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Fast Fabrication of Micropillars for Micromechanical Testing Using a Combined fs-Laser – FIB/SEM Approach

Tim Schubert[1*], Fang Zhou[2], Renato Pero[3], Nicolas Randall[3], Timo Bernthaler[1], Gerhard Schneider[1]

1. Aalen University – Materials Research Institute, Aalen, Baden-Wuerttemberg, Germany.
2. Carl Zeiss Microscopy GmbH, Oberkochen, Baden-Wuerttemberg, Germany.
3. Alemnis AG, Thun, Switzerland.
* Corresponding author: Tim.Schubert@hs-aalen.de

To determine bulk material properties of a large variety of construction and functional materials mechanical testing on cylindrical samples is a long-established method. However, mechanical properties determined on the micro-scale are much more sensitive to local microstructural changes as well as surface effects and therefore are becoming a more desired testing method especially in material and process development.
The bulk testing method with cylindrical specimen can relatively easy be transferred to the micro-scale by the preparation of small mostly cylindrical pillars and following tensile or compression testing also under different temperatures [1].
The most common way to prepare such micropillars is via focused ion beam (FIB) machining but this method is very time consuming and thus inefficient for preparation of larger pillars and also a larger quantity of pillars for statistical rigor [2].
The presented work shows a new approach using a Femtosecond Laser mounted to a standard FIB/SEM machine to prepare a large quantity of micropillars in Tungsten carbide hard metal (WC-Co). Each pillar has a diameter of 25 µm and a height of 70 µm and provides a defect free surface. The combined (Laser + FIB) preparation time per pillar was 3 hours.
In comparison preparation time for a single pillar of these dimensions with FIB alone is calculated to be >12 days which gives an estimated time saving of 98.9% when fs-Laser preparation is used.
Figure 1 left shows an array of 20 pillars after fs-Laser preparation and surface cleaning before FIB finishing. The whole array took around 25 min to prepare and consists of pre-shaped pillars with 50 µm diameter and 70 µm height. The free area around each pillar for tool access has a diameter of around 700 µm. Figure 1 right shows an exemplary pillar after final FIB polishing with 25 µm diameter and 70 µm height. On the pillar surface the WC-Co microstructure can be clearly seen which indicates non-influenced material due to processing and therefore reliable results in following micromechanical testing.

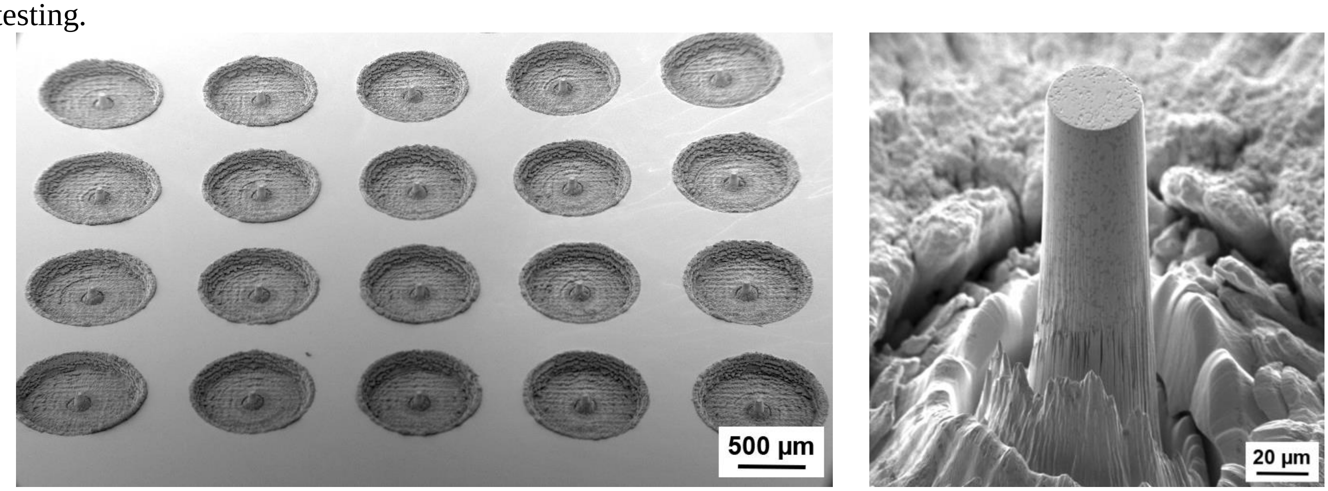

Figure 1. Left: 5x4 pillar array in WC-Co after fs-Laser preparation, preparation time ~25 min; right: single pillar after final FIB polishing, Ø25 µm x 70 µm.

The whole preparation procedure was conducted in a 4-step-process where the first step is to prepare the sample surface mechanically and apply a protective coating afterwards to easily get rid of redeposited material after laser ablation.
As the micromechanical test-device needs an even contact area to the sample material, the very first step was to mechanically grind and polish the surface with standard materialographic preparation methods. Afterwards the polished surface was evenly coated with an ethanol soluble lacquer. This layer was applied so any redeposited material from the laser process could be easily removed just by ultrasonic solvent cleaning. In figure 2 a comparison of a single pillar just after fs-Laser ablation (left) and after cleaning (right) is given. In the cleaned state the pillars are ready for final FIB-polishing.

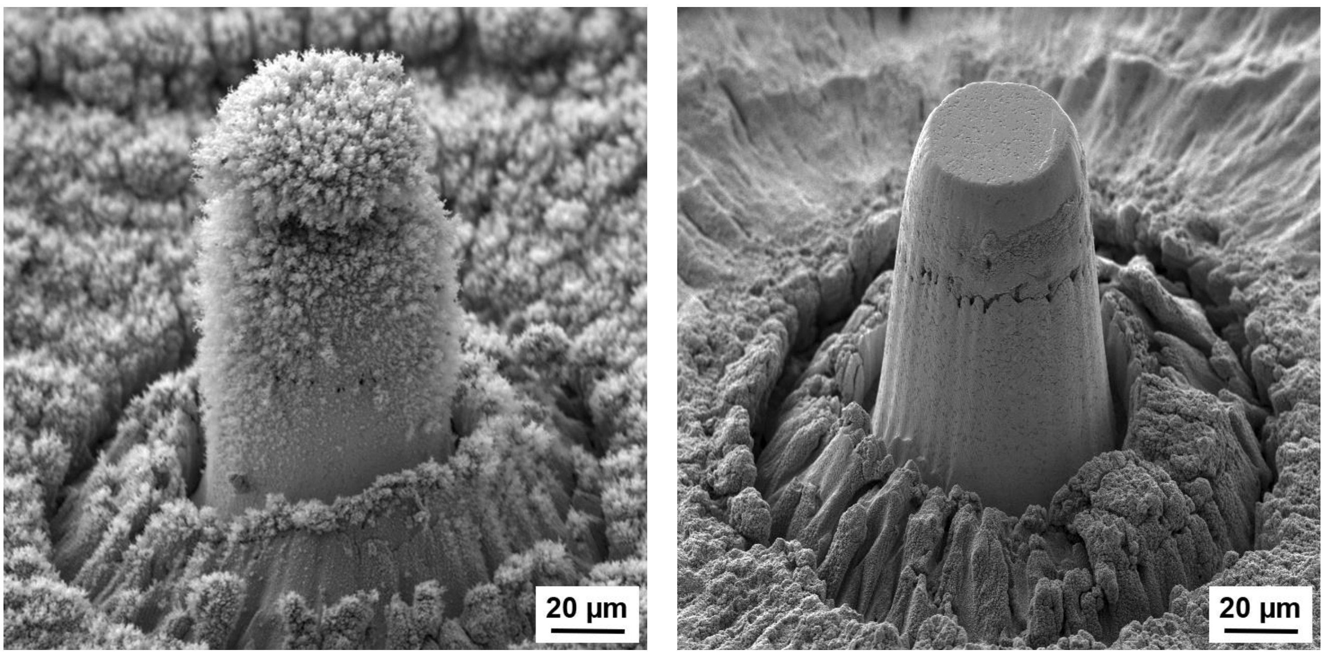

Figure 2. Left: Single pillar after fs-Laser ablation with lots of redeposited material; right: Pillar after ultrasonic solvent cleaning, all redepo is removed.

The next crucial step is to find the right laser recipe with high removal rates and minimal thermal influence on the material. To obtain a good recipe, extensive tests on spare sample material had to be done with varying laser parameters such as power, scanning speed and frequency and also different geometries with respective hatching patterns (e.g. shape, hatch distance, hatch repetitions, hatch rotation). The best suited geometry and hatching was found to be a 2-way pattern where a 700 µm round shape with a 100 µm circular center is filled with parallel hatch lines. This pattern is the repeated 20 times with 19° rotation between each hatch. The second pattern consists of concentric circles with 4 µm distance covering a diameter of 100 µm with 50 µm diameter residual material in the center. The concentric pattern is repeated 15 times. The applied pattern is shown in figure 3 left. Regarding the laser parameters the first outer pattern is exposed with 50% power, 17 kHz frequency and 300 mm/s scanning speed. The inner concentric geometry is exposed with 25% power, 1 kHz frequency and 2 mm/s

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scanning speed.

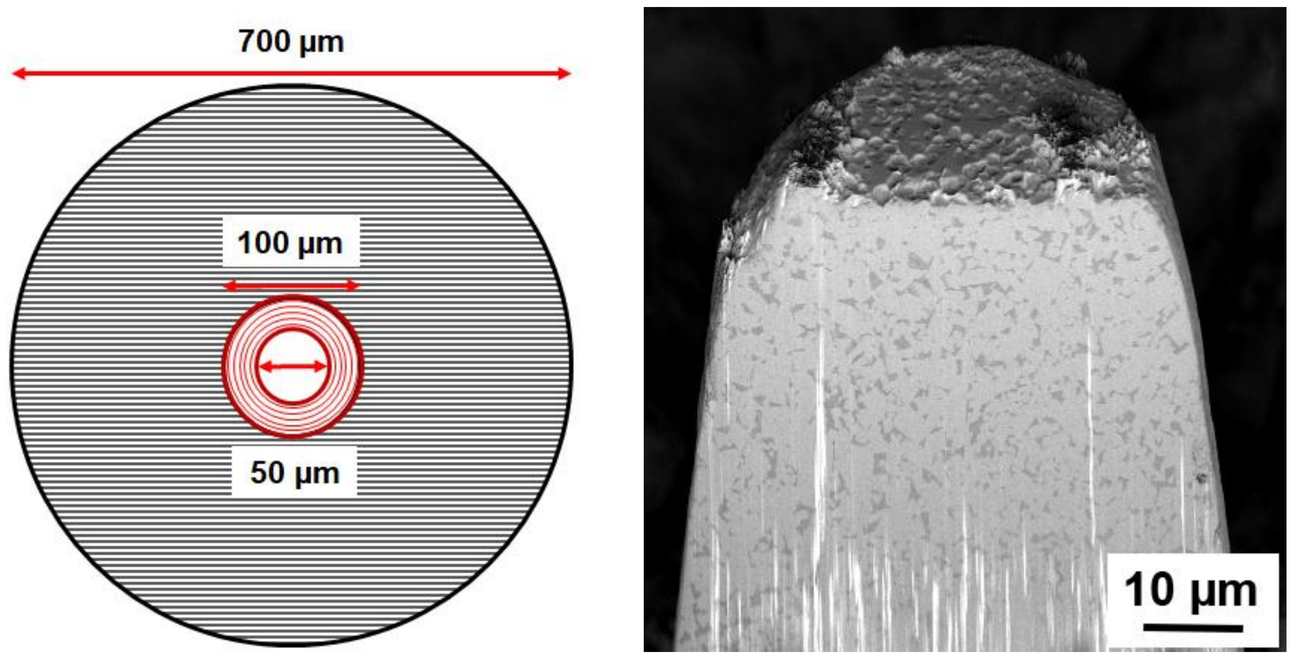

Figure 3. Left: 2-way pattern for fs-Laser ablation of micropillars, outer area filled with linear hatch, inner portion with concentric circles; right: FIB cross section through pillar in laser prepared state, no surface heat induced damage visible in WC-Co microstructure.

To make sure that the applied laser parameters do not alter the microstructure of the investigated material and thus lead to false mechanical properties, a FIB cross section was prepared through the middle of one pillar. Figure 3 right shows the prepared cross section. Over the whole cross section, the typical WC-Co 2-phase microstructure consisting of polygonal WC particles (light grey) in a Co matrix (dark grey) can be observed. The unchanged microstructure indicates that no thermal influence of the laser ablation occurred.
After finding the suitable laser recipe a pattern of 4x5 pillars was manufactured in 25 minutes. The next step then is to thoroughly clean the surface in preparation for FIB polishing (see figure 2).

To further reduce the pillar diameter and obtain perpendicular cylinders, the 4th and last step in the preparation process was FIB milling. In the case of the presented experiments, a 3 nA FIB polishing step for ~2.75 h per pillar was found sufficient for good shape and surface quality. Figure 4 shows the top of an exemplary finalized pillar. The shape is perpendicular and the top as well as the side surfaces clearly show the WC-Co microstructure without any artefacts that might hinder the micromechanical testing that will follow.

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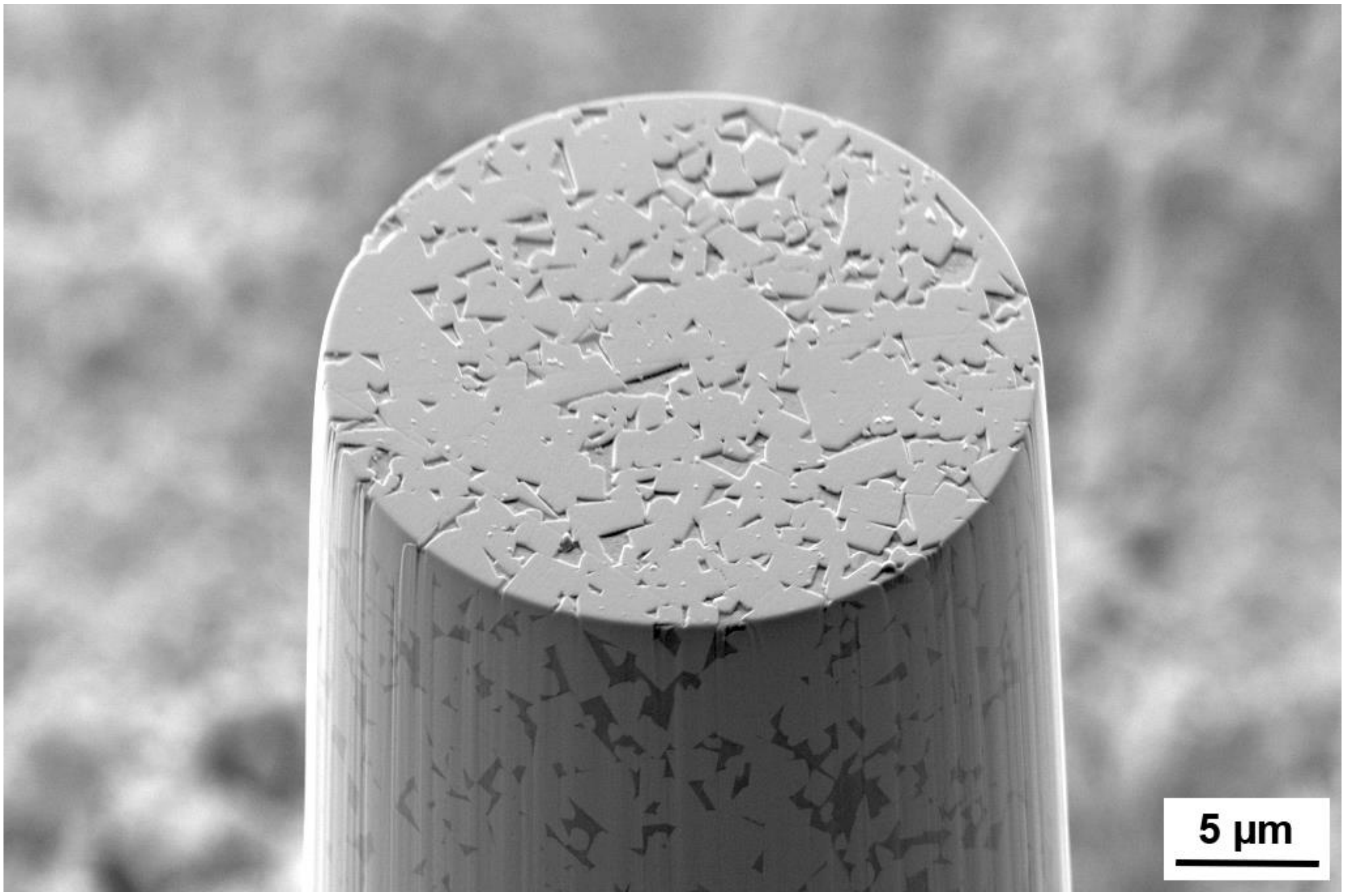

Figure 4. Final pillar quality, perpendicular shape, excellent surface quality, time efficient preparation.

The presented work shows a very convenient way to overcome the issues of time-consuming pillar preparation for highly desired micromechanical testing with focused ion beam preparation alone. The preparation time for 20 pillars in WC-Co hard metal with 25 µm diameter and 70 µm height could be reduced by >98% by applying a fs-Laser pre-preparation step without affecting the microstructure.

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