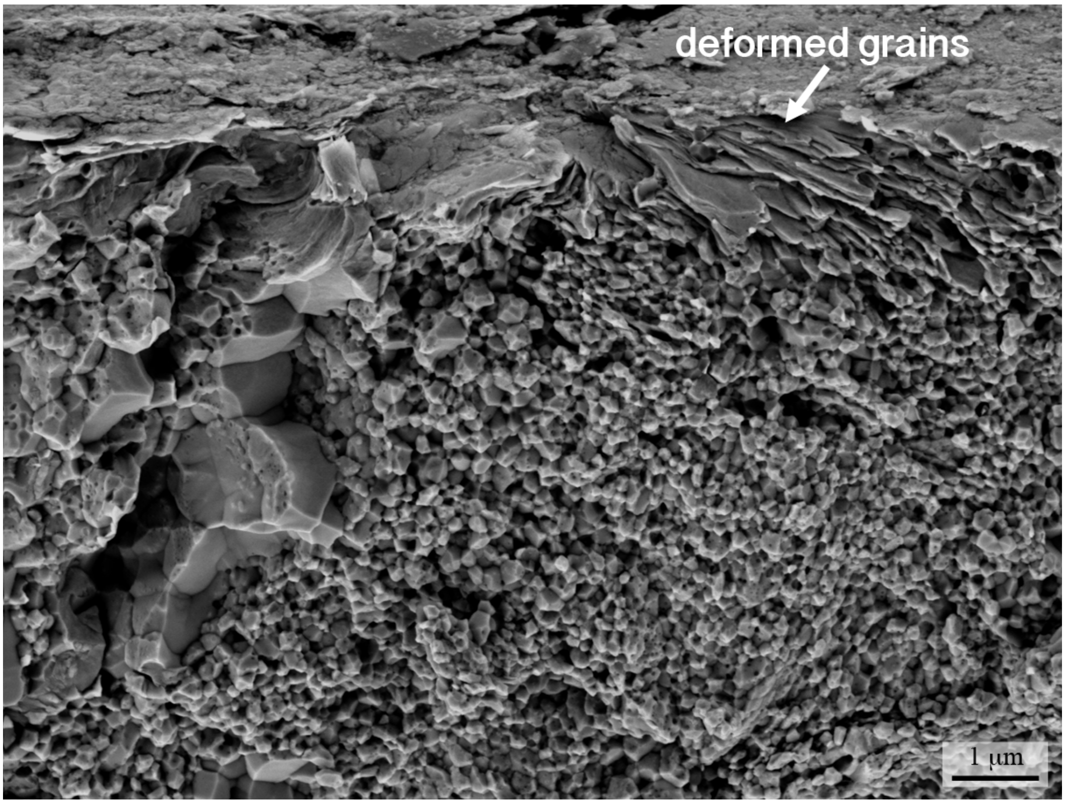

Fig. 6 FE-SEM fracture surface of the SENB specimen performed with diamond wire after TPB tests. Evidence of deformation around the notch root radius is observed.

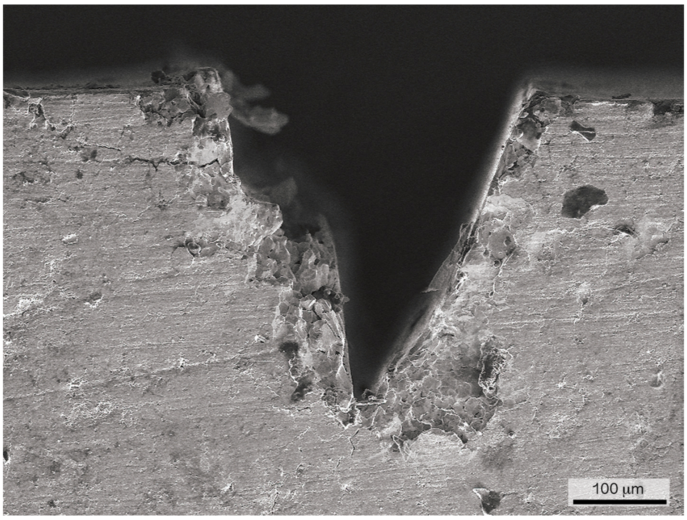

Fig. 7 SEVNB specimen performed with the razor blade.

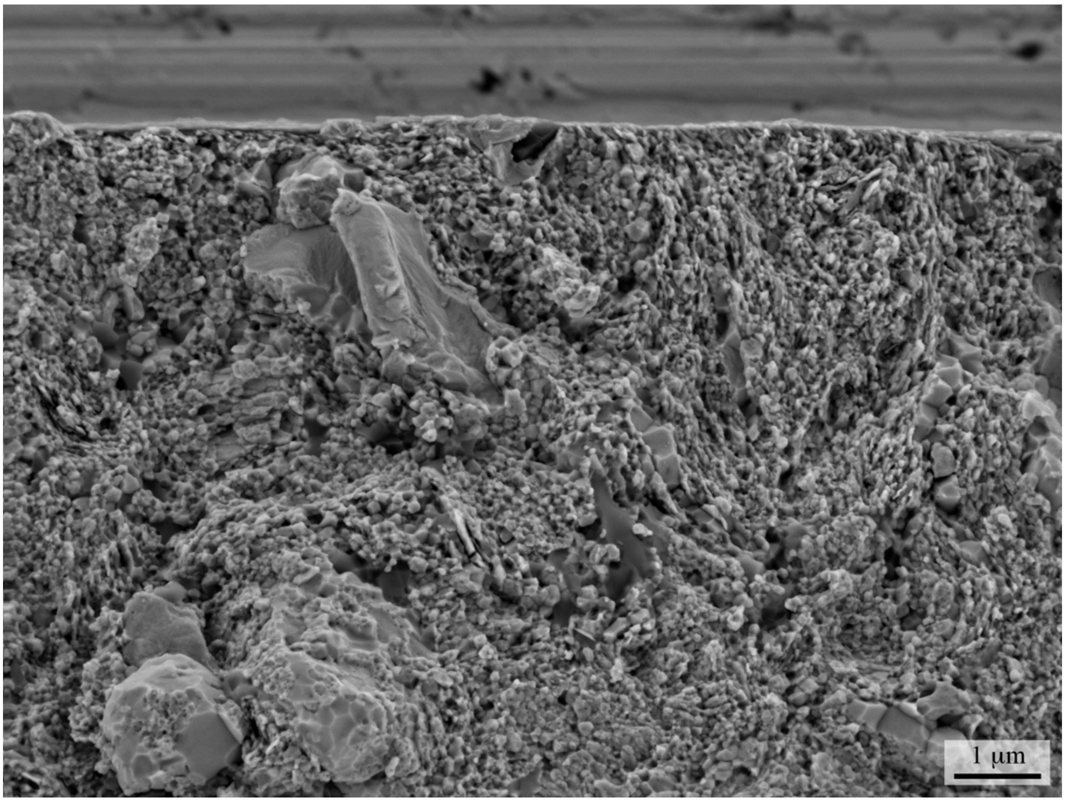

Fig. 8 FE-SEM fracture surface of a SEVNB specimen performed with the razor blade after TPB test.

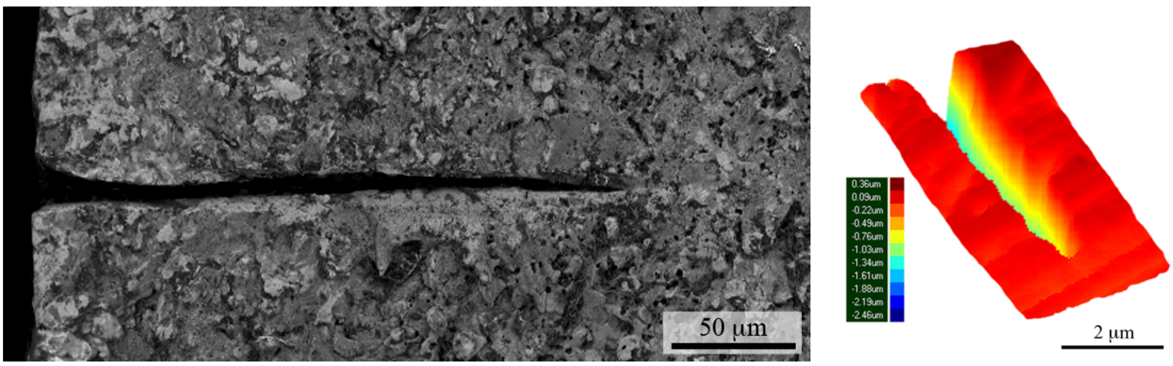

Fig. 9 FE-SEM image of a femtosecond laser notch (left) along with a profilometer image of its notch root radius (right) [13].

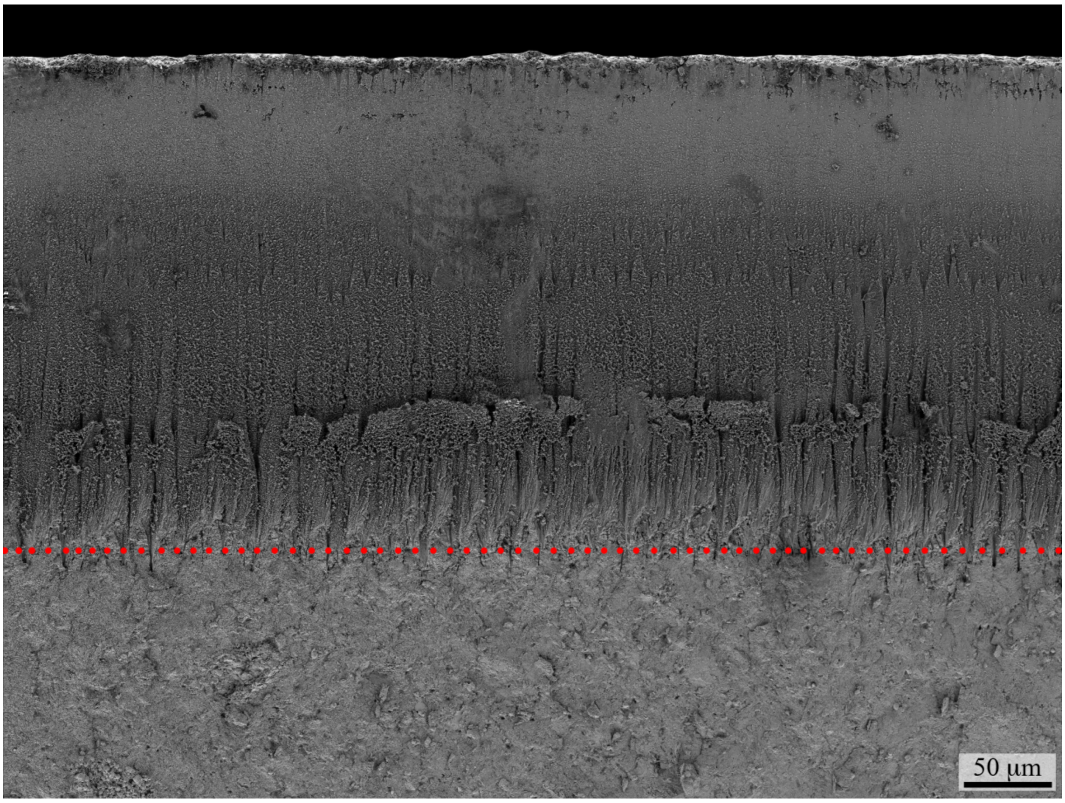

Fig. 10 FE-SEM fracture surface of a laser notched specimen performed after TPB test.

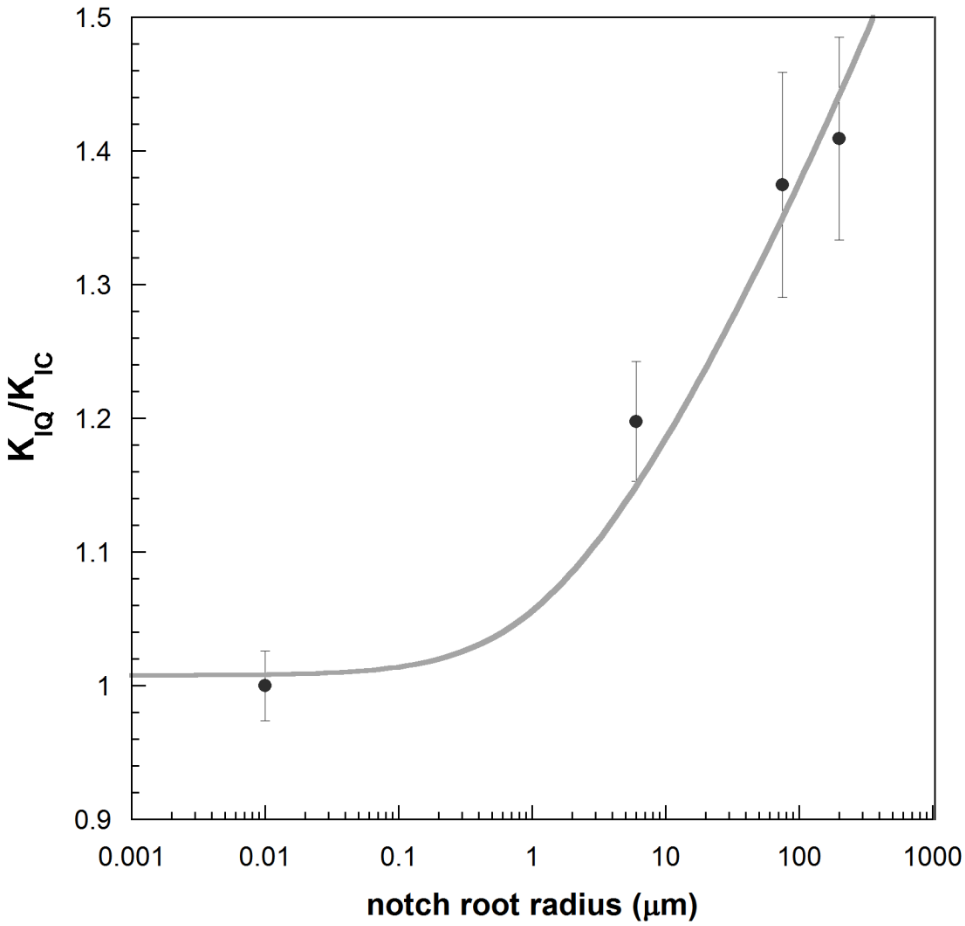

Fig. 11 Comparative of the effect of the notch root radius on the measured fracture toughness (K_{IQ}). Results obtained from laser notched specimens are considered the real fracture toughness (K_{IC}) values for the normalization of results.