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3 **W/EUROFER functionally graded coatings for plasma facing components:**
4 **technology transfer to industry and upscaling**

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9 **Abstract**

10 The tritium breeding blanket is a vital component of future fusion reactors, providing the fuel for the fusion
11 reaction. Tungsten is a viable coating material to protect its first wall from erosion by plasma. The feasibility
12 of applying tungsten coatings by vacuum plasma spraying has already been demonstrated, using a
13 functionally graded material of mixed tungsten and EUROFER97 steel as connecting layer to mitigate a
14 thermal expansion mismatch. This technology was now transferred to industrial level to enable further
15 upscaling. Samples of three different sizes were coated, the largest ones measuring 500×250 mm² and
16 containing mock-up cooling channels. The sample distortion was found to be small. An ultrasonic analysis
17 did not reveal any delamination but indicated potential weaker spots in the corners that may have been
18 subjected to faster cooling. Both the coating's thickness of 2 mm and linear chemical gradation over five
19 interlayers met well with specifications, as verified by scanning electron microscopy, energy-dispersive X-
20 ray spectroscopy as well as image thresholding analysis. The microstructure consisted of typical splat-
21 shaped particles of tungsten and EUROFER, with minor porosity only. In total, these first results indicate
22 that good coating quality can be achieved even in dimensions approaching fusion-relevant size.

23 **Keywords:** functionally graded material (FGM), plasma spraying, first wall, tungsten, EUROFER, plasma-facing
24 components.

25 **1. Introduction**

26 In future fusion reactors such as DEMO, the first wall panels of tritium breeding units will be subject to
27 substantial sputtering erosion [1] and neutron fluxes [2] as well as heat fluxes in the range of 0.3-2 MW/m²
28 [2–4]. To protect the structural steel of the first wall while still enabling heat transfer, a coating with tungsten
29 represents a promising solution. Tungsten's has a high sputtering resistance [5], high melting temperature
30 [6], high thermal conductivity [7] and low neutron activation [8]. The challenge of thermal expansion
31 coefficient mismatch between tungsten and steel may be overcome by creating a functionally graded
32 material (FGM) joint [9,10], using thermal spraying techniques, such as vacuum plasma spraying (VPS)
33 [11–13] or atmospheric plasma spraying [14,15]. Oxide formation can be avoided by using either vacuum
34 or non-oxygen gas shrouding [11,12,15].

35 In previous optimisation steps, coatings with a total thickness of 2 mm were developed, consisting of 0.8 mm
36 of tungsten top coat and 1.2 mm of functionally graded material with a linear gradient in tungsten content
37 over five interlayers with 25, 37, 50, 63 and 75 vol% tungsten [11,12,16,17]. The total coating thickness of
38 2 mm was chosen to match the value used in current design studies of First Wall elements [18–20]. It
39 represents a compromise between high thickness required for heat shielding and low thickness required for
40 sufficiently high tritium breeding ratio of the reactor [21,22]. The 1.2 mm thickness of the functionally graded
41 material was found by means of finite element simulations to minimise creep strains and thus increase the
42 allowed number of thermal cycles [9].

43 These coatings were recently proven to withstand fusion-relevant heat loads [16]: A helium-cooled mock-
44 up with such coatings was successfully tested in the HELOKA facility (Karlsruhe Institute of Technology,
45 Germany), using 1000 cycles with an electron beam of 0.7 MW/m² average heat flux density and with
46 heating and cooling times of 180 s and 150 s, respectively, during each cycle. The surface temperature of
47 the coating remained below 800°C, the substrate temperature below and mechanical properties of the
48 substrate were not diminished after testing [16]. Additionally, smaller samples with similar coating were
49 subjected to thermal shock tests in the JUDITH 1 facility (Forschungszentrum Jülich, Germany) to test the
50 coating under transient heat loads comparable to edge localised modes, using 100 thermal shock pulses
51 with a focussed electron beam, a single pulse duration of 1 ms and testing at base temperatures of both
52 room temperature and 550°C. The resulting thermal shock threshold of the coating was found between 0.19
53 and 0.38 GW/m² for both base temperatures [23]. Furthermore, these coatings withstood 5000 thermal
54 cycles between 300 and 550°C, applied by alternating inductive heating and purge gas cooling, without
55 showing signs of thermal fatigue [24].

56 Here we present a further development of these vacuum/low pressure plasma sprayed coatings. The
57 aforementioned mock-up has reached the size limit (approx. 300x200 mm²) that could be coated on
58 laboratory scale [16]. However, further upscaling with industrial manufacturers is required to coat the metre-
59 sized breeding blanket modules designed for DEMO [2]. At the same time, the European Research
60 Roadmap to the Realisation of Fusion Energy foresees a transfer of technological know-how to industrial
61 companies in order to create an infrastructure for manufacturing parts for fusion power plants when needed
62 [2,25]. To this end, we present the first results of such a technology transfer. Samples of three different sizes
63 (50x50 mm², 300x200 mm², 500x250 mm²) have been coated by low pressure plasma spraying (LPPS) to
64 transfer the technological know-how of FGM production and to test the feasibility of coating larger plates,
65 including a new upscaling record for this coating technology (500x250 mm²). The total layer thickness of
66 2 mm and the linear chemical gradient over five interlayers have been maintained. This work presents a
67 quality inspection of all sample sizes, including distortion analysis and ultrasonic testing for delamination.
68 Furthermore, a microstructure analysis was conducted based on the 50x50 mm² samples.

69 2. Materials and methods

70 2.1 Substrate, powder and coating process

71 Three sample sizes to be coated were prepared: Small sample blocks of 50×50×20 mm³ were chosen for
72 the initial technology transfer and the microstructure analysis reported here. Medium-sized plates of
73 300×200×20 mm³ posed an intermediate milestone as their area resembles the largest area to which this
74 coating technology was successfully applied in a laboratory [16]. Large plates of 500×250×20 mm³ were
75 envisioned to achieve a further upscaling step towards the large areas to be coated for the breeding blanket
76 of the DEMO fusion reactor [2,26–28].

77 The coatings were required to meet specifications equal to those of previous development steps
78 [11,12,16,17], i.e. a total coating thickness of 2 mm +- 10%, consisting of 0.8 mm of tungsten top coat and
79 1.2 mm of functionally graded material. The FGM was required to consist of five interlayers of 240 µm
80 thickness each and with respective tungsten contents of 25, 37, 50, 63 and 75 vol%.

81 As substrate material, P92 steel (1.4901) was chosen rather than the reduced-activation ferritic-martensitic
82 steel EUROFER97 envisioned for DEMO [26,29] because of the limited availability of EUROFER97 [30] and
83 the large size of plates to be coated here. P92 is a ferritic-martensitic steel with a chemical composition
84 close to that of EUROFER97 (Table 1) [31–34] and finds use in high-temperature-high-pressure scenarios
85 of fossil power plants [35,36] as well as in the construction of mock-ups for the DEMO reactor [30]. When
86 compared to P91 steel, another frequently used substitute for EUROFER97, P92 shows improved
87 mechanical performance, especially under creep conditions [36–38].

88 *Table 1. Main elements (in wt%) of P92 and EUROFER97 steels [31,33,34].*

	C	Cr	W	Mn	Mo	V	N	Fe, others
P92	0.07 – 0.13	8.5-9.5	1.5-2.0	0.3-0.6	0.3-0.6	0.15-0.25	0.03-0.07	bal.
EUROFER97	0.09-0.12	8.5-9.5	1.0-1.2	0.2-0.6	<50 ppm	0.15-0.25	0.015-0.045	bal.

89 The P92 substrates were manufactured from rolled sheet (thickness 50 mm) by water jet cutting, sawing
90 and milling. The workpieces underwent a heat treatment as specified in the P92 guidance [33]:
91 Normalisation at 1090°C for 90 minutes, cooling to 170°C and holding for five minutes, then tempering at
92 760°C for 90 minutes, followed by cooling to room temperature. All heating cooling procedures were
93 conducted at a rate of 5-10 K/min. Afterwards, oxide layers were removed by glass bead blasting and the
94 workpieces were milled to their final dimensions and mock-up cooling channels were drilled in the
95 500×250 mm² and 300×200 mm² plates. In the back sides of the 50×50 mm², M8 holes were drilled as
96 bearings for the coating process. The cooling channels in the plates were introduced with regard to future
97 first wall panels, in order to investigate how the presence of cooling channels affects process heat
98 management during coating as well as coating adhesion. Here, a simple cylindrical drill holes were selected
99 as channels rather than the elaborated electric-discharge machining design for DEMO first wall panels [3,27]
100 because of the significantly reduced costs.

101 The EUROFER powder used in the coating process was procured from the company NANOVAL GmbH &
 102 Co. KG (Berlin, Germany). It was fabricated by spray aeration of a melt with argon as described in [16],
 103 followed by sieving to reduce the amount of fine particles to prevent the risks of clogging and metal fire. The
 104 mean particle diameter was $d_{50} \approx 29.8 \mu\text{m}$. The tungsten powder (specified purity >99.8 wt% excl. oxygen)
 105 was procured from Haines & Maassen Metallhandelsgesellschaft mbH (Bonn, Germany) and had reduced
 106 particle size ($d_{20} \approx 19 \mu\text{m}$, $d_{90} \approx 38 \mu\text{m}$) compared to the one used in [16] to reduce the amount of unmelted
 107 particles in the coating.

108 The coatings were manufactured by the company COATEC GmbH (Schlüchtern, Germany). In terms of the
 109 process, low pressure plasma spraying was applied instead of VPS because of availability and expertise
 110 provided by the partner. The applied pressure during LPPS (40 mbar), however, was similar to the pressures
 111 previously used (60-140 mbar) in vacuum plasma spraying [12]. The coating setup comprised two plasma
 112 guns, one for spraying, equipped with four powder feeders (two for tungsten, two for EUROFER) and a
 113 second gun for heat transfer optimisation. The plasma guns were operated with argon, with minor amounts
 114 of He and H₂ as secondary gases for improved heat transfer. An overview of spraying parameters is
 115 provided in Table 2.

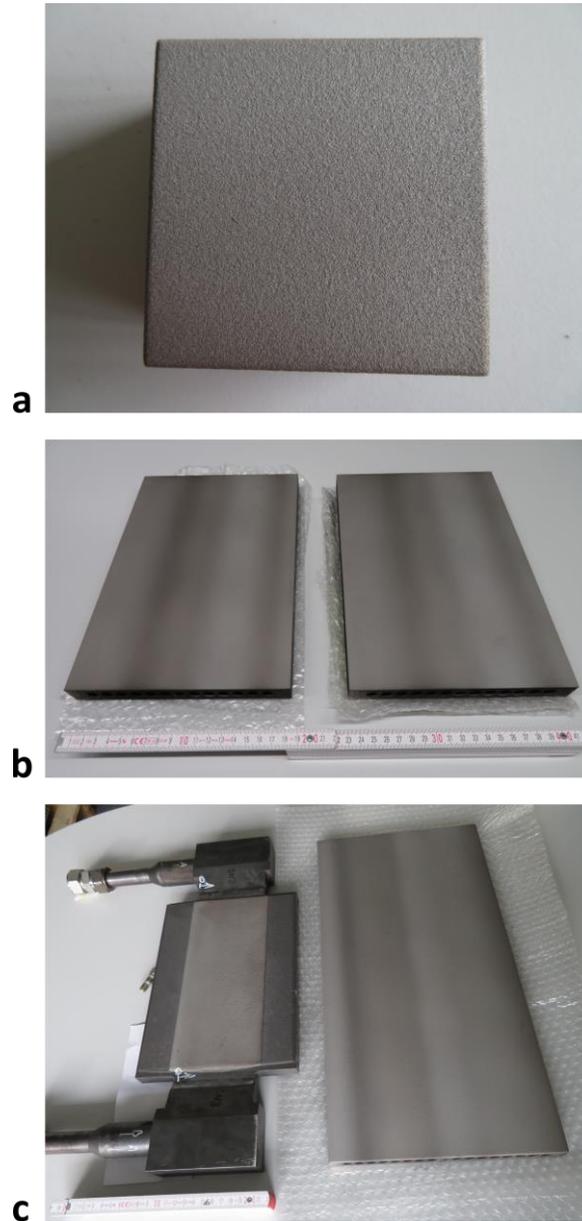
116 *Table 2. Coating parameters used for low pressure plasma spraying.*

Substrate size (mm ²)	50x50	300x200	500x250
Current (A)	1300	1300	1350
Power (kW)	84.5	84.1	84.2
Voltage (V)	65	64.7	62.4
Primary gas Ar (SLPM)	93	93	93
Secondary gas He (SLPM)	10	10	10
Secondary gas H ₂ (SLPM)	12	12.5	13
Feed gas for EUROFER (SLPM)	7	7	7
Feed gas for tungsten (SLPM)	9.2	9.2	9.2
Chamber pressure (mbar)	40	40	40
Spraying distance (mm)	300	300	300
Traverse speed (m/min)	7.5	7.5	7.5
Substrate temperature (°C) (approx.)	695-752	727-758	735-757 (centre position) 677-730 (corners)

117 The substrate temperature in Table 2 was surveyed on test samples during the development of optimised
 118 coating parameters. The test samples were of size equal to that of the final samples and the thermocouples
 119 used were placed in holes drilled from the back side of the parts. For the 500x250 mm² plates, temperature
 120 was measured in the middle of the plate as well as in two opposite corners.

121 The powder feeding rates ranged from 18 to 67 g/min for W and from 0 to 111 g/min for EUROFER,
 122 depending on the respective interlayer of the functionally graded material. The total powder usage was

123 60 kg of EUROFER (of 230 kg sent to manufacturer for cushion purposes) and 100 kg of tungsten powder
124 (of 450 kg sent to manufacturer).
125 In total, ten 50x50 mm² blocks, two 300x200 mm² plates and two 500x250 mm² plates were delivered with
126 coating (Figure 1). Specimens for a metallographic assessment of coating quality were cut from four of the
127 ten 50x50 mm² blocks by electric discharge machining and polished by standard metallographic means with
128 Ø 0.1 µm diamond suspension as last step.



129
130 *Figure 1. (single column image) (a) Close-up top view of the industrial coating on a 50x50 mm² block. (b) Industry-*
131 *coated 300x200 mm² plates with cooling channels. (b) Industry-coated 500x250 mm² plate with cooling channels (right*
132 *side) compared to largest laboratory-coated mock-up (left side).*

133 **2.2 Microscopy and EDX**

134 The total thickness of the coating was determined on as-cut samples by optical microscopy (VHX-1000
135 digital microscope, Keyence, Osaka, Japan). The coating-substrate interface was readily discernible even
136 on unpolished samples. For the coating thickness, four 50×50 mm² substrates and six specimens per
137 substrate were evaluated. For each specimen, 15 thickness measurements were taken over its width, giving
138 a total of 360 evaluated thickness measurements. The thickness of the individual interlayers of the
139 functionally graded (FG) coating was not easily distinguishable by optical microscopy. Instead, polished
140 specimen prepared by standard metallographic procedures were investigated by scanning electron
141 microscopy (SEM, EVO MA10, Zeiss, Oberkochen, Germany, equipped for energy-dispersive X-ray
142 spectroscopy with XFlash detector 410-M, Bruker Nano GmbH, Berlin, Germany) for layer thickness as well
143 as microstructure analysis. Here, one specimen from each of four investigated 50×50 mm² blocks was
144 evaluated. For each specimen, three secondary electron images were taken with an acceleration voltage of
145 20 kV and a working distance of 10 mm. For each SEM image, three to five thickness measurements per
146 interlayer were taken, giving a total of at least 36 thickness measurements per interlayer. The accuracy for
147 correctly determining individual interfaces between layers is estimated to be in the same order of magnitude
148 as the standard deviation of the measurements.

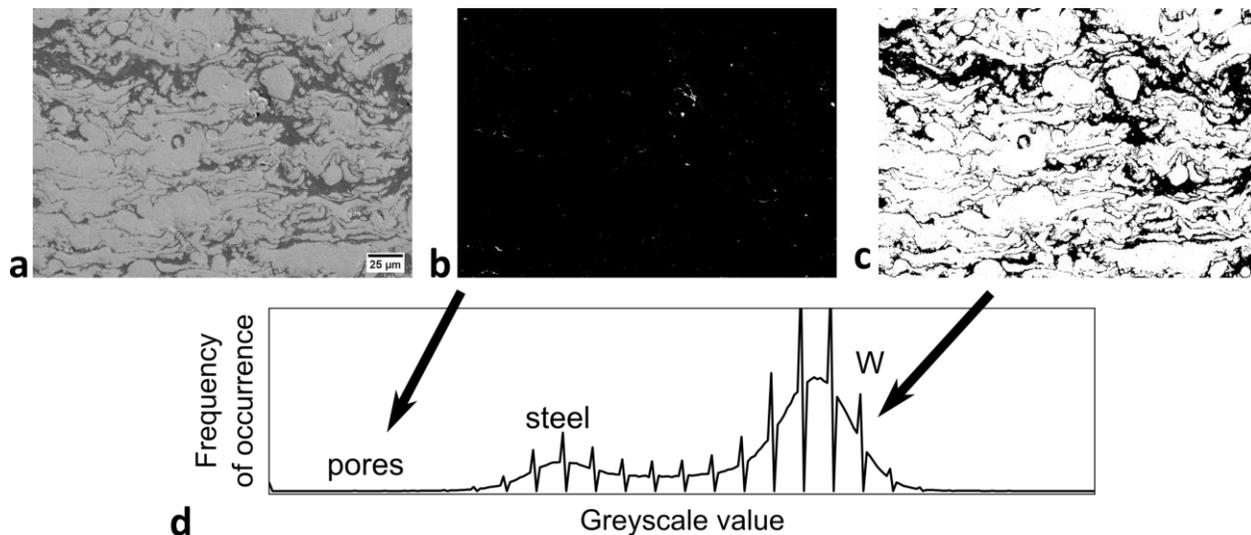
149 For elemental analysis by energy-dispersive X-ray spectroscopy (EDX), five pieces taken from four of the
150 50×50 mm² blocks were investigated, with a working distance of 13 mm. For each coating layer an EDX
151 multi point analysis covered 30×30 points distributed over an area of about 100×100 μm².

152 **2.3 Thresholding analysis of porosity and W content**

153 A thresholding analysis of SEM cross sections was applied to quantify the porosity and tungsten content of
154 the coatings. Five specimens were evaluated, taken from four different 50×50 mm² blocks, using the open-
155 source software distribution Fiji / ImageJ [39]. For each specimen and each coating layer, three secondary
156 electron images were evaluated. A magnification of 1060× allowed to cover representative views of each
157 layer without the risk of imaging more than one interlayer simultaneously (image height approx. 200 μm,
158 interlayer thickness approx. 240 μm). The porosity in each image was determined by thresholding analysis
159 of its grey scale values, with pores corresponding to lowest grey scale levels, i.e. darkest pixels (Figure 2).
160 Since none of the built-in thresholding algorithms in Fiji / ImageJ was able to identify the pores in a reliable
161 way, the threshold was adjusted manually, following the practice described in ref. [40]: For each image, the
162 grey scale value at which the frequency of occurrence starts to increase rapidly marks the onset of non-
163 pore material. For the analysis of tungsten content, the grey scale threshold was set to separate W phase
164 (highest grey scale values, see Figure 2d) from steel phase (medium grey scale) and pores (low grey scale).
165 In this case, the standard thresholding algorithm of Fiji / ImageJ, the “isodata” algorithm [41], reliably
166 identified the W phase.

167 The thresholding analysis yielded the area fraction of pores and tungsten particles in each image, which for
168 isotropic shape equals the volume fraction of pores and tungsten particles in the FGM. As both can be of

169 non-isotropic, flat shape (Figure 6b), the determined area percentages are merely an approximation of the
170 real volume fractions.



172 *Figure 2. (double column image) Thresholding analysis example: (d) shows the grey scale histogram for the input image*
173 *(a). (b) Pores (here white) are isolated by selecting only the darkest grey scale values, left of the “steel” peak in (d). (c)*
174 *Selecting the brightest grey scale values - the “W” peak in (d) - isolates the tungsten particles.*

175 2.4 Ultrasonic immersion analysis

176 All received samples were investigated by ultrasonic immersion analysis in water to search for potential
177 delamination of the coating.

178 The facility used was a KC 200 Immersion testing facility, equipped with USIP40 testing device and KScan
179 evaluation software (GE Inspection Technologies, Hürth, Germany) as described in [42]. A 10 MHz
180 ultrasonic probe was used and all analyses were conducted under 90° angle of incidence with longitudinal
181 sound waves. Data were evaluated either in the form of A-scans (1D plots of echo amplitude versus location
182 of echo source under the surface) or C-scans (2D compilation of A-scan amplitude information for a sample
183 area within a pre-selected depth). To compute the location of an echo source from the sound impulse run
184 time, a sound velocity needs to be provided to the system. For inhomogeneous materials such as FGM, the
185 calculated depth locations may therefore differ slightly from real positions, which is of no importance when
186 searching for delamination though. When the coated front side pointed towards the probe, an averaged
187 sound velocity of 5416 m/s was chosen for the functionally graded coating as the result of averaging the
188 sound velocities of EUROFER97 (5920 m/s) [42] and tungsten (5200 m/s) [16,43], weighted by their
189 respective volume fractions of the coating. For measurements with the uncoated back side pointing upwards
190 towards the probe, the sound velocity of P92 steel (5775 m/s) was taken [35,44].

191 **3. Results and discussion**

192 **3.1 Visual inspection and distortion analysis**

193 The industrial coatings showed good homogeneity upon first visual inspection (Figure 1a). A darker colour
194 as seen in stripes on all four larger plates partially coincided with a slightly increased roughness and is
195 attributed to unmelted W particles on the surface. Even so, the visible surface roughness of all industry
196 coatings was lower than the roughness of previous laboratory-produced coatings [11,12,16].

197 A potential side effect of thermal spraying is the distortion of coated parts, occurring during cooling of the
198 coating. This effect tends to be more severe for larger parts and therefore requires attention during
199 development of coatings for the breeding blanket. A distortion analysis of the 300×200 mm² and
200 500×250 mm² plates was conducted at the company topometric GmbH (Göppingen, Germany). For each
201 of the four plates, a 3D image of the uncoated back side was measured by optical triangulation to find
202 aberrations from the ideal surface. Additionally, this company has conducted a roughness analysis of the
203 coating of one of the 500×250 mm² plates.

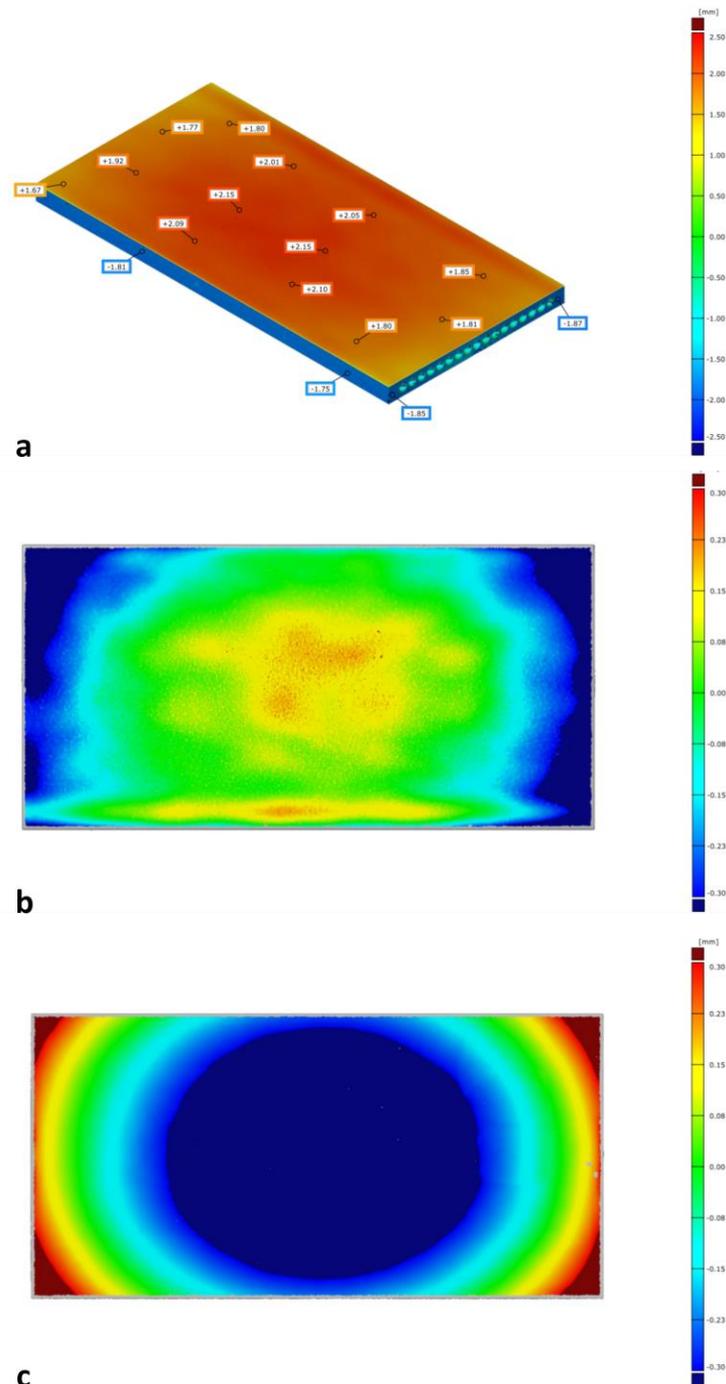
204 The distortion analysis revealed small but clear warping of all plates after coating. An example for a
205 500×250 mm² plate is shown in Figure 3c. The corners of the uncoated back sides all have moved
206 downwards (red colour in Figure 3c) while the plate centres have moved upwards (blue colour in Figure 3c).
207 This can be explained by the larger coefficient of thermal expansion of the steel substrate when compared
208 to the tungsten [9]. As the coating shrinks less upon cooling, it locally hinders a uniform shrinkage of the
209 plate, resulting in warping of the plate with corners pressing downwards. Quantitatively, however, this
210 warping is small. The maximum height difference between corners and centre (approx. 0.4 mm for the
211 300×200 mm² plates and 0.6 mm for the 500×250 mm² plates) is of the same order of magnitude as the
212 tolerance for planarity applied during manufacturing of the substrate plates.

213 On the other hand, the deviation of the side faces from ideal geometry indicates a shrinkage of all plates
214 within the substrate plane. An example for a 500×250 mm² plate is shown in Figure 3a. This deviation is in
215 the order of 1-2 mm, regardless of the plate size, and thus is, in the worst case, in the order of 0.4-1% of
216 the respective length dimensions. It is considered small but not negligible with future upscaling in mind. The
217 order of magnitude of distortion, both in warping and shrinkage, is in agreement with the one found with
218 previous finite element simulations of coated plates with comparable size [45].

219 The roughness analysis of the coated front side (Figure 3a,b) revealed only minor height deviations, mostly
220 within the specified coating thickness of 2 mm ± 10%.

221 The 2 mm offset in Figure 3a results from comparing the 2 mm thick coating with the CAD model of an
222 uncoated plate. Lowest surface heights were found at two opposite plate ends (blue colour in Figure 3b).
223 The reduced height of about 0.3 mm at these two ends roughly coincides with the downwards warpage
224 found during measurement of the back side and is thus regarded as another sign of the warpage, rather
225 than actual reduction of coating thickness. Peak roughness of about 0.3 mm above rest of the surface (red
226 colour in Figure 3b) was found in the middle of the plate and in the middle of one stripe at the lower end,

227 the middle position in line with the upwards movement caused by warpage. This peak roughness is not
 228 homogeneous but caused by single, larger particles presumably from the side regions of the plasma plume
 229 [46]. While the stripe in Figure 3b coincides with a dark stripe found during visual inspection, other dark
 230 stripes had no obvious counterpart in the roughness analysis.

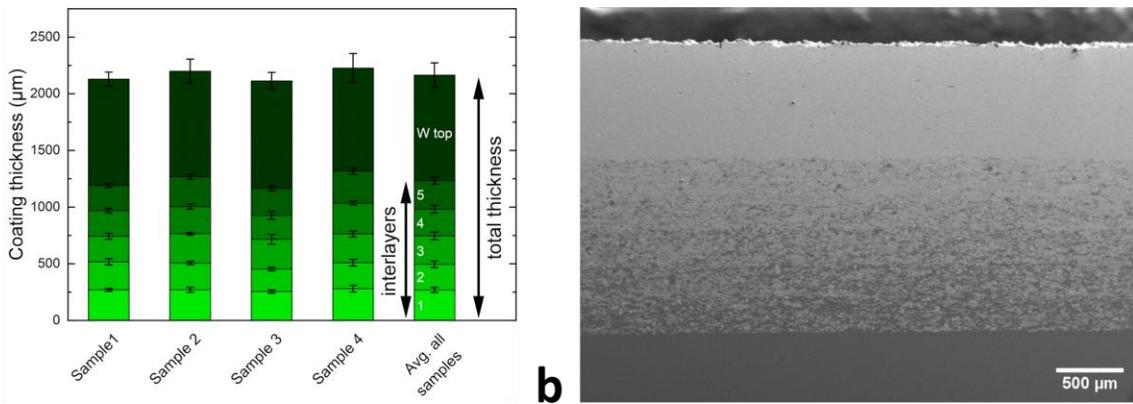


231 **c**
 232 *Figure 3. (single column image) Distortion and roughness analysis for 500x250 mm² plates, conducted by topometric*
 233 *GmbH. (a) Deviations of front and side faces from ideal position. The 2 mm offset of the front face is the target thickness*
 234 *of the coating. (b) Height profile of the coated front side. (c) Warpage of the back side. (a) and (b) refer to the same*
 235 *plate while (c) shows a different plate that displayed the largest warping.*

236 **3.2 Thickness of coating and interlayers**

237 A visualisation of the mean thickness values is shown in Figure 4a. Error bars show the standard deviation
238 of thickness for the entire coating and the five FGM interlayers. Figure 4b shows a SEM cross section of the
239 coating for comparison. The total coating thickness of the samples met the targeted value of 2 mm \pm 10 %.
240 The coating always was slightly thicker than 2 mm because of increased thickness of the W top layer
241 (approx. 900 μ m instead of 800 μ m). The average thickness of the five FG interlayers met the targeted
242 240 μ m for all interlayers, within a margin of \pm 10 %.

243

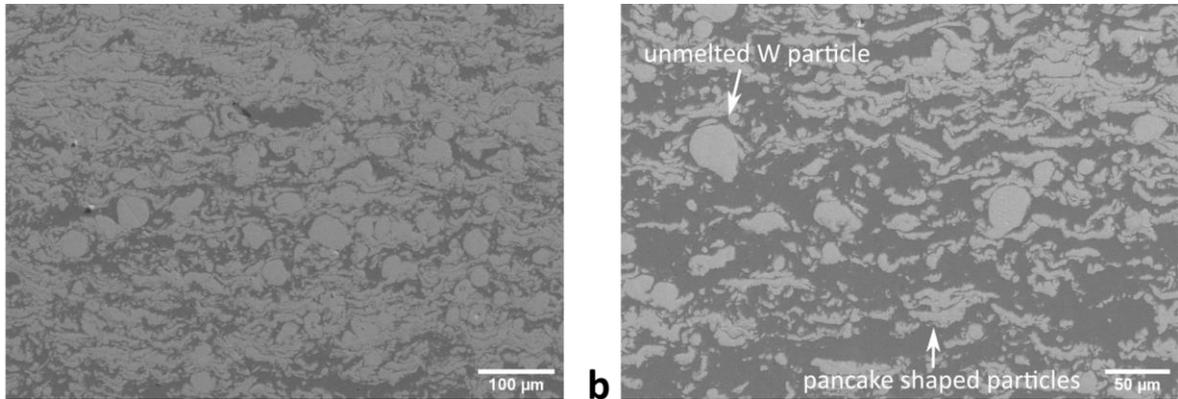


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245 *Figure 4. (double column image) (a) Layer thickness of four 50x50 mm² samples. (b) SEM BSE image of a coating*
246 *cross section.*

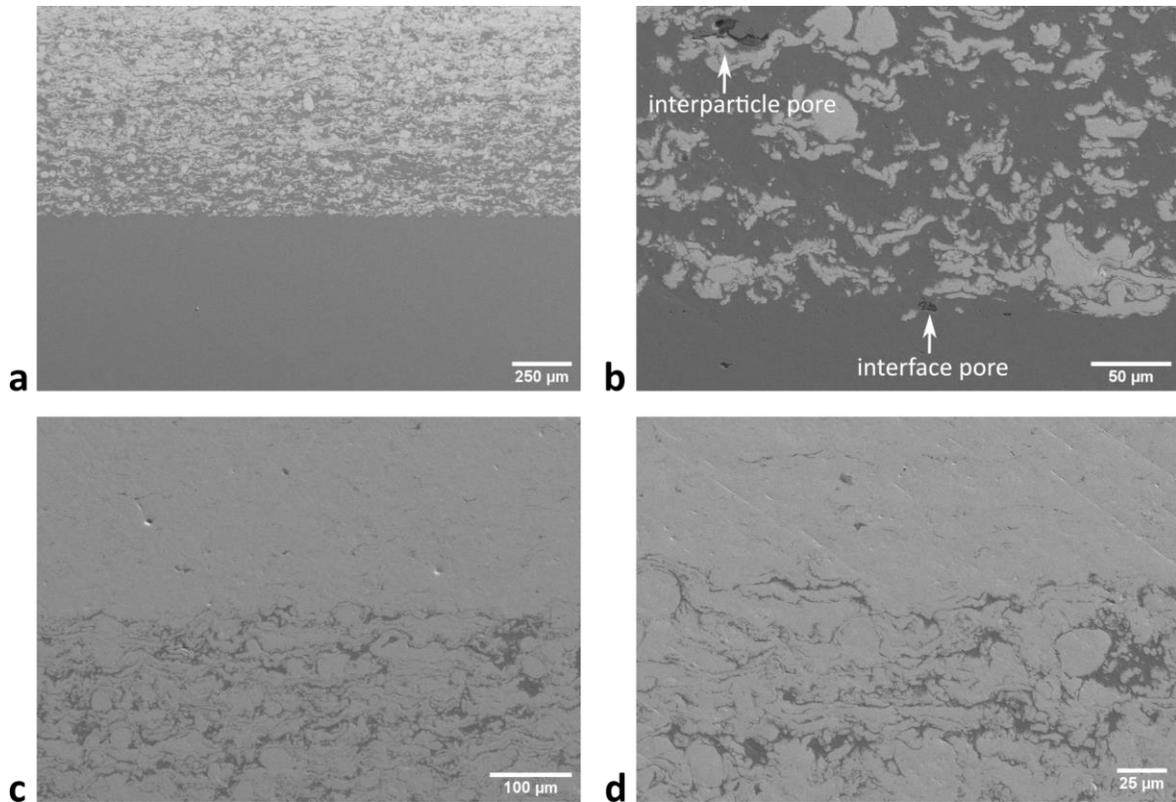
247 **3.3 Coating microstructure**

248 The SEM analysis of the 50x50 mm² blocks showed a microstructure built by stacking of “pancake shaped
249 particles”, as is typical for plasma sprayed coatings. Typical SEM cross sections are shown in Figure 5. The
250 pancake shaped particles measured about 5-10 μ m in thickness and 30-60 μ m in diameter. Occasionally,
251 round tungsten particles with a diameter of about 30-50 μ m, which did not melt during the coating process,
252 were also found (Figure 5a). Both particle types are highlighted in Figure 5b. This microstructure resembles
253 the one of the previous, laboratory-produced coatings [11,17].



254 **a** **b**
255 *Figure 5. (double column image) SEM cross sections of the coating showing a typical microstructure of stacked, pancake*
256 *shaped particles. Larger, round particles as visible in the middle of (a) and highlighted in (b) are unmelted tungsten*
257 *particles.*

258 The interface between coating and substrate (Figure 6a) appears like a sudden onset of tungsten particles
259 within a steel matrix upon magnification (Figure 6b), with the coating steel particles being indistinguishable
260 from the steel substrate. This indicates the establishment of metallic bonding to the substrate, as found
261 previously for laboratory-produced coatings [45,47]. Interface pores as well as interparticle pores within the
262 coating (Figure 6b) were found only in minor amounts. A quantitative assessment of bond strength at the
263 interface will be subject to future study. The interface between FGM and W top layer has a similar
264 appearance as the interface to the substrate (Figure 6c,d) and shows almost no porosity. Within the W top
265 layer, boundaries between single W particles are occasionally visible (top of Figure 6d). They presumably
266 represent minor porosity or stem from leftover steel feedstock, since oxide layers, as a potential alternative
267 [14], are unlikely to form during the vacuum process.



268 **c** **d**
 269 *Figure 6. (double column image) SEM cross section of the interfaces between (a,b) coating and substrate and (c,d) W*
 270 *top layer and FGM. (b,d) show magnified views. Minor porosity is highlighted in (b).*

271 **3.4 Porosity and chemical gradation**

272 Porosity may influence mechanical and thermal properties [48,49] of the coatings as well as their hydrogen
 273 permeability [50].

274 The porosity of each coating layer, estimated by thresholding analysis, is listed in Table 3 and was found to
 275 be approximately 0.1 % for the FG interlayers and 0.5 % for the W top layer. These values are very low
 276 when compared to the porosities of 1-5 % found in the laboratory-produced coating [17]. Although this could
 277 indicate a high coating quality, the porosity of plasma sprayed coatings more typically is in the range of
 278 several percent [48]. Therefore, the porosity values should be treated with caution. The pores of the cross
 279 sections may potentially be clogged with material from metallographic preparation that was not sufficiently
 280 removed. However, thorough ultrasonication did not reveal additional pores. We note though that an earlier
 281 study of plasma-sprayed tungsten (without FGM) has found a similarly low porosity of 0.6 % [49].

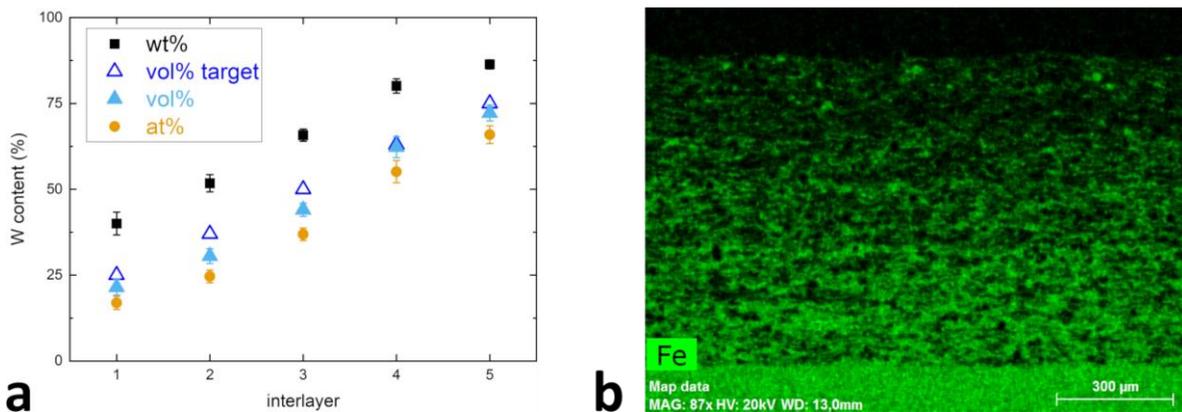
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283 Table 3. Porosity and W content from image thresholding analysis.

Coating layer	porosity (area%)	W content (area%)
total	0.17 ± 0.19	-
W top layer	0.51 ± 0.22	-
interlayer 5	0.12 ± 0.07	80.6 ± 2.2
interlayer 4	0.10 ± 0.07	71.6 ± 2.7
interlayer 3	0.08 ± 0.06	60.3 ± 2.7
interlayer 2	0.08 ± 0.05	47.8 ± 4.9
interlayer 1	0.12 ± 0.14	33.6 ± 4.0

284 In order to evaluate the tungsten content of the FG coating's interlayers, energy-dispersive X-ray
 285 spectroscopy (EDX) was compared with a thresholding analysis. The thresholding results are listed in Table
 286 3 while the EDX results are visualised in Figure 7a. Figure 7b shows an EDX map of the coating's iron
 287 content which confirms and visualises the chemical gradation of the coating. According to EDX results, the
 288 tungsten content of the two uppermost interlayers (4 and 5) meets the target value while in the lower three
 289 interlayers (1 to 3) the W content is about 4 to 7 vol% too low. This is a good result considering the imprecise
 290 nature of the coating process where W and steel powders have to be provided to the spray gun by different
 291 feeders.

292 The tungsten content as found by thresholding analysis exceeded the target value by 6 to 11 % per layer.
 293 This may be explained by the challenge of a 2D slicing analysis of 3D objects and the irregular shape of W
 294 particles. The EDX results are considered more reliable. However, both approaches independently
 295 confirmed the chemical gradation of the FG coating.

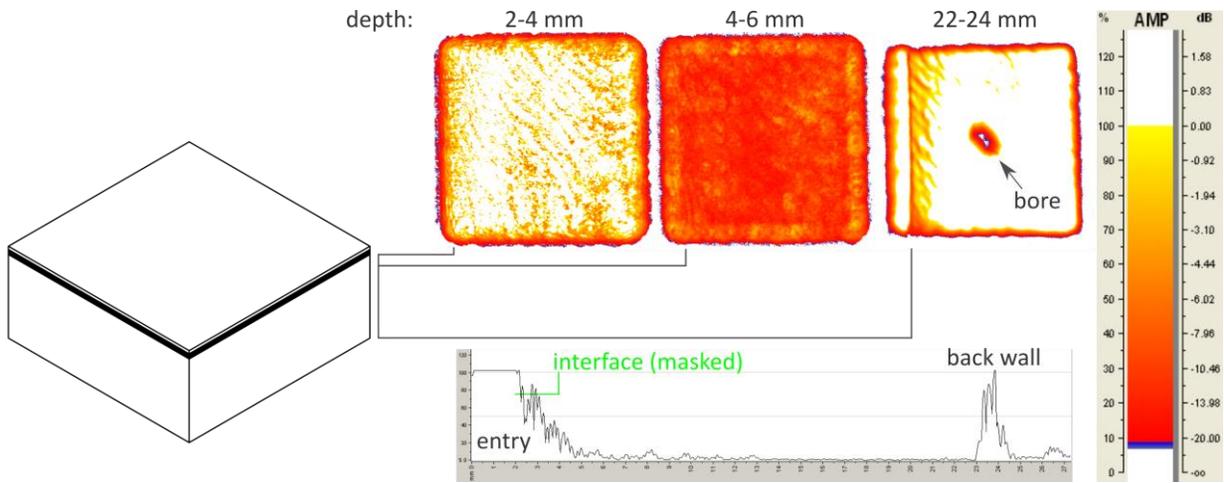


296 **a** **b**
 297 Figure 7. (double column image) EDX analysis of the five FG coating interlayers. (a) Tungsten content of the interlayers.
 298 (b) EDX map showing iron content (green) increasing from the W top layer (black, top) towards the substrate (bottom).

299 3.5 Ultrasonic immersion analysis

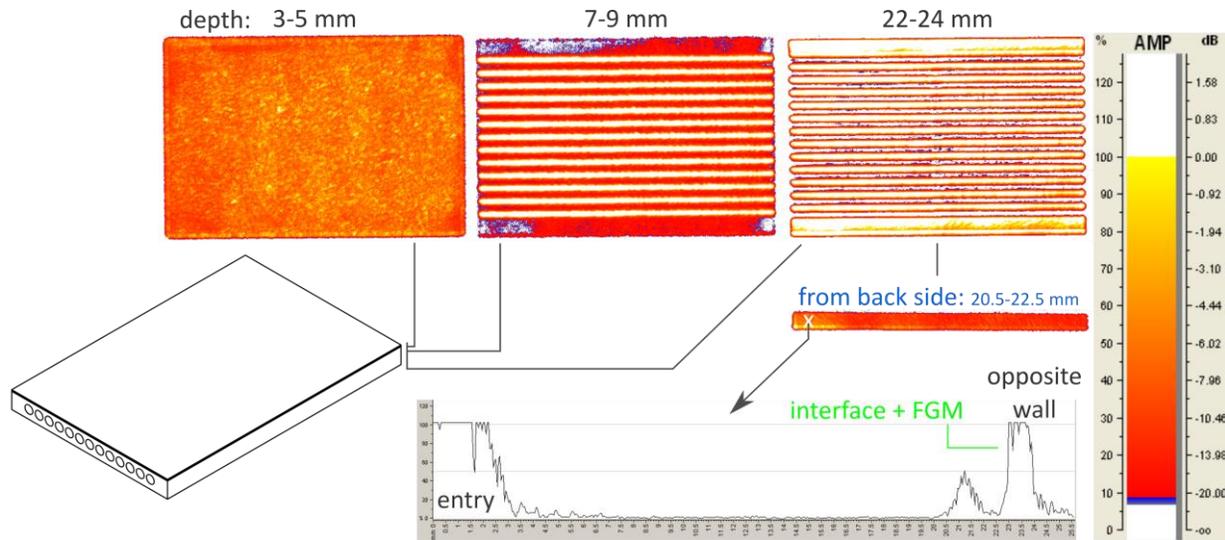
300 Each sample was investigated by ultrasonic analysis. In short, no delamination was detected. However,
 301 inspection of the larger plates provided hints to potential weak spots in the corners. This section presents
 302 the results from one ultrasonic analysis per sample size (Figures 8-10).

303 Figure 8 shows the results for a 50x50 mm² block, displaying C-scans of the accumulated signal for the
 304 regions at 2-4, 4-6 and 22-24 mm below the coating surface. Deviations from sample thickness (20 mm +
 305 2 mm coating) arise from using averaged sound velocity.



306
 307 *Figure 8. (double column image) Ultrasonic analysis of 50x50 mm² block showing C-scans (top) from three different*
 308 *depths (2-4, 4-6 and 22-24 mm below coating surface) and an A-scan (bottom) displaying typical entry and back wall*
 309 *echos. C-scan colour displays signal amplitude. The A-scan does not clearly show the echo from the coating-substrate*
 310 *interface since it overlaps with the entry echo. C-scans for 2-4 and 4-6 mm depth show homogeneous distribution and*
 311 *signal decay of the entry echo. The C-scan for 22-24 mm depth displays evenly distributed back wall echo except for a*
 312 *bore in the middle.*

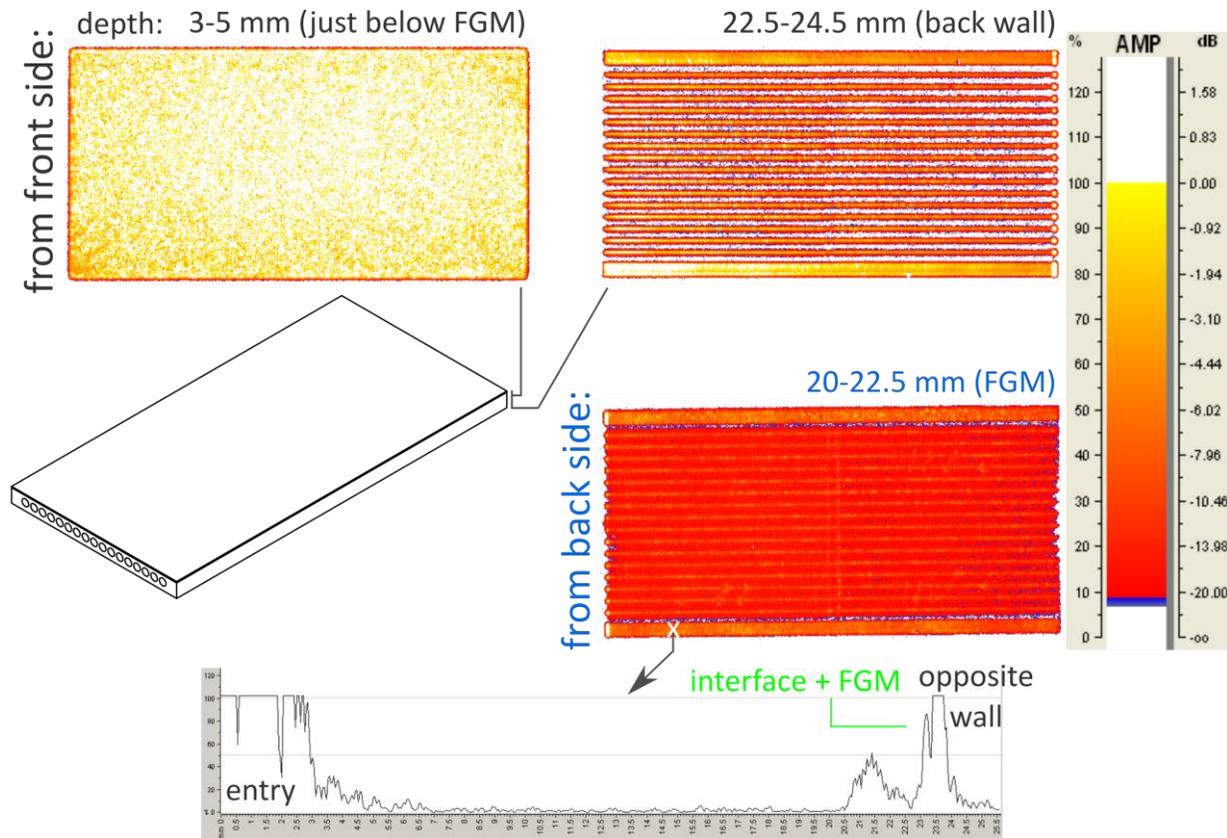
313 The coating-substrate interface at 2 mm depth is not clearly discernible from individual A-scans (Figure 8
 314 bottom), as it is masked by a broad entry echo. However, the C-scans for 2-4 and 4-6 mm depth show
 315 homogeneous distribution of the entry echo as well as an evenly distributed signal decay towards greater
 316 depths. This indicates good adhesion of the coating over the entire block. The same conclusion may be
 317 drawn from the homogeneous back wall echo in the C-scan at 22-24 mm depth. Any inhomogeneity here
 318 would suggest masking by defects above, but was only found for the bore in the middle of the block. The
 319 distinct, vertical line at the left edge at 22-24 mm depth and the round edges in all C-scans are artefacts
 320 from the ultrasound passing the sample edges.



321
 322 *Figure 9. (double column image) Ultrasonic analysis of 300x200 mm² plate showing C-scans (top) from three different*
 323 *depths (3-5, 7-9 and 22-24 mm below coating surface). C-scan colour displays signal amplitude. The C-scan for 3-5 mm*
 324 *depth shows homogeneous decay of entry echo except for some regions on the edges with weaker signal that become*
 325 *more visible at 7-9 mm depth, along with the bores. At 22-24 mm depth, the back wall echo is visible everywhere except*
 326 *behind the bores, but is not completely homogeneous at the edges. The additional narrow C-scan shows the lower edge*
 327 *measured from the back side and focussed to the depth where the interface+FGM signal is expected, as indicated in*
 328 *the adjacent A-scan (bottom).*

329 The ultrasonic analysis of the 300x200 mm² plates, too, indicates coating adhesion over the entire area.
 330 Here, however, the adhesion may potentially be weaker at the edges. Figure 9 shows the results for one of
 331 the two plates. The displayed C-scans in the top line show the accumulated signal for the regions at 3-5,
 332 7-9 and 22-24 mm below the coating surface. Additionally, a C-scan of one sample edge, measured from the
 333 back side instead of the coated front side is shown (Figure 9 bottom). The C-scan for 3-5 mm depth shows
 334 homogeneous decay of the entry echo except for some regions on the edges. These regions have a weaker
 335 signal, i.e. a locally enhanced decay of the entry echo, and become more visible at 7-9 mm depth. Here,
 336 also the bores become clearly visible. At 22-24 mm depth, the back wall echo is visible everywhere except
 337 behind the bores, suggesting that no delamination occurred at the edges since that would otherwise mask
 338 the back wall echo. However, the back wall echo is not evenly distributed along the edges. If no delamination
 339 occurred, this uneven distribution could be interpreted as differences in adhesion strength along the edges.
 340 While echos from FGM and coating-substrate interface are masked in scans of the coated front side, they
 341 are clearly visible when measuring from the back side. In Figure 9, the narrow C-scan of the sample edge
 342 measured from the back side is focussed on the depth region 20.5-22.5 mm behind the back wall, i.e. where
 343 the interface and FGM echos were found. The 0.5 mm offset here may be caused by difficulties of finding
 344 the zero in the broad entry echo or by deviation of the used sound velocity. This back side C-scan, like the
 345 ones taken from the front side, shows inhomogeneous signal amplitude along the sample edge. Apparently,
 346 the interface echo is stronger in certain regions. For one of these regions (marked by the white X in Figure
 347 9) the corresponding A-scan is displayed (Figure 9 bottom). This A-scan reveals an echo in the region of

348 coating-substrate interface and FGM. Behind this echo, the “opposite wall” echo (here stemming from the
 349 coating surface) is at full amplitude, indicating that no delamination occurred. As stated above, the uneven
 350 distribution of the FGM echo may be caused by local variation of adhesion strength. Alternatively, the local
 351 amplitude variation along the sample edge could also be caused by locally increased porosity within the
 352 FGM, higher amounts of unmelted particles or other inhomogeneities that interfered with the ultrasonic
 353 impulses. Also, the amplitude variation in the corners may be partially caused by the slight bending of the
 354 plates (Figure 3), since the ultrasonic analysis assumes a fixed zero position.



355
 356 *Figure 10. (double column image) Ultrasonic analysis of 500x250 mm² plate, showing two C-scans measured from the*
 357 *front side (top line, 3-5 and 22.5-24.5 mm below coating surface) and one C-scan measured from the back side (middle).*
 358 *C-scan colour displays signal amplitude. The C-scan for 3-5 mm depth shows homogeneous decay of entry echo except*
 359 *for corner regions. At 22.5-24.5 mm depth, the back wall echo is visible everywhere except behind the bores. Gradient*
 360 *of back wall echo from lower left to upper right corner is caused by uneven positioning of plate. The C-scan measured*
 361 *from the back side is focussed to the depth where the interface+FGM signal is expected (20-22.5 mm), as indicated in*
 362 *the adjacent A-scan. The echo from interface+FGM does not show interruptions except for where it is masked by the*
 363 *bores.*

364 The ultrasonic analysis of the 500x250 mm² plates revealed a coating quality similar to the one found for
 365 the 300x200 mm² plates. As displayed in Figure 10 for one of the two plates, when measured from the front
 366 side, the entry echo showed a homogeneous decay over the entire sample area, except for a slightly
 367 stronger decay in the corners (top left C-scan of Figure 10, focussed at depth 3-5 mm below coating
 368 surface). A back wall echo was found over the entire plate area except for where it was masked by the bores

369 (top right C-scan of Figure 10, focussed at depth 22.5-24.5 mm below coating surface). Therefore, a coating
370 delamination can be excluded for all areas without bores. An apparent intensity gradient of the back wall
371 echo from lower left to upper right corner of the plate is assigned to a slightly uneven positioning of the plate
372 inside the ultrasonic bath. The assumption of homogeneous coating adhesion is supported by a
373 measurement from the back side of the plate (C-scan in middle of Figure 10), which covers a depth 20-
374 22.5 mm from the back side, i.e. where the FGM and the coating-substrate interface are expected. Their
375 echo is evenly distributed over the plate area, except for where it is masked by the bores. The A-scan at the
376 bottom of Figure 10 (taken on the edge position marked by a white X in the back side C-scan) shows the
377 echo from FGM and coating-substrate interface. Behind this echo, the “opposite wall” echo (coating surface)
378 is at full amplitude, indicating that no delamination occurred. In contrast to the 300×200 mm² plate in Figure
379 9, the back side measurement in Figure 10 did not reveal an increased FGM echo in the corners, where the
380 front side measurement showed stronger signal decay. Therefore, whether or not the coating is weakened
381 in the corners remains unclear from this ultrasonic analysis. The second 500×250 mm² plate (not depicted
382 here) showed a completely homogeneous coating, without irregularities on edges or corners. Potentially,
383 the coating adhesion of the 500×250 mm² plates may therefore be stronger than for the 300×200 mm²
384 plates. A mechanical investigation is planned.

385 **4. Conclusions**

386 This work reports first results on a transfer of W/EUROFER FGM coating technology to industry with regard
387 to upscaling for future fusion first wall application. Samples of three different sizes, the largest with an area
388 of 500×250 mm², have successfully been coated using low pressure plasma spraying. The specified coating
389 thickness of 2 mm was met, including 0.8 mm of tungsten top layer and 1.2 mm of functionally graded
390 material. The distortion of the larger samples was quantified, with warping remaining within the plate's
391 manufacturing tolerances. Chemical gradation was verified by EDX as well as image thresholding analysis,
392 the latter also indicating low porosity. The coating's microstructure consisted of a dense packing of splat-
393 shaped particles of W and EUROFER, as is typical for plasma spraying, with minor amounts of unmelted
394 particles and pores. Neither SEM nor ultrasonic analysis showed delamination at the coating substrate
395 interface. However, the ultrasonic analysis revealed potential weaker spots at the corners of the large
396 samples which will be subject to a future mechanical analysis, along with further microstructural analysis of
397 the larger plates. Taken together, the transfer of W/EUROFER FGM technology to industry was successful
398 with tests so far indicating an overall good coating quality. Further investigations envisioned include thermal
399 fatigue tests, experiments under fusion-relevant heat loads including plasma exposure, and the
400 characterisation of thermo-mechanical properties of single interlayers within the functionally graded
401 material.

402 **Conflicts of interest**

403 There are no conflicts of interest to declare.

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409

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