V4 Japan project

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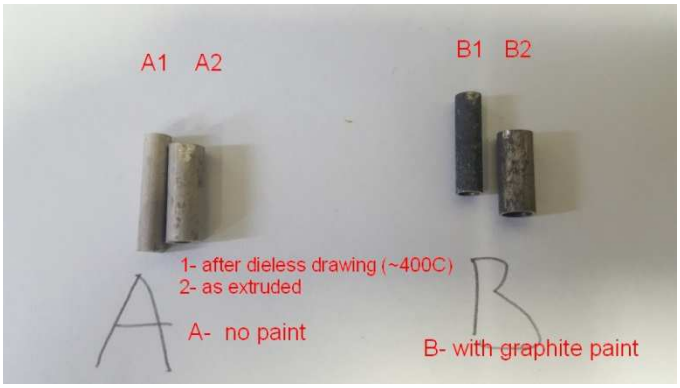
Report on AZ31 tubes (light miscroscopy, tube dimensions, grain size)

1. Samples and methodology

Four specimens were characterized in terms of light microscopy and subsequent grain size and shape analysis was made using an ImageJ program. As-extruded and laser dieless drawing (LDD) processed AZ31 tubes were characterized. Graphite paint was applied to the tubes in order to homogenize and enhance the thermal efficiency of laser induced heating of the tubes. To observe the effect of the graphite paint, samples without application of this paint, processed with the same parameters, were analyzed. The nomenclature was established as follows: **A1** – LDD (not painted), **A2** – as-extruded (not painted), **B1** – LDD (graphite painted), **B2** – as-extruded (graphite painted).



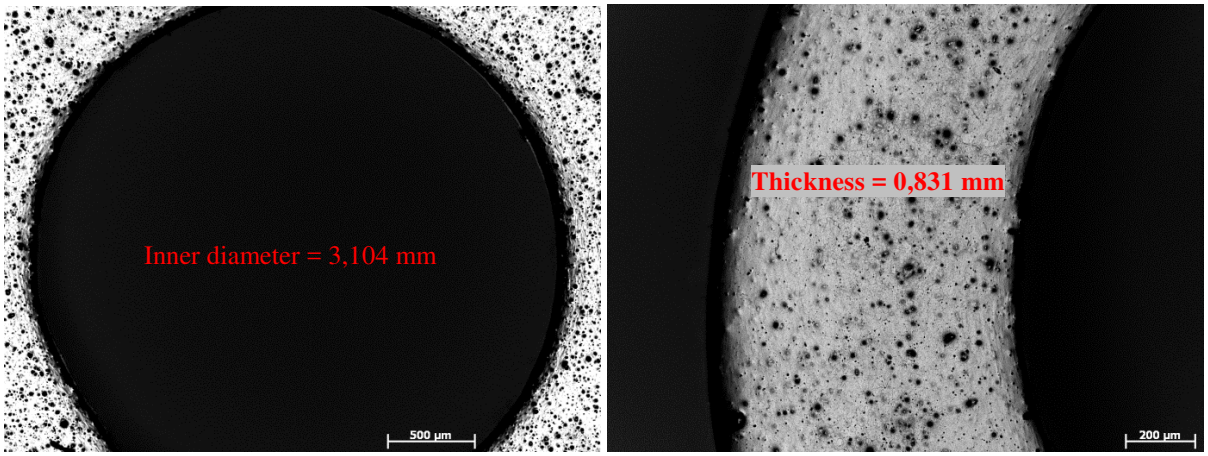
For the purpose of the light microscopy a Zeiss Axio Observer D1m microscope was used. The samples were cut in two plains: i) **perpendicularly to the axial direction (RD-RD plane)** and ii) **parallel to the axial direction (AD-RD plane)**, where AD and RD stands for axial and radial direction respectively. These samples were then mechanically grinded using a SiC paper with P2000 grit. Thereafter, 3 µm and 1 µm polycrystalline diamond suspensions were used for the mechanical polishing steps. As the last step, a mechanical-chemical polishing using Struers OP-S solution was carried out. The polished surfaces were afterwards etched using 8 vol.% water solution of HNO_3 in order to obtain distinctive grain boundary morphology.

For each plane (AD-RD and RD-RD) of each sample a minimum of 10 images were obtained, distributed across all available magnifications. From these images, a total of 4 images for each sample were chosen which provided the best possible detail for subsequent tube dimension analysis. The analysis itself consisted of fitting an inner circle and two circle segments for the determination of RD-RD dimensions, regarding the tube diameter and its thickness respectively. AD-RD tube dimensions were determined in a same manner with the usage of two parallel lines. In case of precise fitting, for instance with the least-square method, the standard deviation would be given. In case of these images, and for the required precision, the fitting of geometrical shapes was done by using naked eye and Corel software. This approach was determined to be sufficient. Therefore, there is no standard deviation given for values shown in the images. Since the inner diameter inconsistency (AD-RD plane) can arise significantly by not precisely cutting in the position in the middle of the tube, only standard deviation values of the tube thickness are presented and considered.

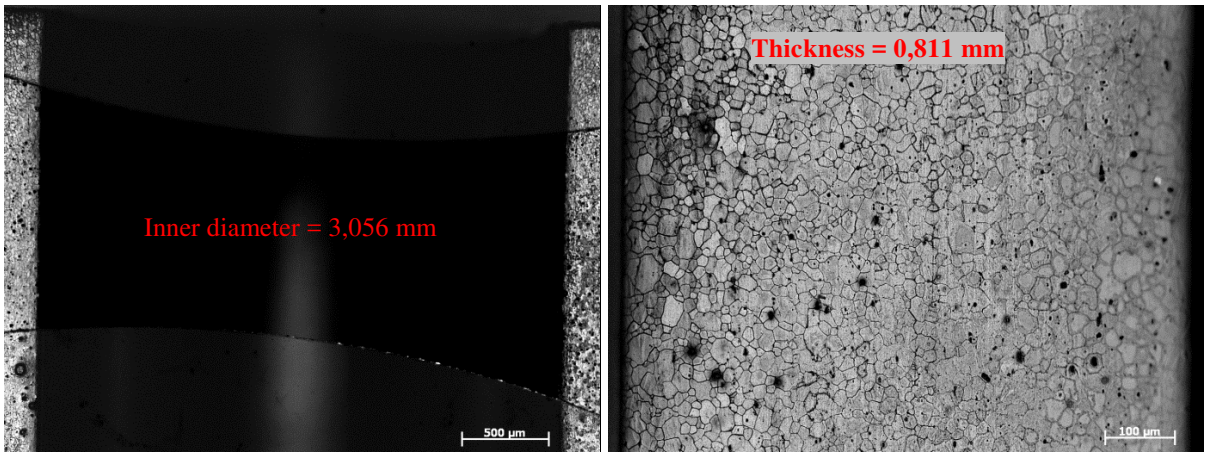
2. Tube dimensions

A2 – as-extruded (not painted)

RD-RD plane



AD-RD plane

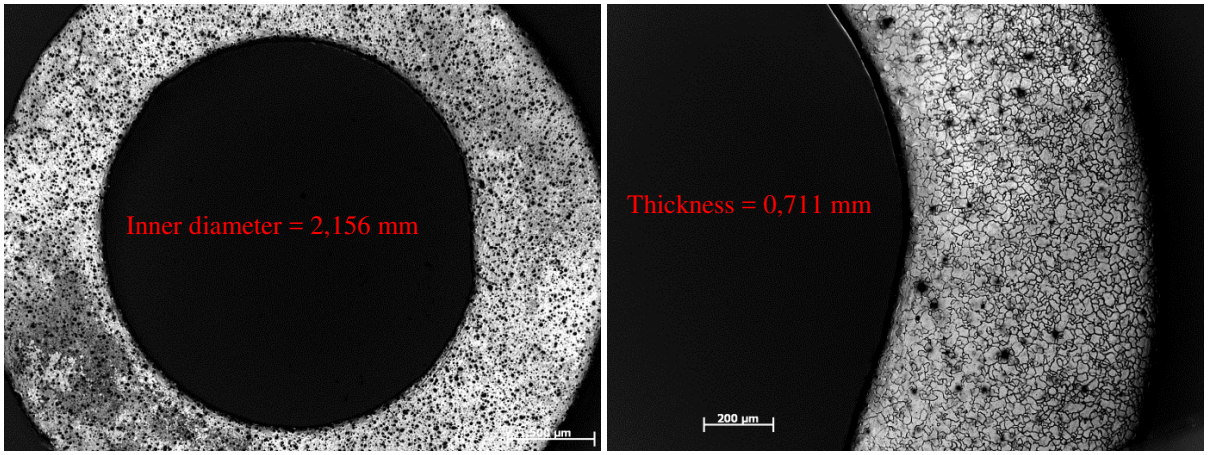


A2 Average inner diameter = **3.104 mm**

A2 Average tube thickness = (**0.821±0.010) mm**

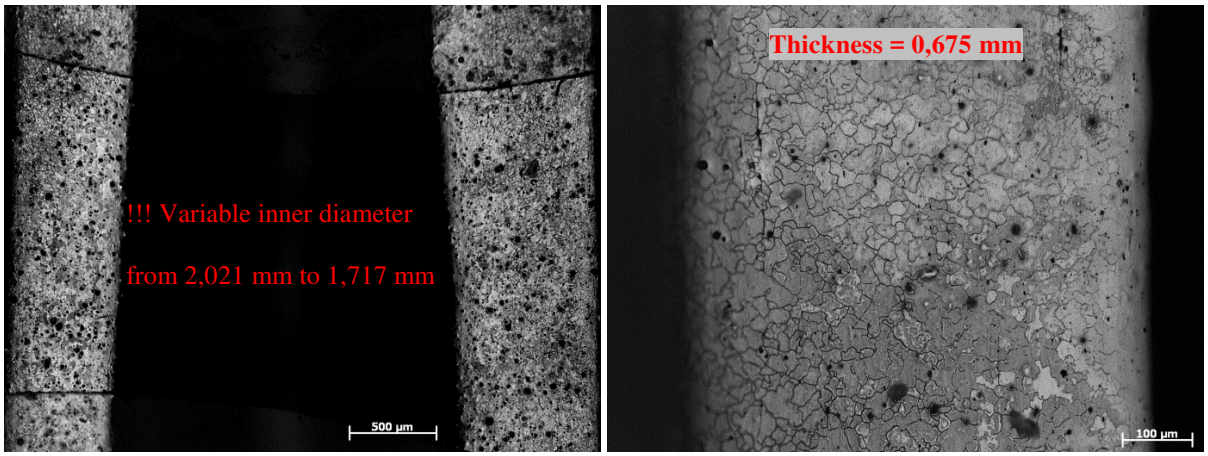
A1 – LDD (not painted)

RD-RD plane



AD-RD plane

AD-RD plane inner diameter calculated as an average value of the maximal and minimal value.

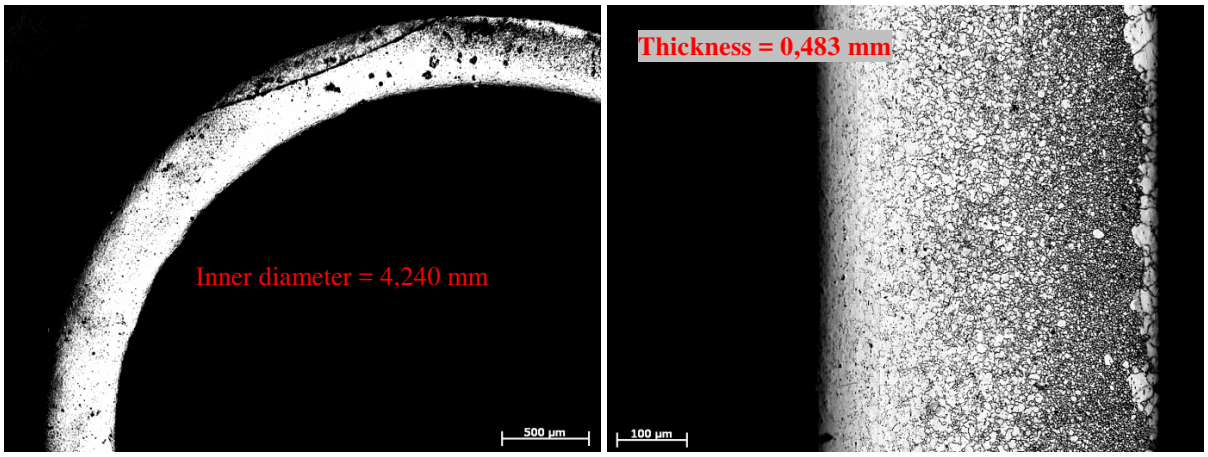


A1 Average inner diameter = **2.156 mm**

A1 Average tube thickness = (**0.693±0.018) mm**

B2 – as-extruded (graphite painted)

The tube dimensions were too large to measure them using the same approach as before. Only one value is considered.

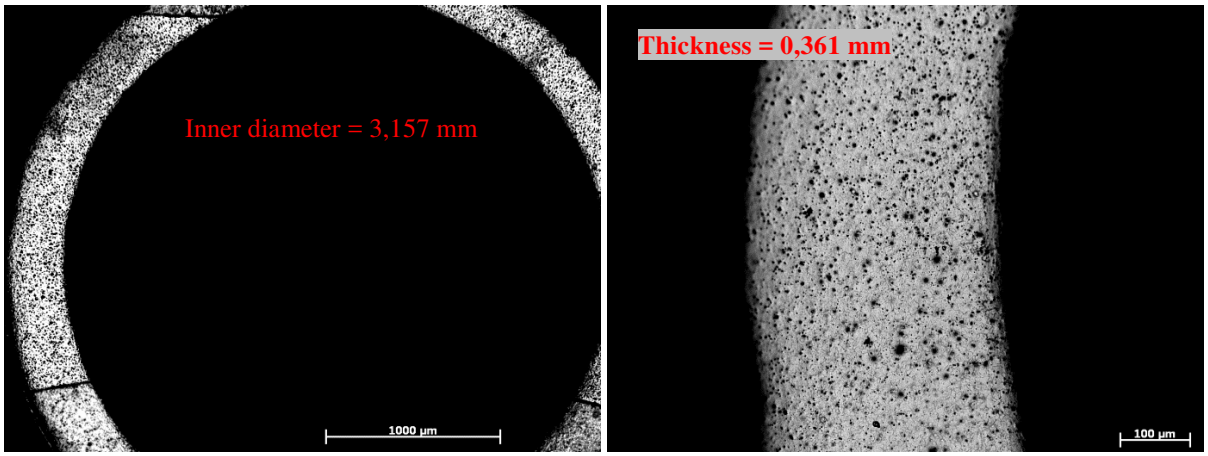


B2 Average inner diameter = **4.240 mm**

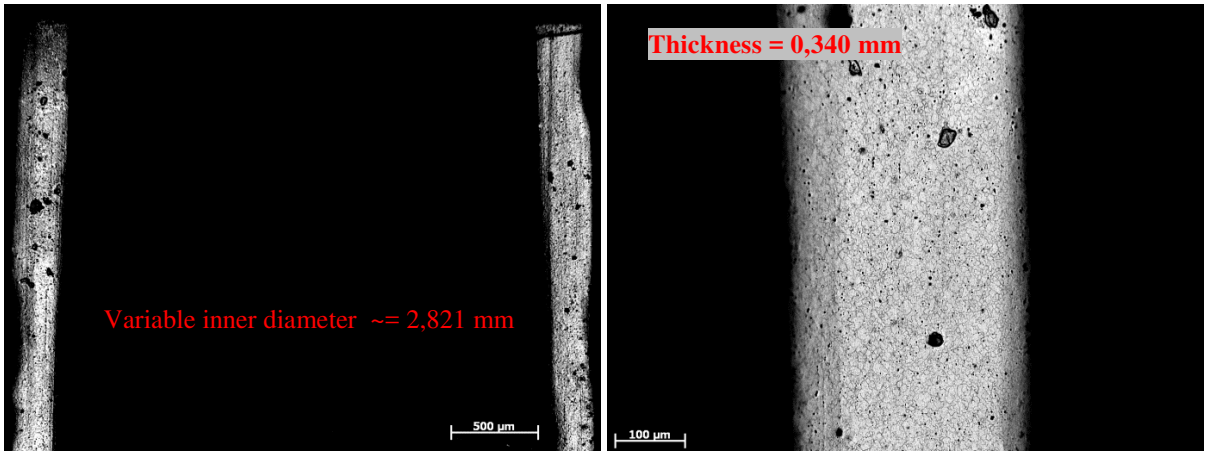
B2 Average tube thickness = **0.483 mm**

B1 – LDD (graphite painted)

RD-RD plane



AD-RD plane



B1 Average inner diameter = **3.157 mm**

B1 Average tube thickness = (**0.351±0.011) mm**

3. Grain size measurements

The images with higher magnification from the previous part of the report were used. Two typical micrographs were chosen for each sample (one in RD-RD and one in AD-RD plane). These were thereafter analyzed using the Linear Intercept Method, with five horizontal and five vertical lines across each micrograph (see Figs. 3.1 and 3.2). Resulting mean intercept length was multiplied by the factor of

1.74 to obtain respective average grain size. This factor is a result of stereographical calculations made in the literature [B Roebuck, C Phatak and I Birks-Agnew, A Comparison of the Linear Intercept and Equivalent Circle Methods for Grain Size Measurement in WC/Co Hardmetals, NPL Report MATC(A)149, National Physical Laboratory Teddington, Middlesex TW11 0LW, UK (2004)]. For each sample and each plane we provide three micrographs, **of which the most left one was analyzed**. This method is not suitable to provide reasonable standard deviation value as the normal distribution of grain size cannot be expected. The standard deviation of grain size is normally provided only when the analysis is carried out by a more advanced technique which is able to determine complete grain size distribution histogram, like the EBSD. This is not necessary for this microstructure and required precision.

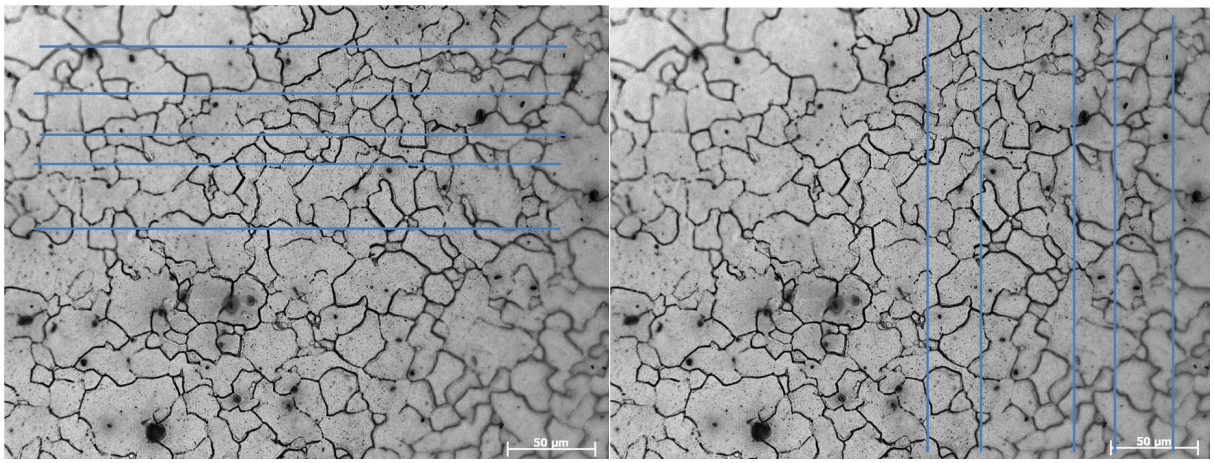


Fig. 3.1 Example of five horizontal and five vertical analyzed lines (RD-RD direction, A1 sample)

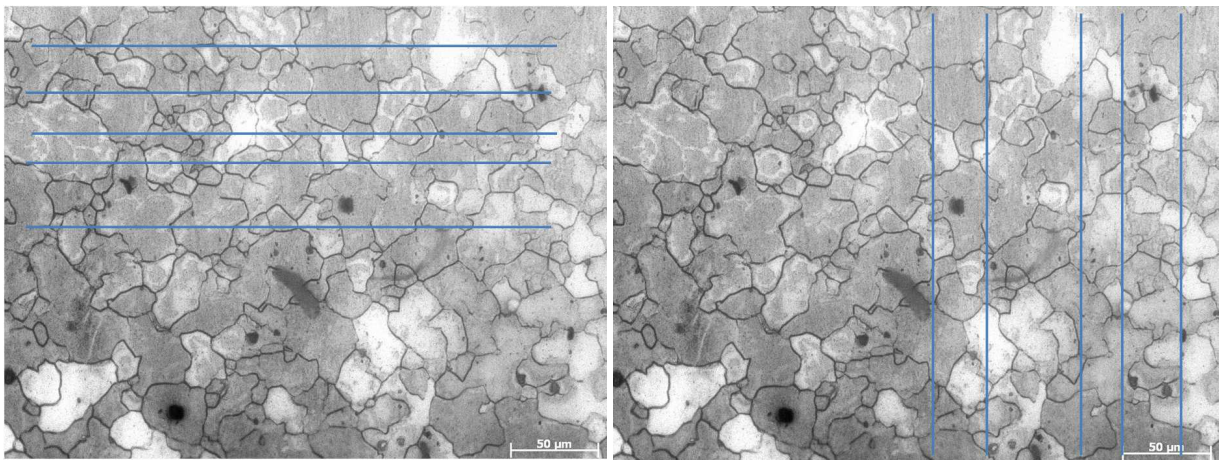


Fig. 3.2 Example of five horizontal and five vertical analyzed lines (left: RD direction, right: AD-direction; A1 sample)

Results are summarized in the tables below.

AD-RD plane (plane parallel to tube axis)				
Sample	**A1**	**A2**	**B1**	**B2**
x-line length (um)	1500	1500	750	-
y-length length (um)	1000	1000	652	-
RD intercept count	81	114	171	-
AD intercept count	71	86	114	-
RD average intercept (um)	18.52	13.16	4.39	-
AD average intercept (um)	14.08	11.63	5.48	-
Factor	1.74	1.74	1.74	-
RD average grain size (um)	**32.22**	**22.90**	**7.6**	**-***
AD average grain size (um)	**24.50**	**20.20**	**9.5**	**-***

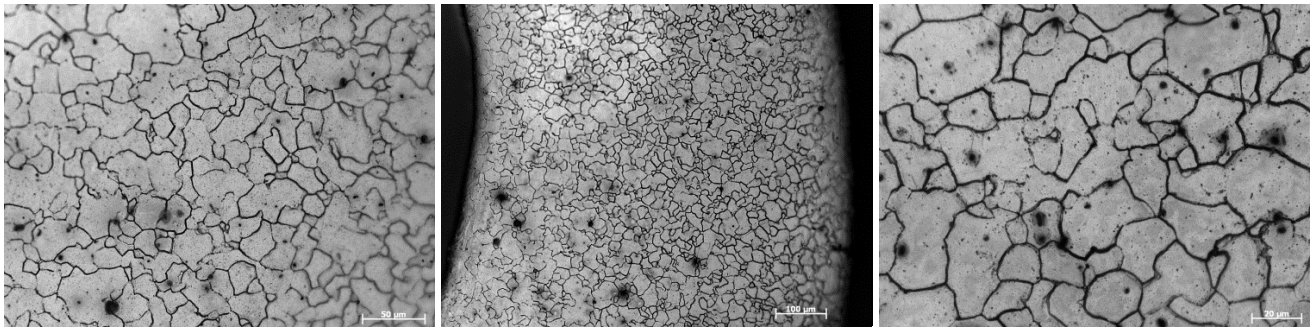
* not analyzed

RD-RD plane (plane perpendicular to tube axis)				
Sample	**A1**	**A2**	**B1**	**B2**
x-line length (um)	1500	750	750	750
y-length length (um)	1000	625	625	625
x intercept count	94	56	172	171
y intercept count	78	43	127	131
x average intercept (um)	15.97	13.39	4.36	4.39
y average intercept (um)	12.82	14.53	4.92	4.77
Factor	1.74	1.74	1.74	1.74
RD average grain size (um)	**25.10**	**24.30**	**8.10**	**8.00**

4. Images

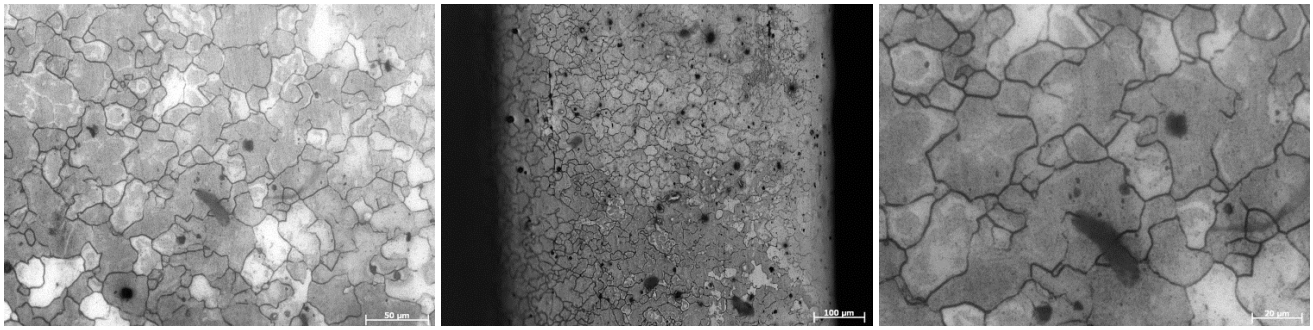
A1 (RD-RD) (perpendicular)

x average Grain size = 27,8 um
y average Grain size = 22,3 um
average RD Grain size = 25,1 um



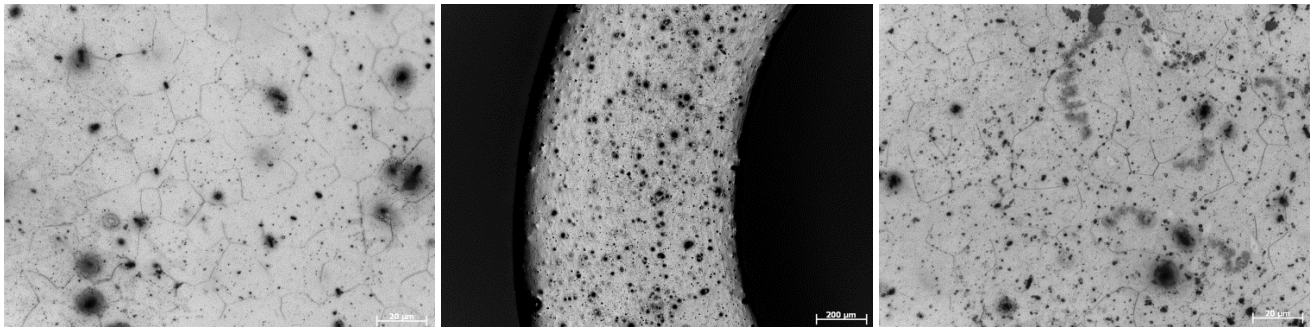
A1 (AD-RD) (in axis)

RD average Grain size = 32,2 um
AD average Grain size = 24,5 um



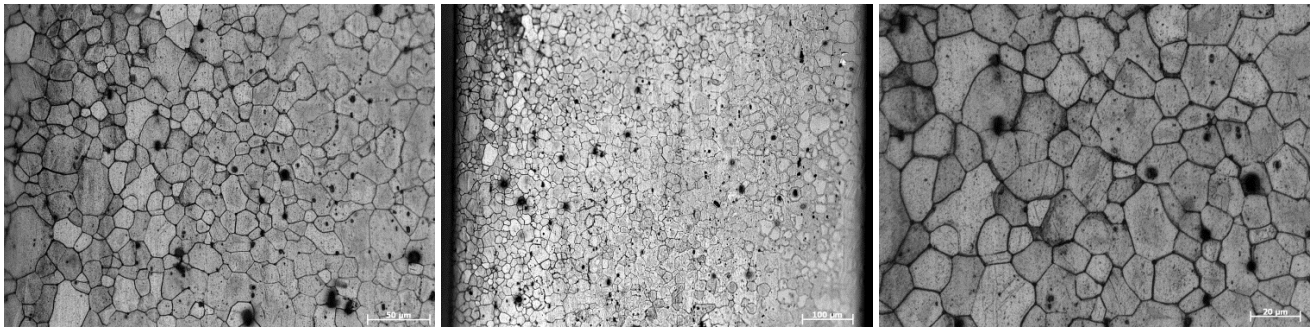
A2 (RD-RD) (perpendicular)

x average Grain size = 23,3 um
y average Grain size = 25,3 um
average RD Grain size = 24,3 um



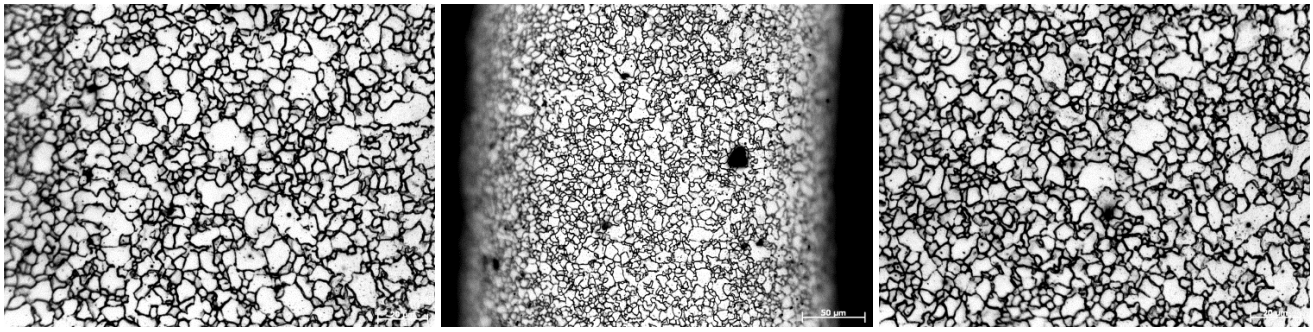
A2 (AD-RD) (in axis)

RD average Grain size = 22,9 um
AD average Grain size = 20,2 um



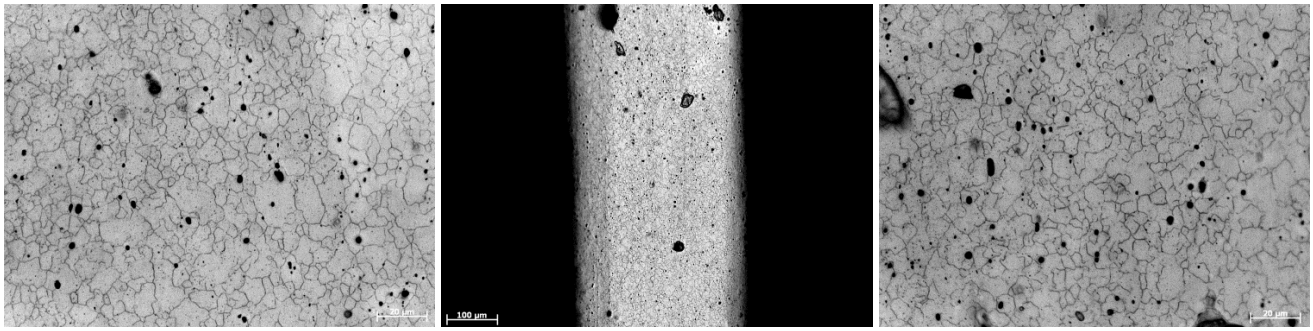
B1 (RD-RD) (perpendicular)

x average Grain size = 7,6 um
y average Grain size = 8,6 um
average RD Grain size = 8,1 um



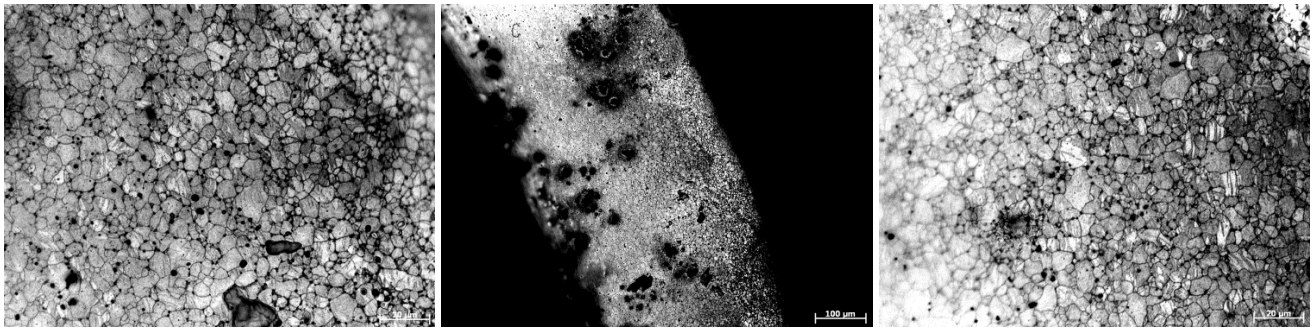
B1 (AD-RD) (in axis)

RD average Grain size = 7,6 um
AD average Grain size = 9,5 um



B2 (RD-RD) (perpendicular)

x average Grain size = 7,6 um
y average Grain size = 8,3 um
average RD Grain size = 8,0 um



B2 (AD-RD) (in axis)

- high radial variations of grain size! (constant in AD)
- Lower grain size near the outer surface (~3 um)
- Grain size on the inner side converges to the RD Grain size values
- Average grain size values not assessed