Selection of tungsten bricks for the target of the European Spallation Source

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One year ago (13/10/2015):

we presented a protocol for selecting a supplier of W bricks among 6 candidates and some preliminary results from a 1st stage of tests

The selection was made in two steps:

- 1st stage of tests for selecting 3 potential suppliers
- 2nd stage of tests for deciding on the definitive supplier

The two stages were completed.

We present today an overview of the results of the selection*

*(Two reports have been written giving detailed account of them)

SCOPE

- Brief introduction: the critical property from the design perspective of the target
- First stage of tests from 6 tentative suppliers. Selection of 3 of them for a 2nd stage
- Second stage of tests. Ranking of the selected 3 from a technical point of view
- Suggestions for specifications to be met by the definitive supplier and for the protocol of acceptance/rejection of a lot of bricks

Bricks should resist 5 years of:

- Thermomechanical cycles (resists static and fatigue failure, RT < T < 400°C)
- Erosion and oxidation (resists erosive wear, He atmosphere)
- Neutron irradiation cycles (increasing damage: consider safety factor for ageing)

Critical properties from the design perspective of the target:

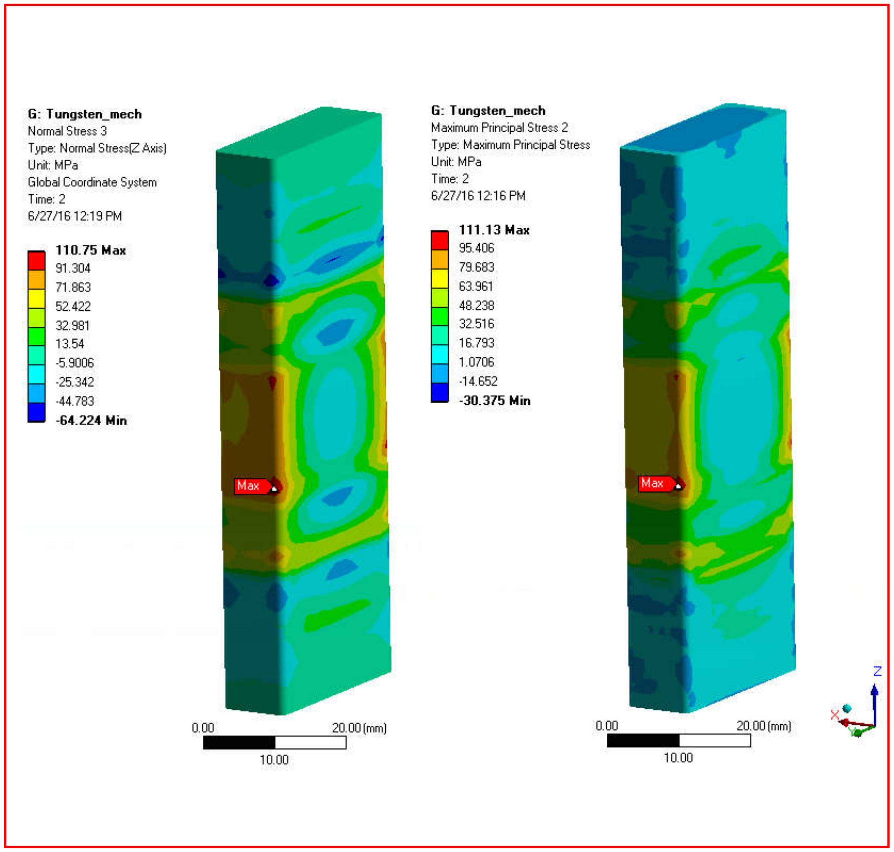
- Strength (yield stress, fracture stress) & its anisotropy
- Ductility / Toughness & its anisotropy
- Surface integrity (roughness, defects, residual stress state)

…connected to structural parameters:

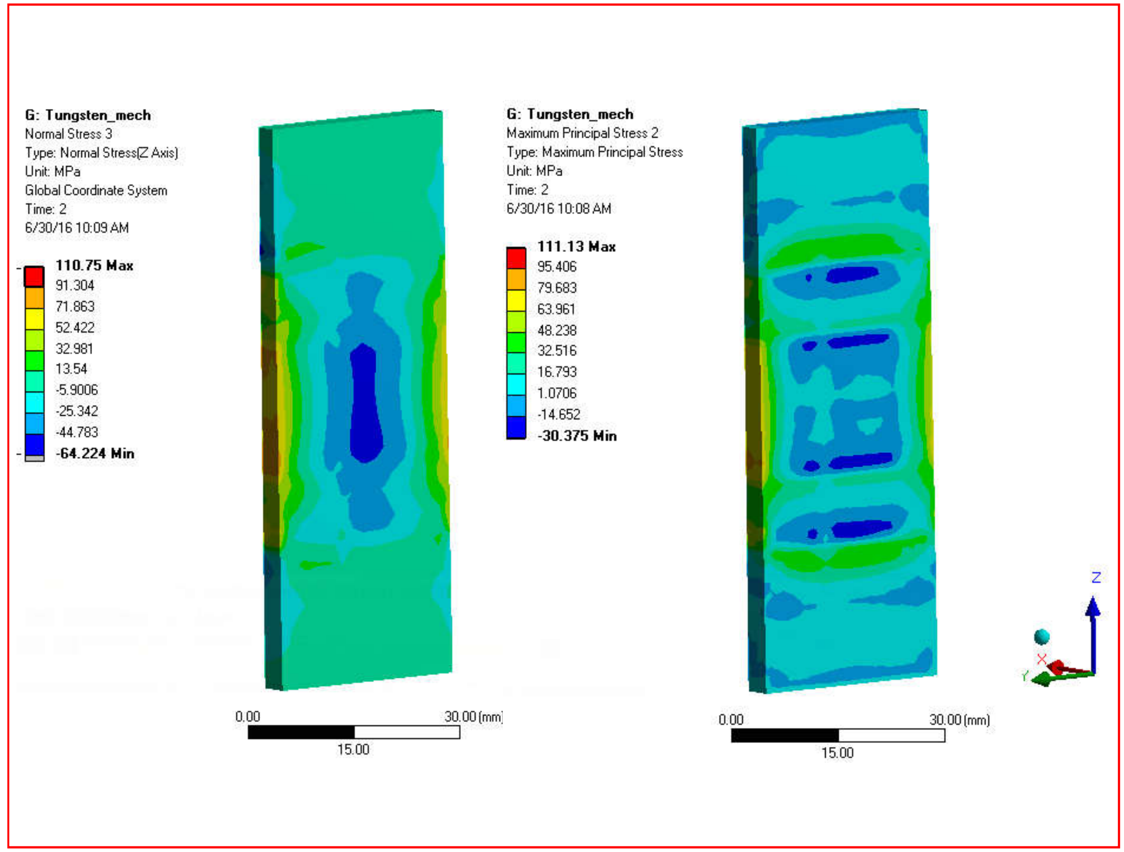
- Purity
- Relative density and elastic modulus
- Grain size (decreases yield stress and toughness)
- Deformed structure (increases yield stress and toughness)
- Texture

…all of them connected to processing.

Maximum applied tensile stress: **<u>~ 111 MPa in longitudinal direction</u>** at the surface, in the central part of bricks



Surface



Mid-thickness

Summary of the conditions requested to potential suppliers of tungsten bricks

Chemical composition	W, pure (commercial purity)
Processing	Hot rolling
Tensile strength	> 600 MPa
Dimensions	10 (h10)x30 (h9)x80 mm^3
Surface roughness	$R_a \leq 6.3$ µm
Dimensional and mechanical stability	Above geometry and strength stable to brazing operation to stainless steel (~3 h in a vacuum furnace at 10^{-5} mbar, ~1000ºC)

A comment on the requested W strength for the bricks

(600 MPa vs. maximum expected stress per cycle, 111 MPa)

- About half of each brick cycle takes place below the W DBTT
- Below DBTT, failure is brittle (i.e., fracture stress is a stochastic variable)
- Brittle fracture stress of metals: Weibull distribution probability function with shape factor $10 \leq m \leq 20$ (few published data for W)
- Assuming a "*safety factor*" of 3 for the failure stress (on account of in-service degradation, etc.), we need for the W bricks a low probability of fracture under 333 MPa (minimum *acceptable strength level of a brick individual*)
- Assuming a Weibull shape parameter $m = 10$, the distance of the acceptable strength level from a mean strength of 600 MPa is about 4 times the standard deviation*
- i.e., a mean strength of 600 MPa assures a very low probability of in-service failure

[* For $m = 10$, the standard error is 0.115 and the Weibull scale parameter is 1.05 relative to the mean]

Bricks from 6 different suppliers were provided by ESS-Bilbao, identified by a numeral

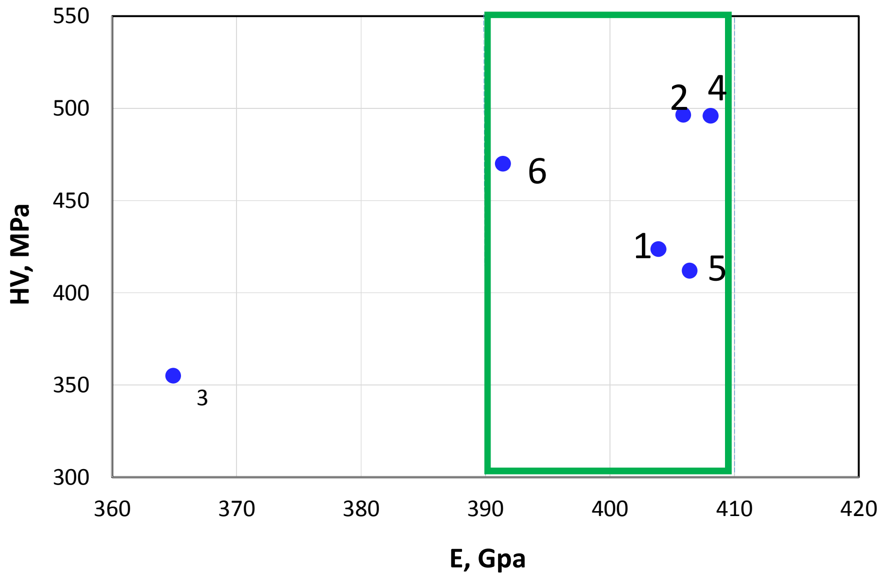
Ceit-IK4 was blind towards the identity of brick suppliers

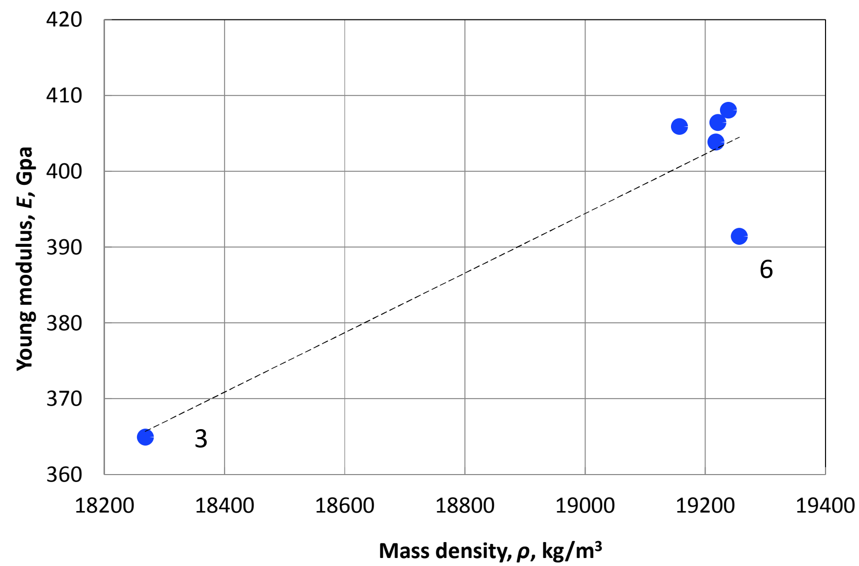
STAGE 1 OF SELECTION
The bricks were examined by or underwent testing for:

- Visual inspection
- Chemical composition (C, S, O, N)
- Mass density
- Young elastic modulus in short-transverse direction (full thickness)
- HV (1kg) Vickers hardness (on the rolling plane, as-received surface)
- Residual stresses in the as-received surface (RX)
- Tensile strength measured at RT in 3P bending with tensile failure nucleating from the as-received (intact) surface of the bricks (rolling plane)
- Fracture surface characteristics (SEM)

SUMMARY OF RESULTS Stage 1

W supplier	Visual inspection	Density, ρ (g cm^{-3}) Geom. ± assoc. error	Density, ρ (g cm^{-3}) Water displ. ± sd	E, Young modulus RPN (GPa) ± assoc. error	HV (1 kg) RP (kg mm^{-2}) ± 95% cl	Res. stresses, surface (MPa) ± sd σ_{11} (LD)	Res. stresses, surface (MPa) ± sd σ_{22} (TD)	Fractography	Chemical composition Impurities above threshold
1	Grey spots (oxide) on surface	19.22 ±0.03	18.95 ±0.22	403.9 ±0.7	423.7 ±25.7	-1276 ±9	-1074 ±13	Brittle, transgranular, distorted cleavage , oriented facets Minor intergranular fraction Scarce nano-porosity	-
2	Thin continuous (oxide) layer on surface	19.16 ±0.03	19.21 ±0.03	405.9 ±0.8	496.5 ±9.5	-789 ± 11	-1088 ± 9	Brittle, transgranular, distorted cleavage , oriented facets Minor intergranular fraction Some micro-porosity	-
3	Damaged edges. Scratches on surface, slightly oxidized (finger prints).	18.27 ±0.03	17.69 ±0.03	364.9 ±0.7	355 ±6	-956 ± 20	-1166 ± 8	Brittle, intergranular fracture, equiaxed grains, high porosity Some precs. at grain edges	-
4	Bright smooth surface, free from oxides	19.24 ±0.03	19.20 ±0.03	408.1 ±0.8	496 ±6.0	-225 ± 27	-1113 ± 11	Brittle, transgranular, distorted cleavage , oriented facets Minor intergranular fraction Scarce nano-porosity	-
5	Brightest, smoothest surface. Free from oxides	19.22 ±0.03	19.23 ±0.01	406.4 ±0.8	412 ±16	-230 ±24	-247 ±26	Brittle, transgranular, distorted cleavage , oriented facets Minor intergranular fraction Scarce nano-porosity	>30 ppm O (44 ppm)
6	Rough surface, free from oxides. Bricks slightly shorter?	19.26 ±0.03	19.15 ±0.05	391.4 ±0.7	470 ±5.0	-709 ± 18	-1055 ± 7	Brittle, transgranular, distorted cleavage , oriented facets Minor intergranular fraction Porosity not detected	-



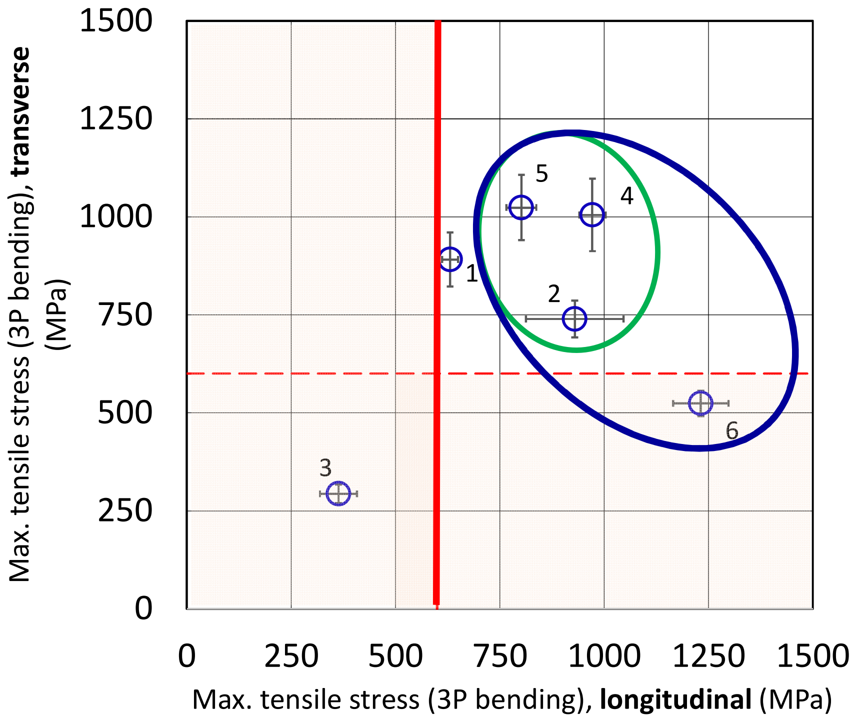


Comments:

3, out of bounds (high porosity, low hardness)
6, lower *E* but high density (?)
1 and 5, significantly softer tan the others
2 and 4, same supplier of raw material?

HV, E & ρ do not depend on the surface state

TENSILE FRACTURE STRENGTH AT RT

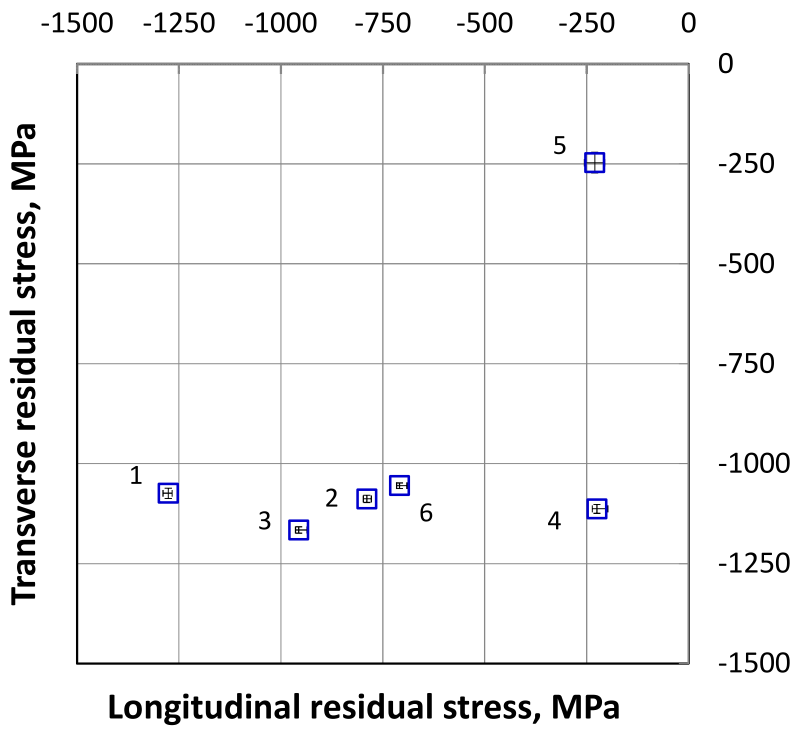


3, to be rejected
1, borderline
2, 4, 5, 6, OK considering the longitudinal fracture strength
6, high anisotropy of tensile strength (although transverse strength still acceptable according to design)

SURFACE RESIDUAL STRESSES

Biaxial residual stress pattern of **brick 5** very different from that of other bricks

Longitudinal residual stresses of **brick 4** also Rather weak



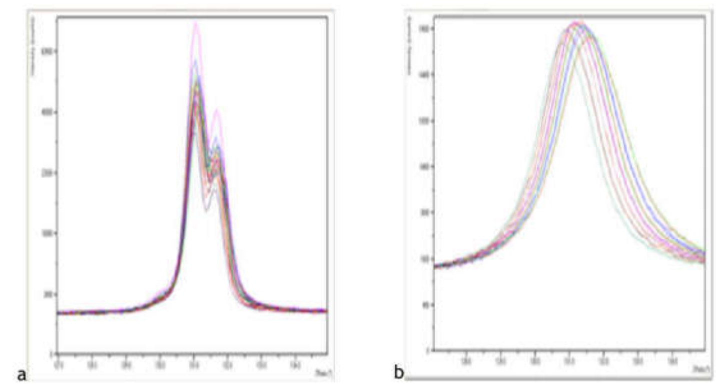
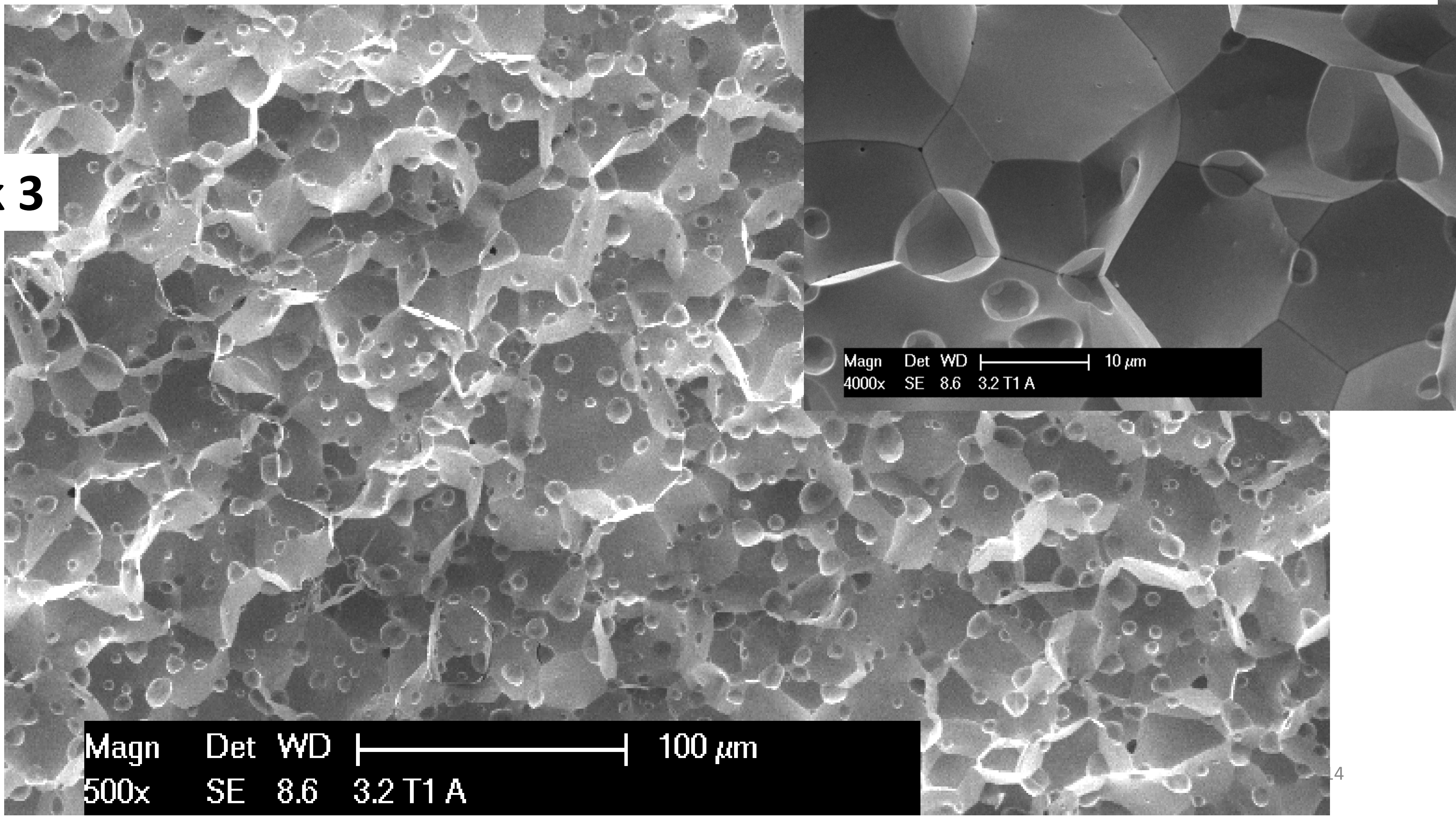


Figure 3. X-rays (321) diffraction peaks, samples 5 (a) and 2 (b).

Diffraction peaks of **brick 5** do not show any broadening typical of deformed microstructures, as the others do

FRACTOGRAPHY (fracture under tension stress in longitudinal direction)

Brick 3



FRACTOGRAPHY (fracture under tension stress in longitudinal direction)

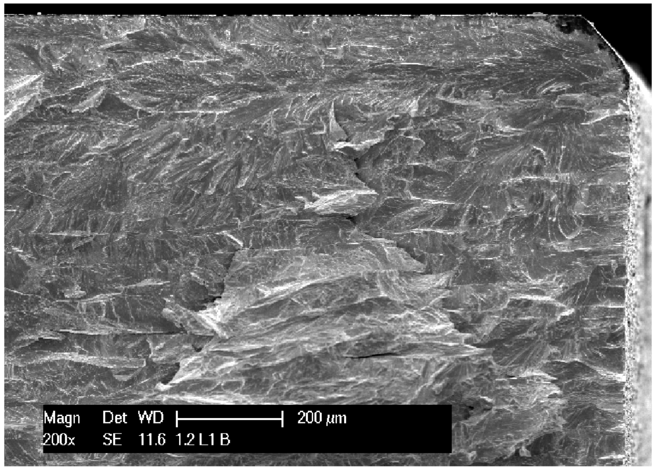


Figure 20. 1, L, x200. Horizontal direction is the presumed transverse-to-the rollin direction.

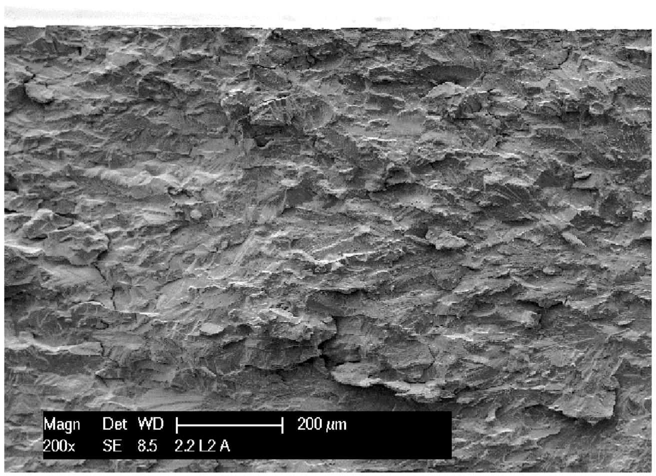


Figure 21. 2, L, x200

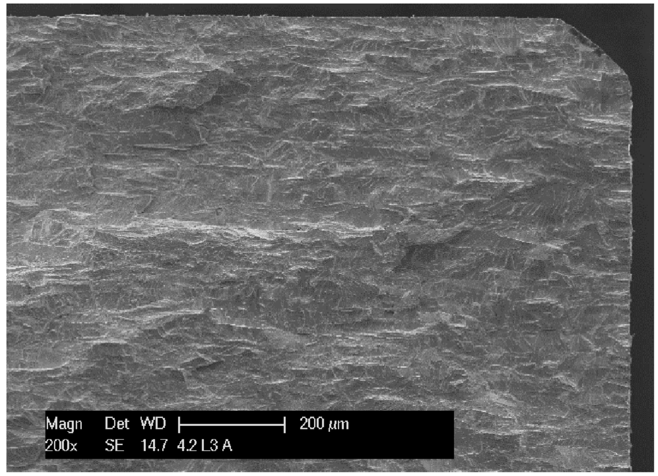


Figure 22. 4, L, X200

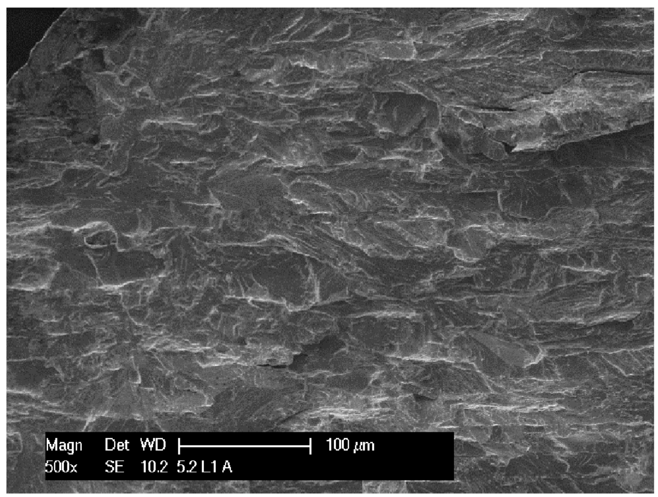


Figure 23. 5, L, x500

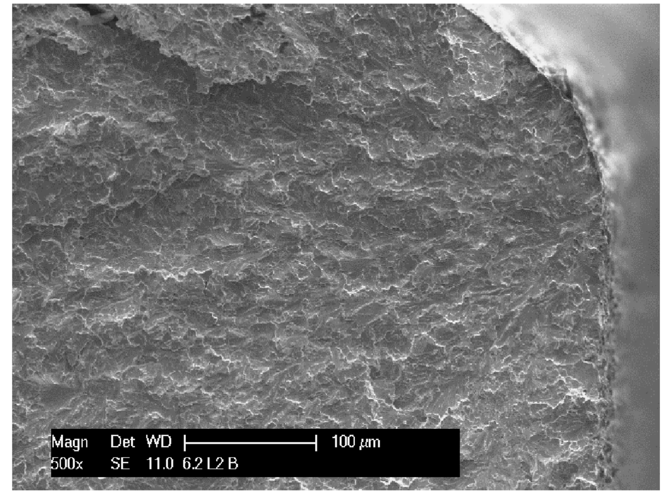


Figure 24. 6, L, x500

Fractographic patterns coherent with the other observations of mechanical or physical properties

CONCLUSION FROM Stage 1 of selection of W supplier

- Brick 3: reject
- Brick 1: at the borderline of acceptability for the application; reject for Stage 2
- **Bricks 2, 4, 5 and 6**: meet the mechanical requirements; could be accepted for Stage 2

….however, notice that:

- Brick 5 shows several unexpected aspects:
  - lower level of compressive residual stresses
  - signs of recrystallized microstructure
  - softer than 2, 4 or 6
- Brick 6 shows:
  - smaller than expected elastic modulus (despite high density)
  - poor tensile strength in transverse direction

On the basis of the set of results and observations,

ESS-Bilbao decision:

Bricks 2, 5 and 6 pass to Stage 2 selection tests

STAGE 2 OF SELECTION

Bricks 2, 5N* and 6 tested for assessing:

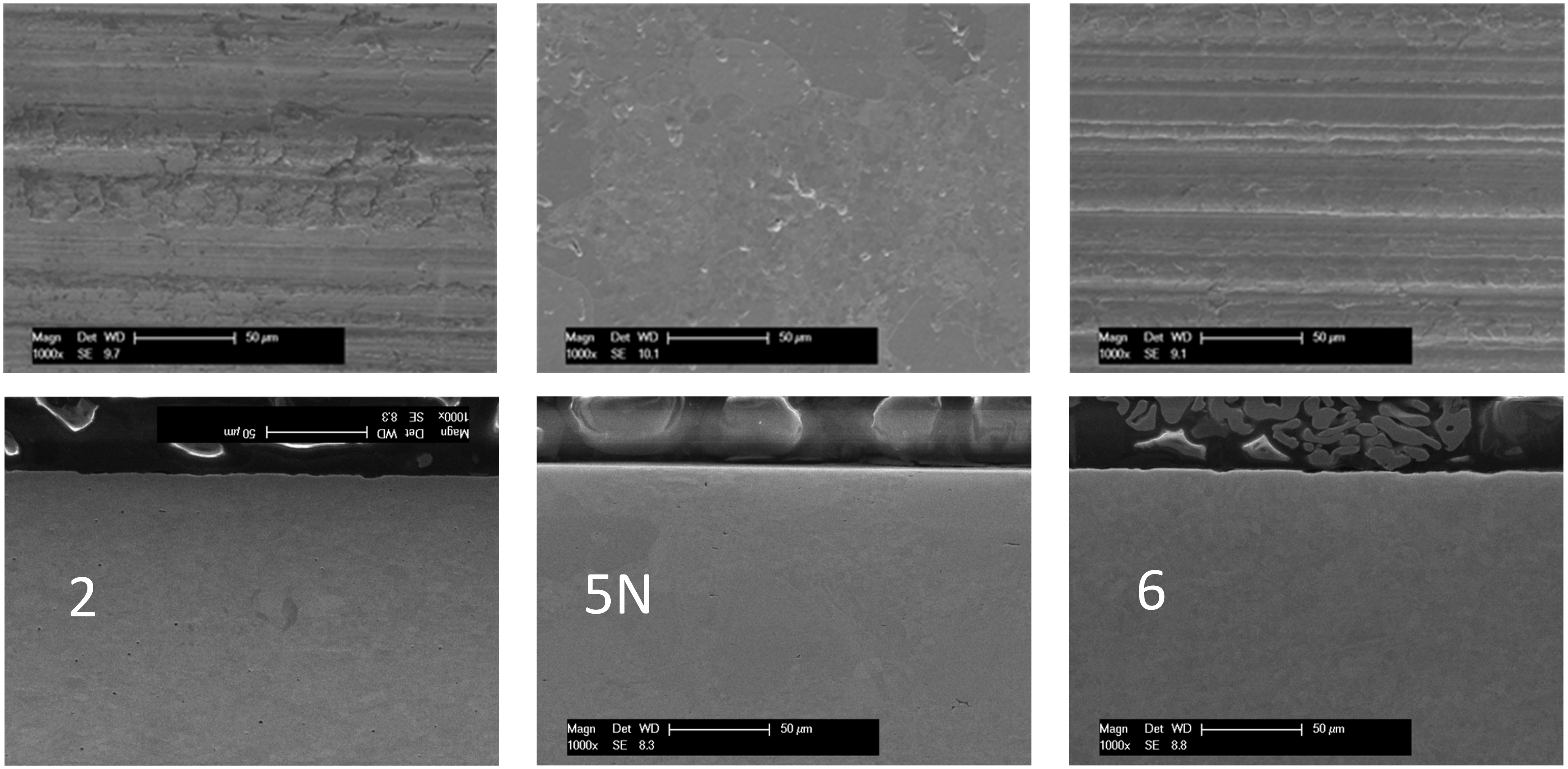
- Surface state (SEM)
- Microstructure (SEM-OIM-EBSD)
- Crystallographic texture (EBSD)
- Residual stress state (DRX, surface and sub-surface)
- Tensile strength at 400°C (3P bending)
- Fractography (400°C)
- Fracture toughness at RT, K_{Ic} (Barker chevron-notched specimens)

* Supplier 5 sent new samples of his bricks with a new polishing treatment. They have been named 5N

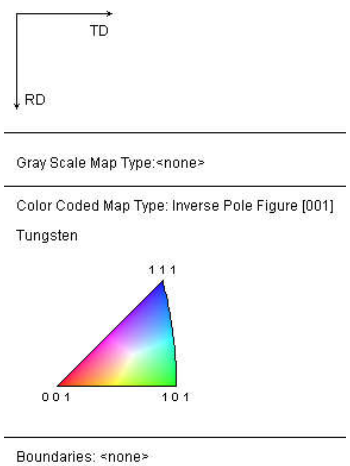
A previous question: **5N vs. 5 bricks**

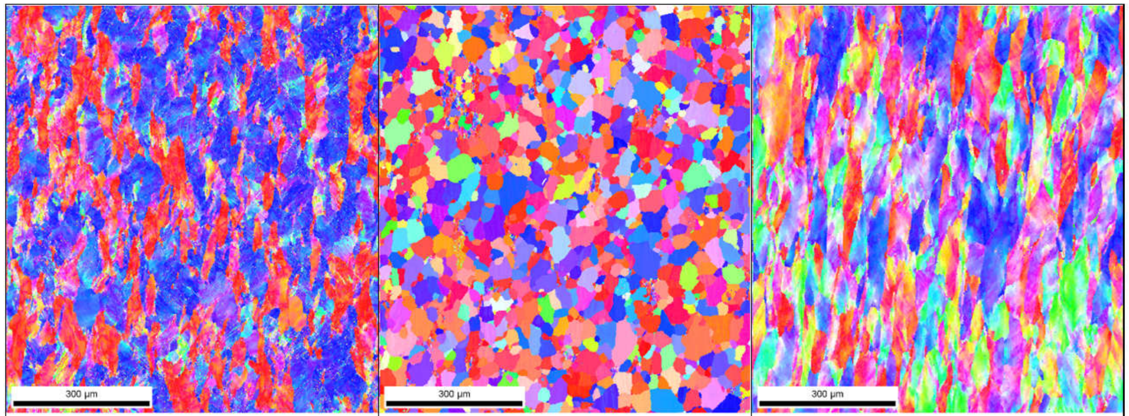
- Same HV
- Weakened compressive stress state
- Same narrow DRX peak width
- Smaller tensile strength at RT (<600 Mpa)

Surface state (SEM): rolling surface and NT section

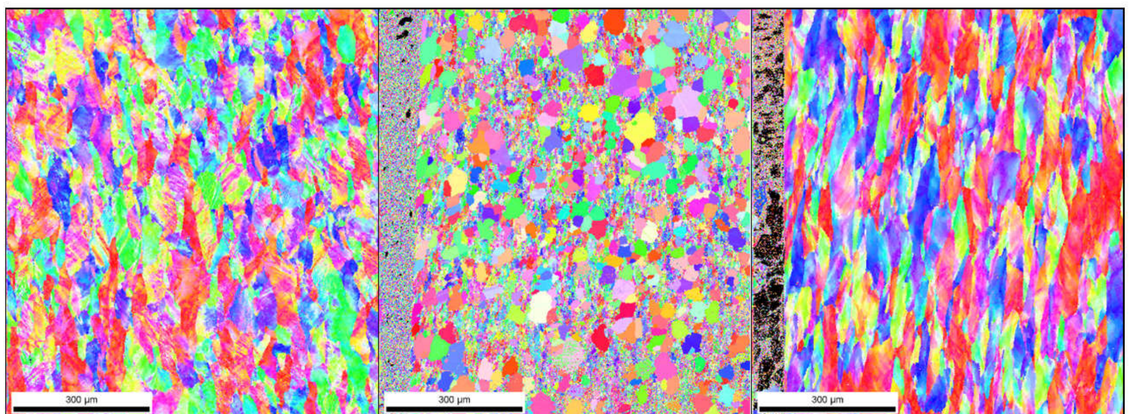


Microstructure (SEM-OIM-EBSD)





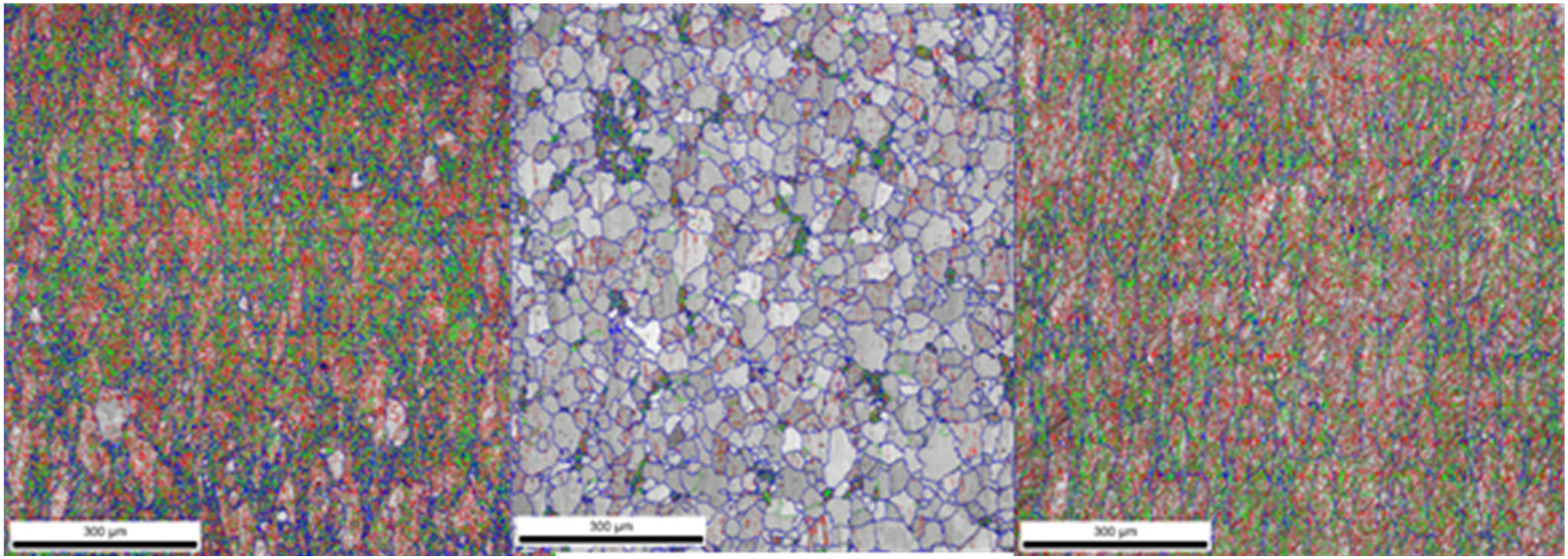
LN section, midplane. From left to right: 2, 5N, 6



LN section, surface. From left to right: 2, 5N, 6

Notorius structure/texture differences between both, different materials or between surface and interior of the same bricks

2 5N 6



Superposed: HAB (blue), LAB (green or red) and image quality gray scale

Grain sizes (LN sections)

Criterion for grain boundaries: 15°. Edge grains included in the analysis. Bracketed: standard deviation p.

Average diameter (by number), µm	No. 2	No. 5N	No. 6
MIDPLANE	24.0 (21.3)	21.3	36.7 (30.2)
SURFACE	34.4 (25.7)	14.1 (8.8)	34.0 (30.9)

TEXTURE

Typical bcc rolling texture components in the midplane:

{001} <110>
+
{111} <110>

Texture intensity is weak

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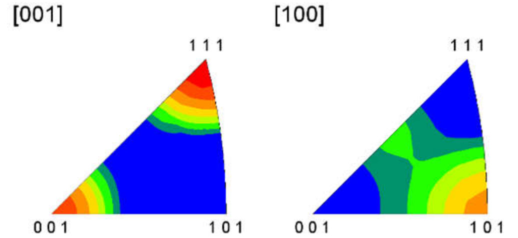
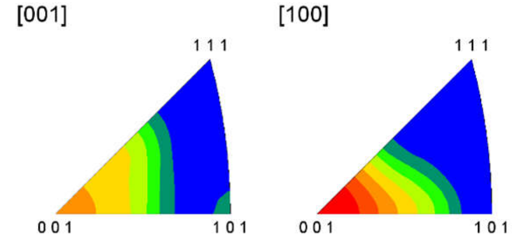
<tr><td>2</td><td>
</td><td>
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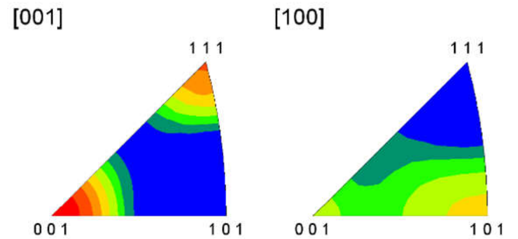
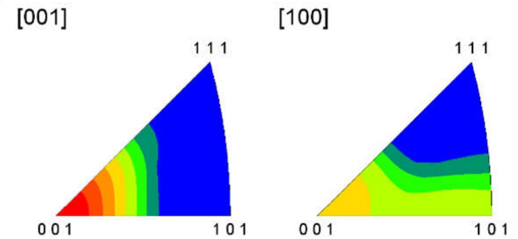
N</td><td>
</td><td>
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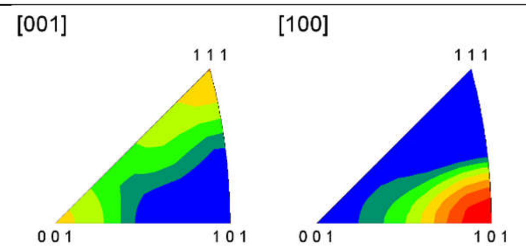
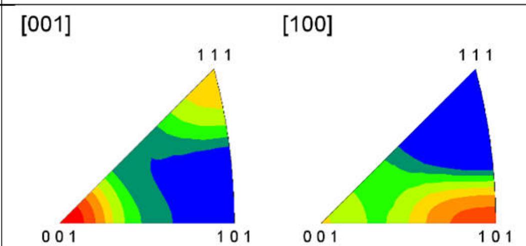
<tr><td>6</td><td>
</td><td>
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<tr><td colspan="2"></td><td>Inverse pole figures (normal direction, [001] and rolling direction, [100]).</td></tr>
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Different textures at the surface

Texture intensity is weak

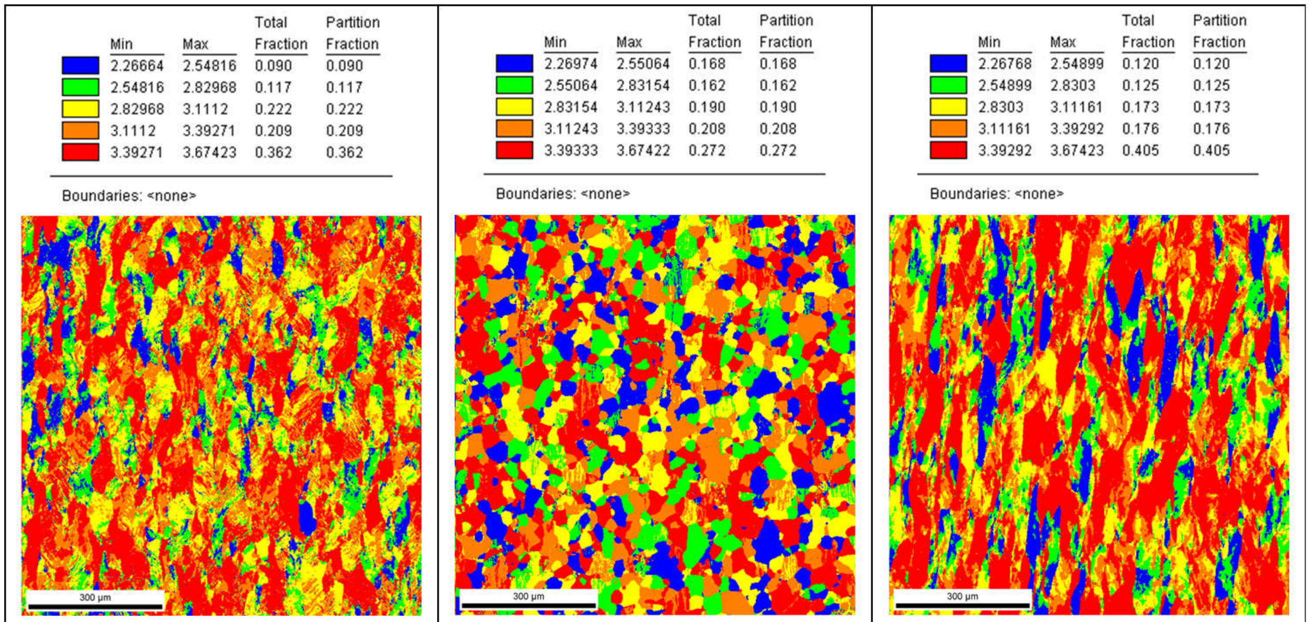
Texture intensity is weak

Plastic anisotropy is not important

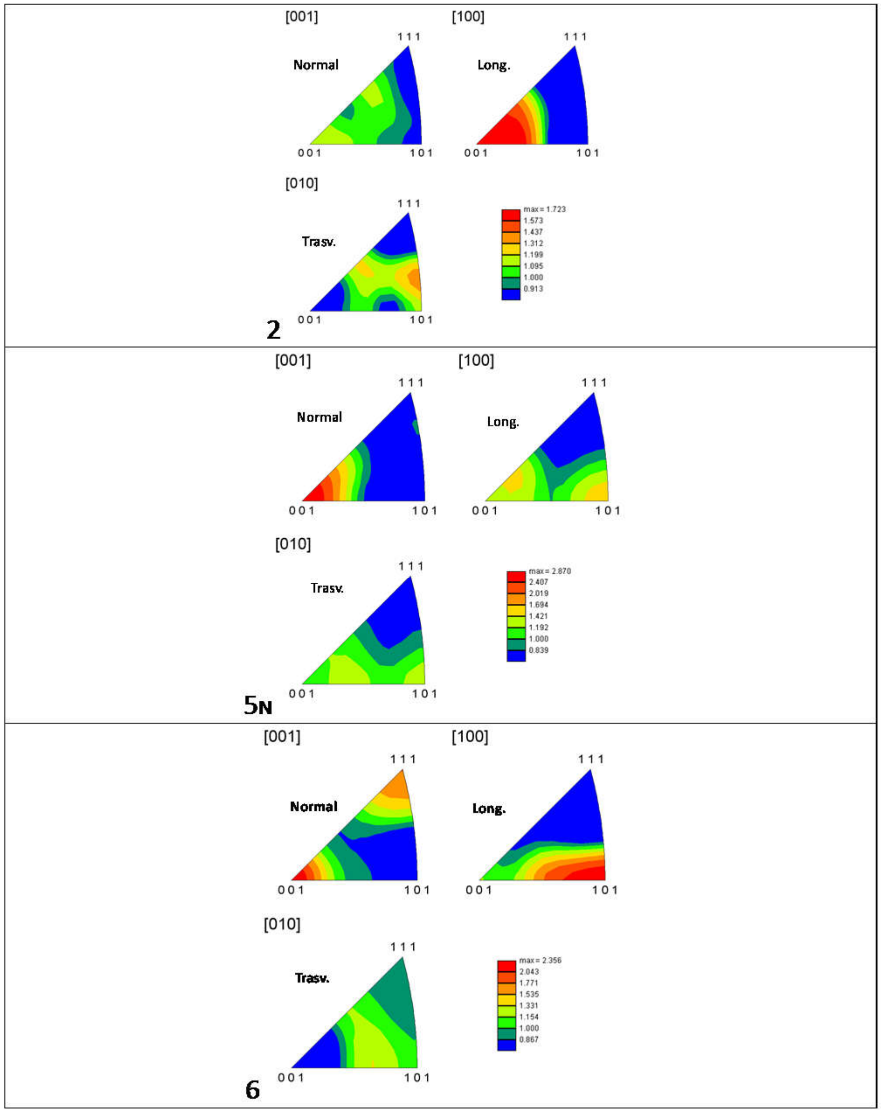
Taylor factors, axisymmetric tension

Sample	M_L	M_T
2 midplane	3.164 (0.383)	3.185 (0.373)
5N midplane	3.048 (0.413)	3.028 (0.427)
6 midplane	3.161 (0.418)	3.087 (0.436)

Maps of M_L



2 midplane, M_L | 5N midplane, M_L | 6 midplane, M_L



TEXTURE (IPF)
100 μm SURFACE LAYER

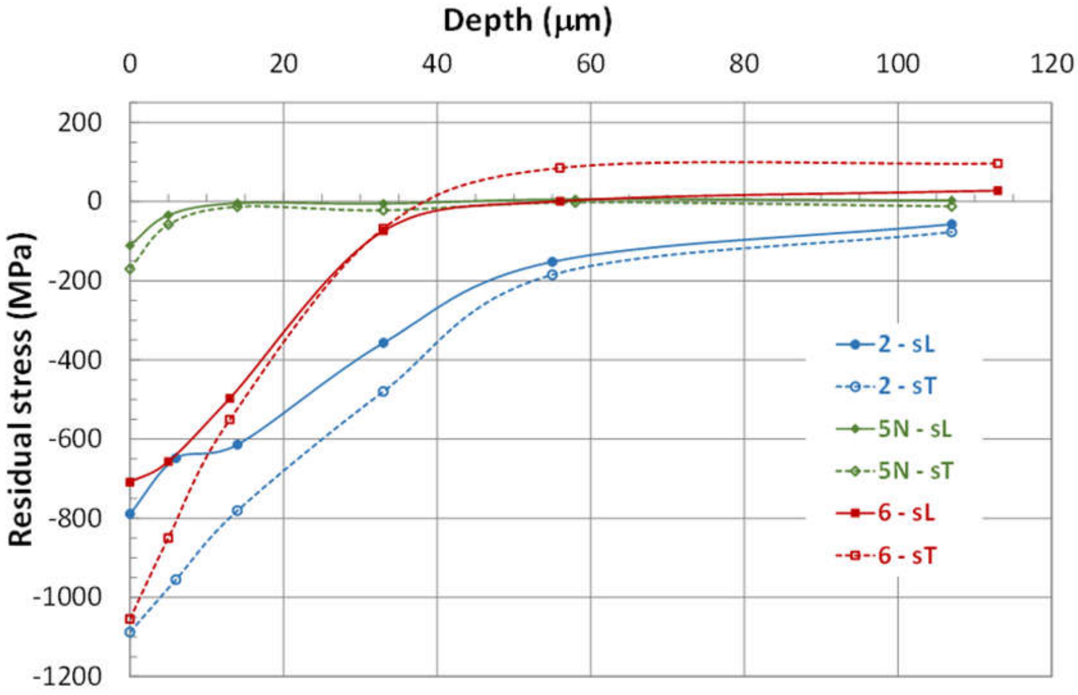
Intensity levels of cleavage-related plane normal weak and rather similar

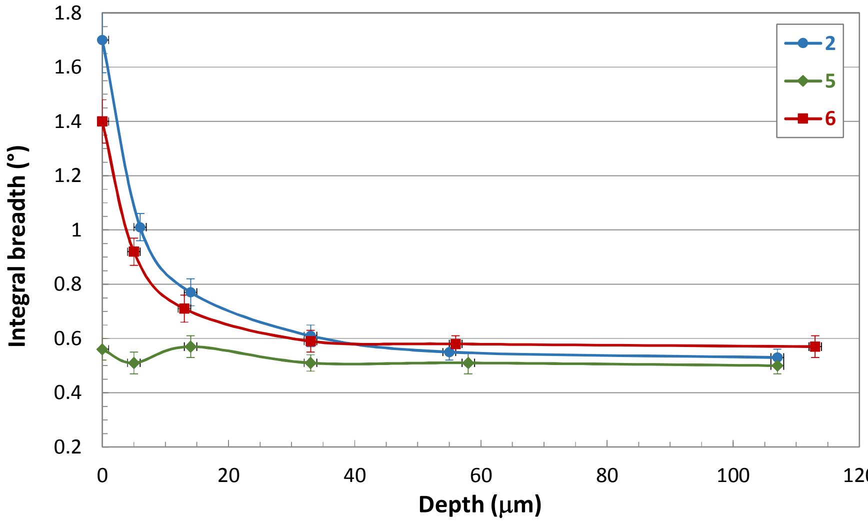
	Intensity in L direction	
Brick	<001>	<110>
2	1.57-1.72	< 0.91
5N	1.19-1.41	1.42-1.69
6	1.15-1.53	2.04-2.36

Residual stresses

<u>Bricks 2 & 6</u>: strong level of sub-surface biaxial compressive stresses up to resp. 30 and 50 μm

<u>Brick 5N</u>: weak compressive surface stress up to less than 10 μm

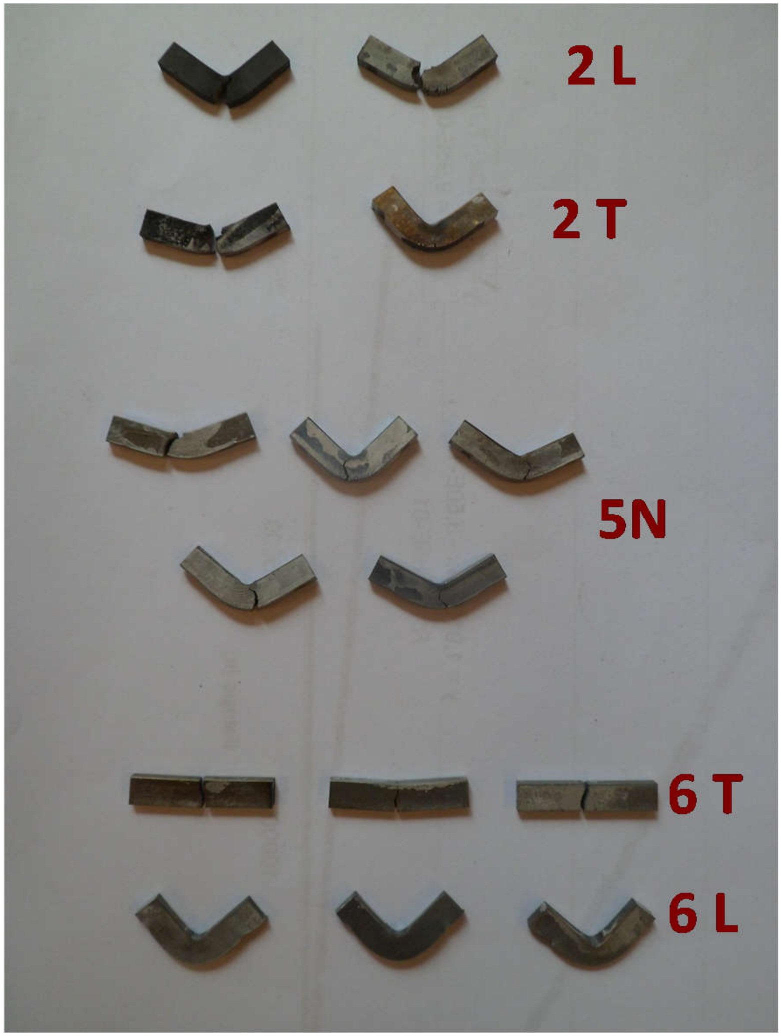




Tensile strength
400°C in air (3P bending)

In contrast with the RT behavior (brittle fracture in the elastic loading range),all fractures at 400°C occurred after some plastic deformation (although samples 6 of T orientation broke after a minimal strain after the elastic limit)

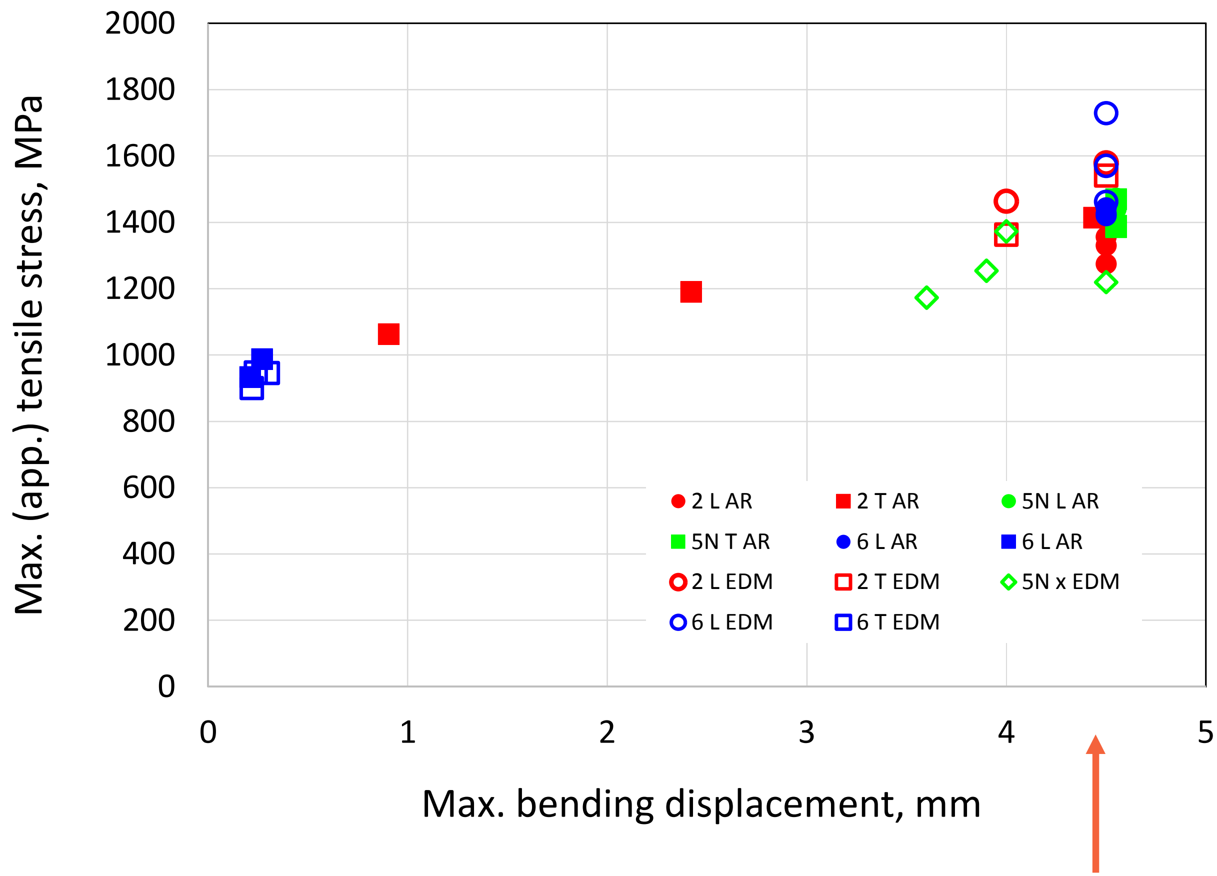
The facies of the fracture surfaces was brittle in all cases (mainly by distorted cleavage, with some intergranular decohesion for bricks 2 and 6, mainly by intergranular decohesion for brick 5N)



Series of broken samples, fracture nucleated from EDM surface

Tensile strength, 400°C in air (3P bending)

L and T orientations, fracture nucleated either from as-received or from EDM surface



Surface in tension:

AR: as-received surface, filled symbols

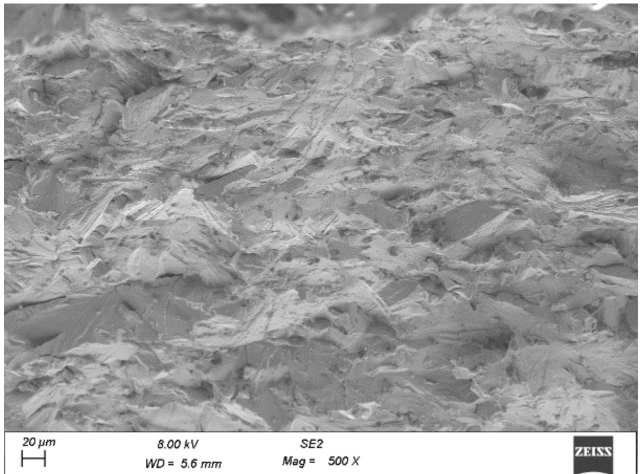
EDM: electro-discharge machined

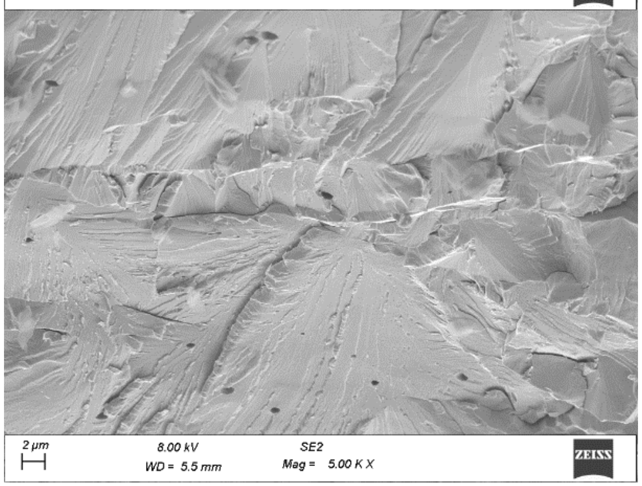
Circles, **L** direction (long.)

Squares: **T** direction (transv.)

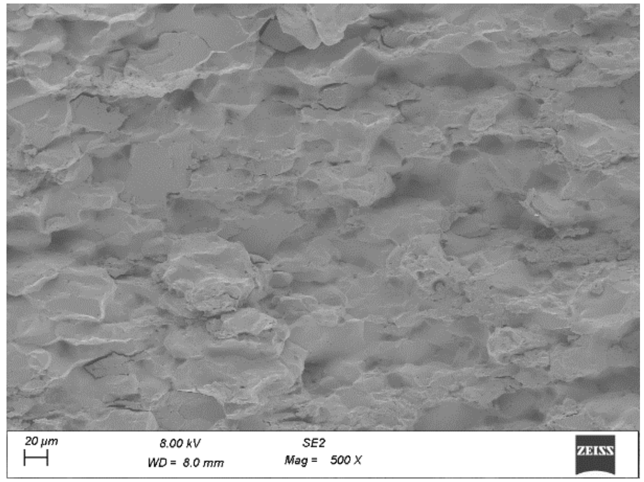
The maximum displacement allowed by the bending rig was 4.5 mm

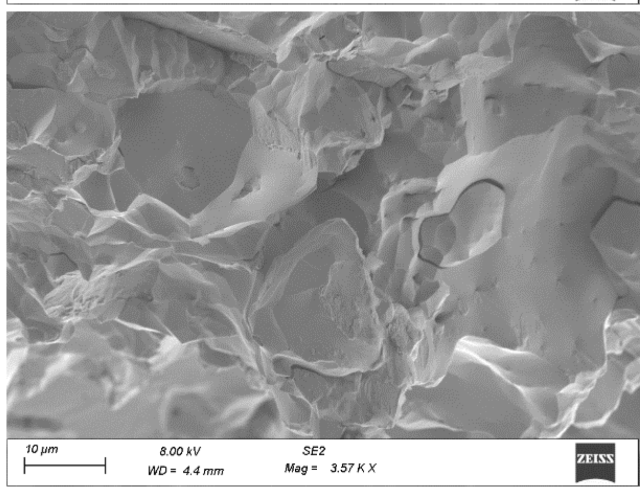
2



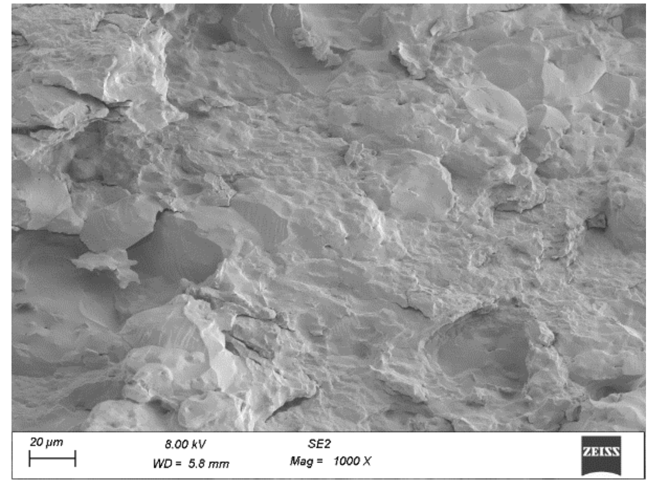


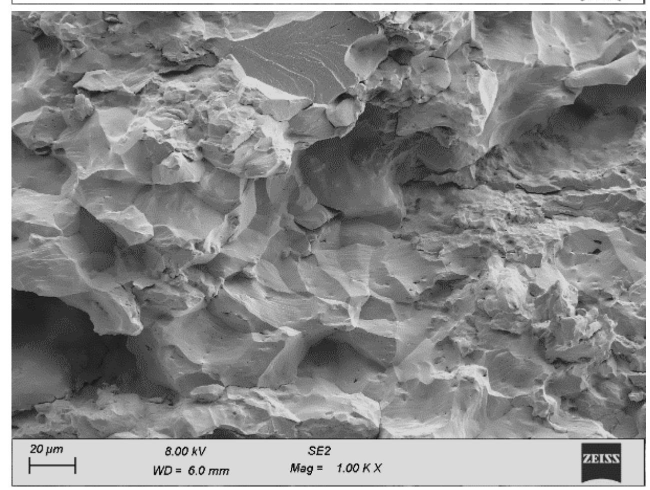
5N





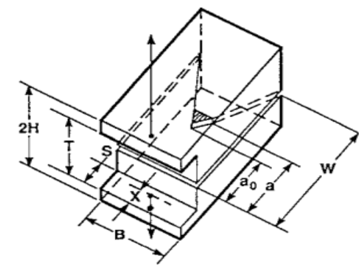
6

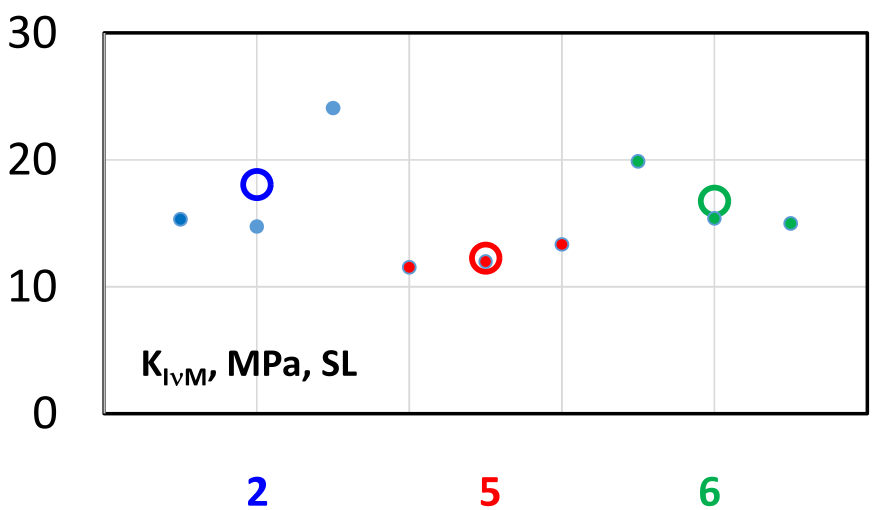


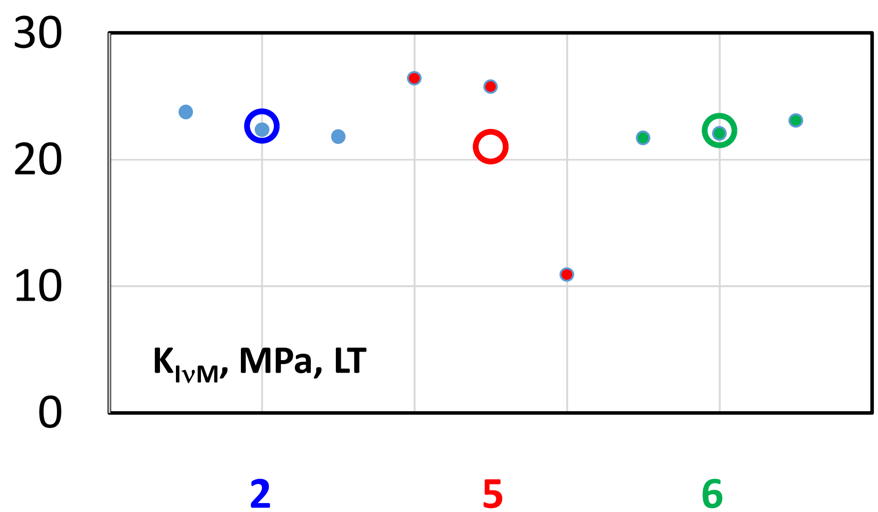


3P-bending, 400ºC in air, L orientation of tensile stress

RT plane-strain FRACTURE TOUGHNESS
Barker chevron-notched specimens
Crack propagation toughness, ASTM 1304-97







Orientation convention: SL: S (short-transverse) loading direction, L (longitudinal) crack propagation direction
LT: L loading direction, T (transverse) crack propagation direction

Considerable anisotropy, smaller toughness for crack propagation parallel to the rolling plane

CONCLUSION

In our opinion, from a technical point of view, the three materials studied in the Stage 2 of selection rank as 2, 6, 5N

- **2 and 6 have the expected rolled structure and favorable residual stress pattern. Their mechanical properties are above the thresholds assumed in the target design**

- **5N has a recrystallized equiaxed structure free from intragranular deformation structure. Such structure has unfavorable implications in the mechanical properties (strength and toughness)**

- **Properties of 2 outperform those of 6 in several aspects**

Suggestions for specifications of the definitive lot and for its acceptance

SPECIFICATIONS (supplier)	**Chemical composition**	Pure W bricks (specify W minimum content)	Commercial purity; supply analysis
	Processing (structure and surface integrity)	Rolling, stress relieving	Deformed and recovered structure
		Compressive residual surface stress state, no visible defects	
	Statistical process data of the room temperature tensile fracture stress in longitudinal direction L	$C_{pl} \geq 1.5$ LSL = 333 MPa	Quality control of the process
			Supply histogram or probability function parameters (obtained from a minimum of 30 specimens)
			Process capability according to ISO
ACCEPTANCE SAMPLING (AS) PLAN (ESS)	**AS sampling plan**	$\alpha = 5\%$ $\beta = 10\%$	Aim: defining sample size, n and estimate critical distance, k, from the sample data
		AQL = 80 ppm LTPD = 500 ppm	
	Tensile tests	Bending tests at RT of as received bricks, sample size according to AS plan	Aim: verification of process statistical data
	Structural and dimensional inspection	SEM-EBSD metallography, longitudinal section, 3 bricks from the n sample used for mechanical testing (those having the lower, mid and highest fracture stress of the sample) Vickers hardness	Aim: verification of specified structure and uniformity of rolling and heat treating processing
		X-ray diffraction, residual stress analysis Surface inspection Metrologic control	Aim: measurement of surface stress state and uniformity of machining process