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Functionally graded tungsten/EUROFER coating for DEMO first wall: From laboratory to industrial production

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A B S T R A C T

The First Wall is a crucial component for the realisation of DEMO. It has to protect the tritium breeding blanket from erosion by high-energy particles while letting neutrons pass to enable breeding of tritium fuel. Furthermore, the First Wall needs to pass incoming heat in the MW/m^2 range to a cooling system for conversion to electric power. These requirements sum up to one of the harshest environments imaginable for a man-made material. Structural steel components alone cannot withstand these conditions. Tungsten is a viable armour material for the First Wall because of its low sputtering yield, high melting point, low activation and good thermal conductivity. It is not suitable though as bulk structural material because of its brittleness. Instead, the DEMO design foresees a First Wall of reduced-activation EUROFER steel, covered with a protective layer of tungsten. Direct tungsten-steel joints suffer from failure during processing or operation because of the thermal expansion mismatch between the two materials. This is solved by application of a functionally graded material as intermediate layer between steel and tungsten. Such coatings made of both tungsten and EUROFER, with a compositional gradient, have been produced with vacuum plasma spray technology. This technology enables manufacturing of the required millimetre-thick coatings and is suitable for upscaling. The development was supported by thermo-mechanical finite element simulations of load scenarios during processing and in-vessel service. Driven by promising results of high heat flux tests on larger, coated mock-ups the technology was transferred to industry for upscaling. Plates with a record size of 500×250 mm^2 and cooling channels were successfully coated. This contribution presents an overview of the development process, covers the latest results of ongoing research on the coating of curved First Wall structures and addresses future requirements.

1. Introduction

The European Demonstration Power Plant (DEMO) is a future fusion energy facility that requires self-sufficiency in terms of tritium fuel [1,2]. Following the helium-cooled pebble bed design (HCPB) as a possible solution for tritium generation [3], the reactor's plasma will be surrounded by tritium breeding modules which are covered by the First Wall (FW). The First Wall consists of plates of reduced-activation ferritic martensitic steel, e.g. EUROFER [4], and contains cooling channels connected to a helium cycle for heat transfer and energy harvesting [5]. During reactor operation the First Wall will be exposed to cyclic thermal load in the MW/m^2 range as well as bombardment by high-energy particles from the fusion plasma. The unprotected steel would suffer mechanical softening as well as sputtering erosion under such conditions [6,7]. Therefore, a protective 2 mm thick coating of tungsten is foreseen for the First Wall [7,8]. However, the lasting application of such coatings is impeded by the high thermal expansion mismatch between tungsten and steel [6,9].

This issue can be solved by creating a smooth transition between the material properties by applying a functionally graded material (FGM) between steel and tungsten. Over the past years such tungsten coatings with FGM interlayers have been developed in a collaboration between Karlsruhe Institute of Technology (KIT, Karlsruhe, Germany) and Forschungszentrum Jülich (FZJ, Jülich, Germany) by applying a multilayer system onto steel substrates [6,10,11]. Each interlayer of this multilayer system consists of mixed EUROFER steel and tungsten. The tungsten

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content is gradually increased from the steel substrate towards a 100% tungsten top coating. Out of various potential FGM production techniques vacuum plasma spraying (VPS) has been identified as suitable for coating of the First Wall [10–16]. VPS readily achieves the desired material gradient at sufficient microstructural quality, is suitable for the high coating thickness (2 mm) specified in DEMO, avoids excessive oxidation of the coating and in general thermal spraying bears potential for process upscaling to large coated areas [11,12,14,16]. This contribution aims to provide an overview over past and present development steps of these functionally graded W/EUROFER protective coatings for the First Wall and addresses future challenges.

2. Review of previous development steps

In order to assess feasibility and to find good parameters for both processing and FGM design, early test FGM coatings were created on small EUROFER blocks (100 × 100 × 18 mm^3) [11,14,17], or on similarly sized blocks of the tungsten alloy WL10, the latter building on similar developments for divertor joints [12]. amongst the parameters to be optimised were the size distribution of the feed stock powders, current of spray gun, stand-off distance, chamber pressure, velocity of the spray gun, but also the number and composition of the interlayers and the thickness of the FGM [11,12,14,17,18]. The quality of these coatings was assessed in view of microstructure, achieved gradient in chemical composition as well as mechanical hardness [12,14], by quantifying the residual stresses in the coating [11], and the interface toughness between coating and substrate [18,19], the thermal fatigue behaviour and thermal diffusivity of the coating [16,20] and ultimately by investigating their resistance against transient heat loads that simulate edge-localised modes [17,21].

With such a multitude of parameters to be optimised both in processing and coating design, a purely experimental approach would be excessively expensive and time-consuming. Therefore, the development of these FGMs was accompanied by finite element simulations in order to predict optimum parameters, e.g. for the number of interlayers, the type of chemical gradient (linear/quadratic over thickness) and the thickness of the FGM [6,22]. Optimised parameters were experimentally investigated for their suitability [12,14,19]. As an example, the finding of an optimum thickness will be briefly discussed here. A good tungsten coating requires a certain minimum thickness of FGM interlayer to achieve long-lasting performance under reactor conditions, because the above-mentioned mismatch in thermal expansion coefficient between tungsten and steel needs to be mitigated. On the other hand, simulations on the tritium self-sufficiency of future fusion reactors, covering either neutronics [23] or tritium retention in wall components [1] both call for a tungsten coating to be as thin as possible in order to achieve a higher tritium breeding ratio and to trap less tritium in the wall. Indeed the predictions on trapped tritium have to be seen as a major challenge to the realisation of fusion energy, with predicted tritium retention losses being as high as half a kilogram [1]. However, these simulations usually assume a 2mm thick coating of dense, 100% tungsten. The tritium retention study reported in [1] compared this with a functionally graded coating as presented here, finding comparably high retention, but assuming pure EUROFER properties for the FGM in absence of tritium permeation data. The FGM coatings investigated here contain only a fraction of steel and a minor level of porosity [14,24] which may substantially influence the tritium retention characteristics. A future experimental characterisation of the hydrogen retention of these coatings is therefore of major interest.

Regarding the optimum thickness of the FGM, a number of different FGM thicknesses was modelled for a finite element simulation covering several hundred operational cycles of a fusion reactor [6]. With this simulation the accumulated creep strain within the coating was evaluated (Fig. 1a), finding that a minimum FGM thickness of 1.2 mm is required for long-term coating performance [6]. This thickness has been used as design rationale for all following coating experiments, in order to achieve coatings as shown in Fig. 1b, with a 1.2 mm thick linearly graded FGM and a 0.8 mm thick W top layer realising the 2 mm coating thickness specified for DEMO [7].

The promising performance results obtained during this early development phase sparked a drive towards upscaling of this coating technology using the optimised parameters. Fig. 2 shows an overview of upscaled components coated up to now and includes an outlook towards current and future experiments to transfer the coating technology from flat surfaces to fusion-relevant curvatures.

As a first upscaling step, several mock-ups of EUROFER steel were fabricated at KIT and coated at FZJ on an area sizing up to 270 × 115 mm^2 (specimen on the left in Fig. 2) [16]. One of these mock-ups (Fig. 3a) was subjected to a high heat flux test (1000 cycles of 0.7 MW/m^2) [16,25] in the Helium Loop Karlsruhe (HELOKA) [26]. During the high heat flux test, surface temperatures of up to 800 °C were measured (Fig. 3b), but both coating and steel substrate remained undamaged [16].

After this promising outcome, further upscaling of the coated area was pursued which required a larger coating facility. For this, the knowledge of the FGM coating technology was transferred to the industrial company COATEC GmbH (Schlüchtern, Germany). As a result of this technology transfer, plates of P92 steel which contain cooling channels were coated. P92 has similar non-irradiated thermo-mechanical properties as EUROFER steel but is more readily available [24]. Two of these plates with uniform coating are depicted in the middle of Fig. 2, sizing 300 × 200 mm^2 and 500 × 250 mm^2, respectively. This

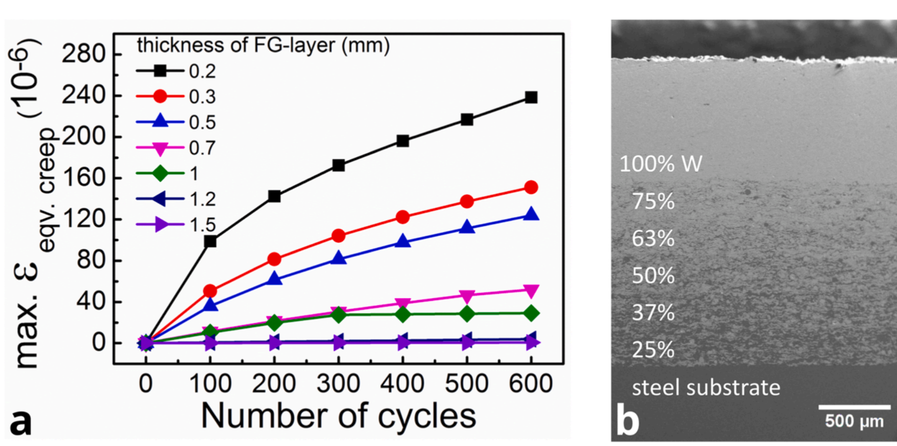

Fig. 1. (a) Maximum equivalent creep strain in the steel substrate for varying coating thickness, over several hundred simulated operational cycles. Image reprinted from ref. [6] with permission from Elsevier. (b) Cross section of functionally graded coating with interlayer tungsten content indicated.

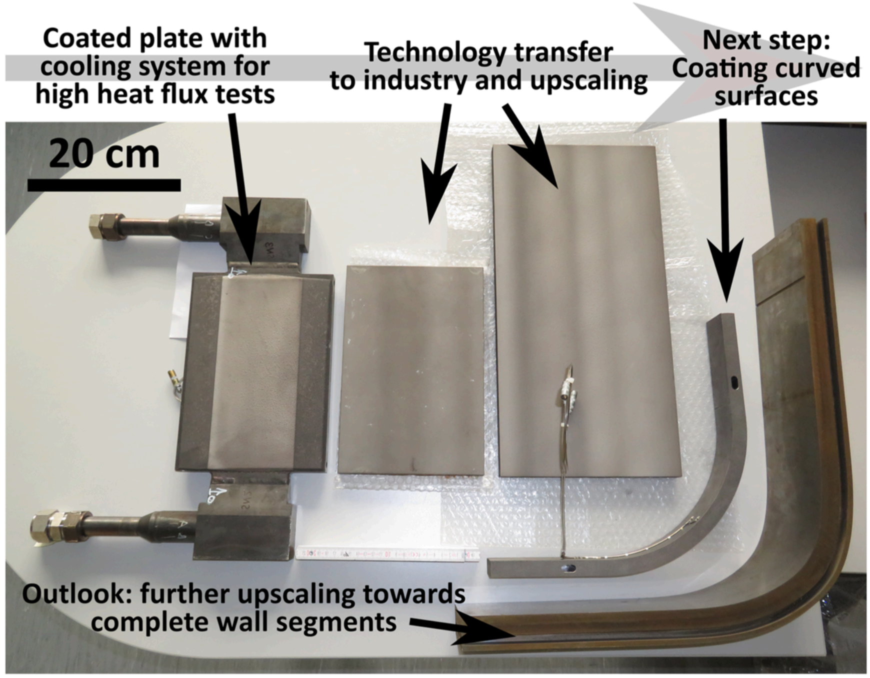

Fig. 2. Overview of different development steps during upscaling of coating technology.

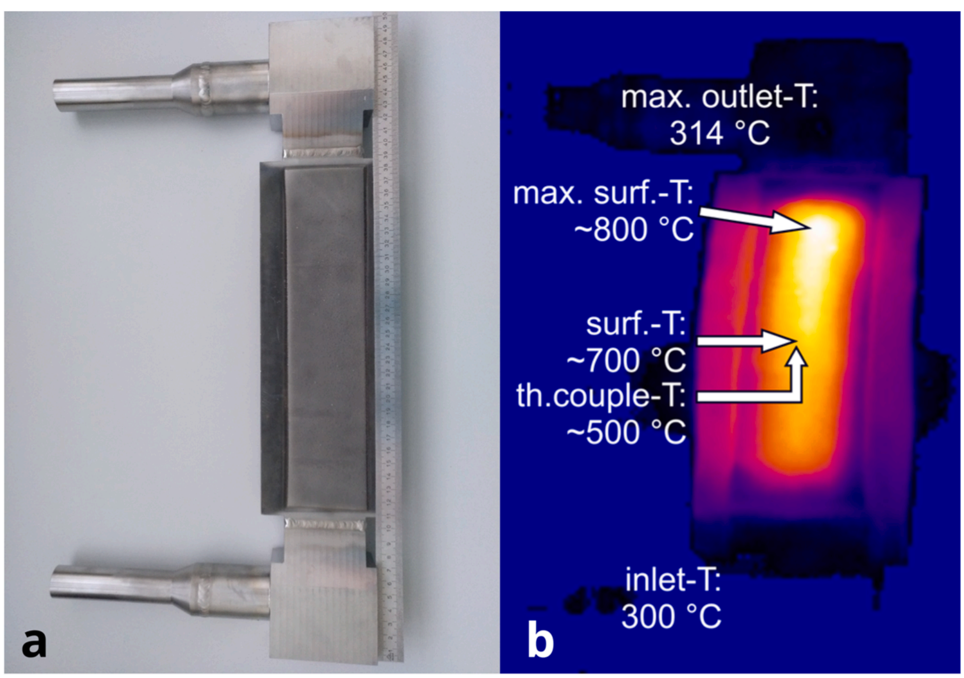

Fig. 3. (a) Mock-up with coated area 270 × 65 mm^2 for use in high heat flux test. (b) Infrared thermograph showing temperature of the mock-up during high heat flux test. Image reprinted and adapted from ref. [16] with permission from the IAEA and Euratom.

technology transfer was successful, with good achievement of specified coating thickness, gradation and microstructure [24].

At this point, four potential fields for future research on these coatings may be identified:

(i) Further process development and upscaling of coated components. As of now, the above-mentioned steps pose the state of the art. Progress in this field, both in size and geometry of the coated components, is required in order to achieve technology readiness for the equipment of a full-size breeding blanket with the required tungsten protection.

(ii) Detailed characterisation of thermo-mechanical properties of the coating, taking mixing ratio and the heterogeneous microstructure into account. The coating's material properties are difficult to obtain by standard testing techniques, yet are required to enhance the predictive power of simulations of large scale coated components.

(iii) Hydrogen retention of the coating and its performance under plasma exposure. The issue of tritium fuel retention in a heterogeneous, porous microstructure was already discussed above. Furthermore, the alteration of the coating's surface should be investigated here.

(iv) Coating performance after neutron irradiation will remain an important point for future study, even though irradiation-induced defects caused by displacements of lattice atoms (dpa) in steel are expected to recombine and be annealed out at the relatively high operating temperature of the coating [27]. Tungsten, however, will not undergo such annealing at the given temperatures. In addition, high-energy fusion neutrons would yield in both steel and tungsten transmutations where the transmuted products, e.g. helium in steel and rhenium in tungsten, may form clusters, bubbles [28] and even intermetallic precipitates [29] which would affect the thermo-physical and thermo-mechanical properties of the coating. Hence, investigating the irradiation effects on the FGM coating considering irradiation experiments amongst others in a fusion-orientated neutron source like IFMIF-DONES is an important step towards its qualification.

This contribution sets a focus on the first of these four research fields. In the following sections, the coating quality of the above-mentioned industry-coated plates is assessed further and a transfer towards coating of curved surfaces is presented.

3. Materials and methods

3.1. Further quality analysis of industry-coated plates

The industry-coated plates sizing 300 × 200 mm^2 and 500 × 250 mm^2 were fabricated from P92 steel (1.4901) and coated by vacuum/low pressure plasma spraying by the company COATEC GmbH as detailed in ref. [24].

Non-destructive ultrasonic testing was conducted on the entire coated area of the plates to identify potential weak spots. For this, a KC 200 immersion testing facility was used, which included a USIP40 testing device and KScan evaluation software (GE Inspection Technologies, Hürth, Germany). Details on the setup may be found in [30].

From selected positions in the middle and corner of a 300 × 200 mm^2 plate (marked I and II in Fig. 4a), four-point bending specimens of dimensions 45 × 4 × 3 mm^3 were cut by electric discharge machining, in order to assess the quality of coating adhesion. The specimens consisted of steel substrate in their lower half and coating in the upper half of the 4 mm dimension. In the middle of the 45 mm dimension, 1.5 mm deep and approx. 0.1 mm wide notches were cut into the coating side by electric discharge machining to provoke crack starting in the middle of the samples. Further notch sharpening and crack growth towards the coating-substrate interface was achieved with a resonating fatigue machine (RUMUL CRACKTRONIC, Russenberger Prüfmaschinen AG, Neuhausen am Rheinfall, Switzerland), applying an R-ratio of 0.1, a dynamic load of 0.75 Nm and a change of the resonance frequency by −1.5 Hz as stop criterion indicating sufficient crack growth at the notch tip.

Delamination of the coating was provoked by four-point bending the above-mentioned specimens in a modified universal testing machine (Model 8062, INSTRON, Darmstadt, Germany) which additionally included a vacuum high temperature furnace (MAYTEC Mess- und Regeltechnik GmbH, Singen, Germany). The four-point bending was performed at fusion-relevant operation temperature of 550 °C in vacuum (10^{-6} to 10^{-3} mbar) using a displacement speed of 0.2 μm/s. The delaminated coating-substrate interface was cut free by diamond wire cutting and examined by scanning electron microscopy (SEM, EVO MA10, Zeiss, Oberkochen, Germany).

3.2. Finite element analysis of curved substrate coating

Transferring the developed coating technology from flat to curved substrates will lead to new residual stress states in the curved coating. Finite element models of coated L-shaped specimens (Fig. 5a) were

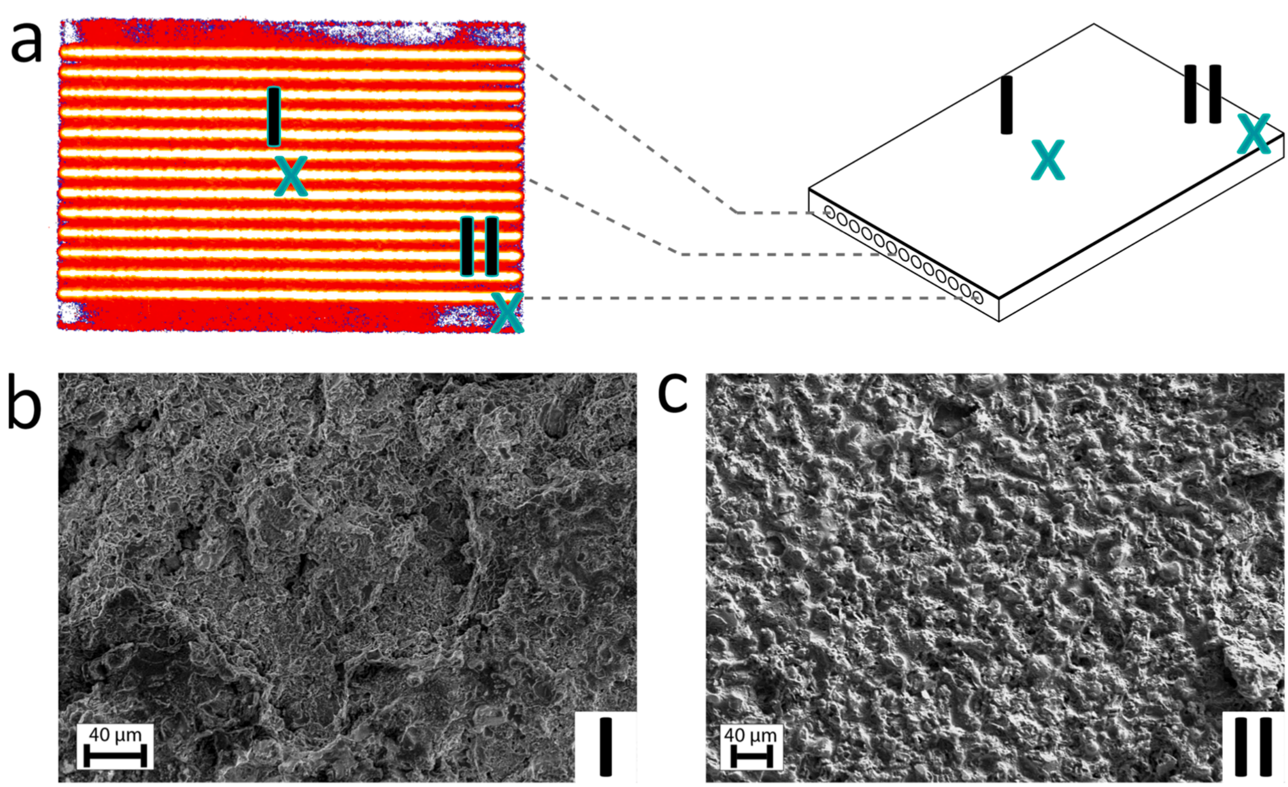

Fig. 4. (a) Ultrasonic analysis of coated 300 × 200 mm^2 plate and schematic illustration of plate. (b,c) Fracture surfaces at coating-substrate interface after provoked delamination, samples from regions marked I, II in (a).

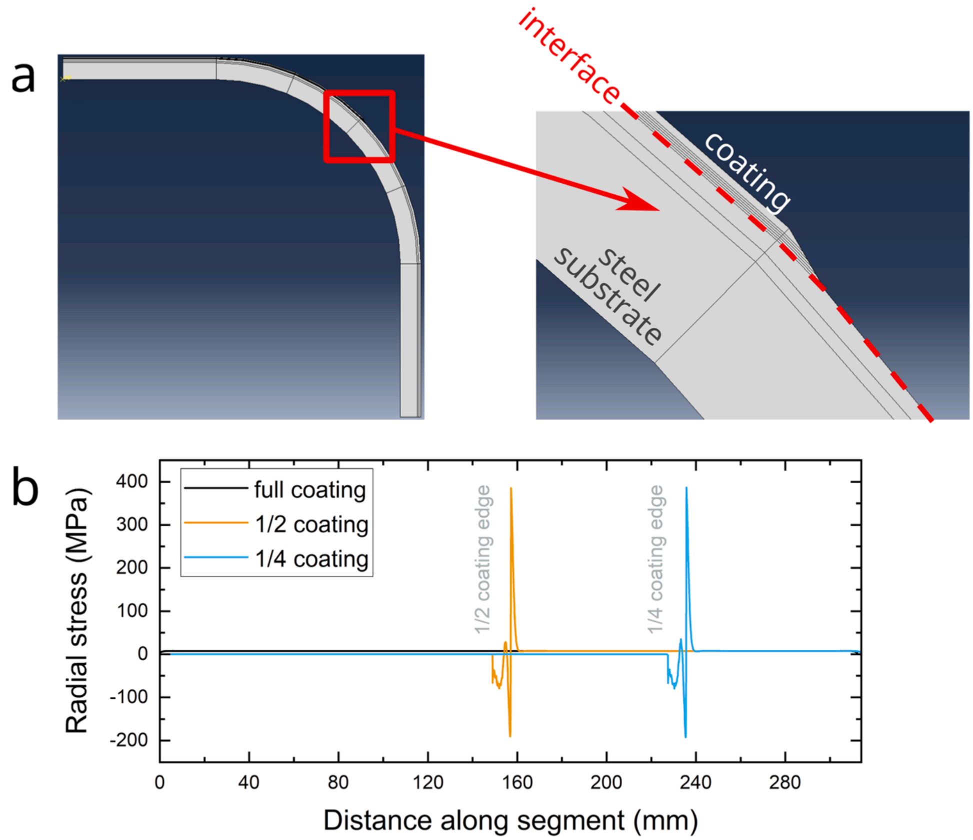

Fig. 5. (a) Finite element model of a curved substrate with outer radius of 200 mm and coating over the top half of the outer surface ("½ coating" in text). Magnification shows edge of coating. (b) Simulated radial stress at coating-substrate interface along curved segment with full / ½ / ¼ coating. For the ½ and ¼ coatings, the uncoated region is on the left (zero stress) and the coating begins towards the right, at the stress peaks.

prepared to characterise the radial stress level to be expected at the coating-substrate interface when coating a fusion-relevant curved surface. Three different models were compared, one with a coating over the entire outer surface, one where only half of the curved segment is coated (the one shown in Fig. 5a) and one where only a quarter of the curved segment is coated. The comparison was done to assess whether a partial coating is feasible since part of the curved segments in FW panels will be shadowed by neighbouring panels so that a coating there is not necessarily required. The simulations were run with ABAQUS, using a mesh of 4-node, bilinear generalised plane strain elements (ABAQUS: CPEG-4).

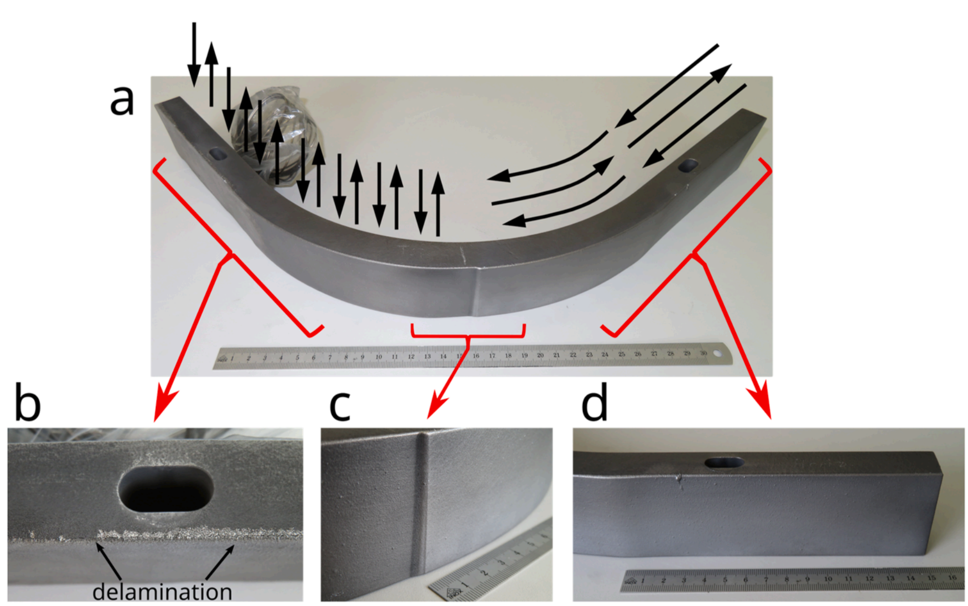

Fig. 6. (a) Experimental coating on substrate with transition from straight to curved geometry, comparing vertical coating deposition (left, b) with horizontal deposition (right, d). Delamination was found near bearing for vertical coating direction (b). No delamination was found near coating tips in middle (c) or for horizontal coating direction (d).

The use of generalised plane strain conditions is justified because in current First Wall designs the out-of-plane dimension is very long (several metres) without significant change of the in-plane geometry. The smallest mesh dimension (40 μm) was applied around the coating-substrate interface. Material properties for the FGM interlayers were linearly interpolated between those of pure tungsten and EUROFER [6]. Elastic-ideal plastic material behaviour was simulated. Fixed and loose bearing boundary conditions were applied to the two straight ends, respectively. The cooldown process of a coated part after the coating process was simulated by allowing the models to cool from the stress free state at 750 °C to 20 °C, with homogeneous temperature over the parts. The resulting radial residual stress component was evaluated at the coating-substrate interface in the curved region to predict if critical stresses would occur that endanger the successful bonding.

3.3. Coating of substrates with curved surface

To test the feasibility of transferring the coating technology to convex curved substrates an L-shaped specimen (Fig. 6a) was prepared from P92 steel. The outer surface to be coated received a machined finish and comprises two straight, flat sections as well as a central curved section with outer radius of 200 mm to match radii currently achievable in manufacturing of FW components [5,31]. The coating of this outer surface was conducted at FZJ, similarly to previously published coating experiments and using powder of similar grain size distribution [11,16]. However, the velocity of the spray process was reduced to 100 mm/s on the flat segments to achieve comparable movement speeds in flat and curved regions, since the angular velocity of the setup was limited. Two different coating procedures were tested, each on one half of the specimen. For the first procedure, the spray process followed a vertical movement pattern as indicated by the arrows in Fig. 6a, for the second procedure it followed a horizontal pattern. For this pattern, a short dwell time occurred while switching from rotational to linear movement. This led to a slightly larger coating thickness at the transition area between straight and curved section. All other spray parameters were kept constant. The substrates were preheated using the spray gun without powder feed. For this first feasibility test, only a mixture of 50% W / 50% EUROFER was applied. The full FGM coating will be subject to a future investigation. The quality of the coating was investigated by visual inspection and measurement of the achieved coating thickness.

4. Results and discussion

4.1. Industry-coated plates: importance of temperature management

The result of the ultrasonic analysis of a 300 × 200 mm^2 plate is shown in Fig. 4a, along with a schematic of the plate for ease of understanding. The ultrasonic result depicts a C-scan, where red colour marks the uncoated bottom side of the plate and the yellow/white horizontal stripes mark the cooling channels. Blue/white colour in the corners of the plate marks potential weak spots within the coating, even though no cracking was observed here and microstructure appeared similar to that in other regions. To scrutinise these areas in more detail four-point bending specimens were cut out of a corner and out of the middle of the plate for reference (positions marked I and II in Fig. 4). The specimens were bent at 550 °C in vacuum, to provoke delamination under fusion-relevant conditions.

The delaminated surfaces at the coating-substrate interface were investigated with scanning electron microscopy (Fig. 4b,c). For the middle of the plate the delaminated surface showed a ridge pattern typical for ductile fracture (Fig. 4b), indicating good bonding between coating and substrate. For the corner of the plate the fracture pattern indicated a brittle delamination (Fig. 4c) and thus, potentially, insufficient bonding. Such behaviour may be favoured by residual stress peaks in the coating close to the corners. The combined results from ultrasonic testing and provoked delamination prove that the corners of this investigated plate pose weak spots. The reason for the weak bonding between coating and substrate at the corners may be found in the temperature distribution during the coating process. Here, the spray gun is the only source of heat and moves back and forth over the plate, allowing some regions to cool down. In the absence of countermeasures, the corners will then cool down faster than the rest of the plate. This was verified with thermocouples as detailed in ref. [24]. Taken together, these results highlight the importance of temperature management during the coating of larger components. With a sufficiently warm substrate good bonding of the coating was achieved, but the temperature gradient over the coated area needs to be minimised for a uniform coating quality. We note that the process could be optimised for coating 500 × 250 mm^2 plates. There, no more weak spots were detected during ultrasonic testing [24].

4.2. Coating of curved surfaces

All of the above-mentioned developments were performed on flat surfaces. Yet the design of First Wall elements involves sections with a curved outer surface to be coated with tungsten [3]. The applicability of the W/EUROFER FGM coating technology onto curved substrates thus needs to be proven. The curvature can introduce out of plane stress components which can lead to delamination. Before testing the feasibility of coating curved substrates in laboratory, a finite element analysis was therefore conducted in order to assess the stress levels to be expected as well as potential critical spots. For the substrate, a simple L-shaped geometry (Fig. 5a) was modelled that involves a fusion-relevant outer radius of 200 mm as well as two straight ends to model the transition from straight to curved sections. Three models were compared, one being coated on the entire outer surface and two partial coatings that only cover one straight section and half or a quarter of the curved section (named ½ and ¼ coating). Even though a partial coating moves the coating edge with its stress concentration into the critical curved section where additional out of plane stress components occur, the ½ and ¼ coatings were investigated to see whether the percentage of coated curvature influences the stress underneath the coating. In a fusion reactor such as DEMO, parts of the curved segments and sidearms of First Wall panels will be shadowed by neighbouring panels and thus do not necessarily require a protective coating [32]. If partial coating of the curved segment is successful, the shadowed regions may be omitted during coating. Besides material savings, this would save substantial amounts of processing time considering the large amount of required First Wall panels, and may thus help to reduce the cost of a reactor.

For this study the residual stress buildup after coating application was investigated by allowing the models to cool down from stress free state at 750 °C to 20 °C. The radial residual stresses at the coating-substrate interface were investigated (Fig. 5b) since they are most critical for the stability of the coating. Since ABAQUS provides poor interpolation of values at such interfaces, the analysed path was minimally shifted by one element's length (40 μm) into the steel substrate. The stress levels in Fig. 5b are, however, representative, as was verified by continuous stress profiles perpendicular to this path (not shown here). Following Fig. 5b from left to right along the length of the curved segment, the radial stresses are constant (approx. 8 MPa) for the fully coated model (black line). For the ½ (orange) and ¼ coatings (blue) they start with zero stress on the uncoated surface, then passing a stress peak of about 400 MPa at the coating edge and then levelling down to a constant low value of about 8 MPa underneath the coating. This constant radial interface stress underneath the coating is identical for all three models, and its low value can be seen as uncritical for the performance of the coating. A stress discontinuity at the transition from curved to straight coating was minimal, i.e. a step from above-mentioned 8 MPa to zero radial stress. These results indicate that it does not matter how much of a curved section will be coated, the only critical point will be the stress concentration at the coating edge. This concentration will, however, also occur at the edge of a fully coated component and thus cannot be avoided. Whether such an edge actually leads to failure needs to be verified experimentally.

To test the feasibility of coating curved surfaces, L-shaped specimens were prepared to match the simulated geometry (Fig. 6a). At the time of writing this contribution, only the first experiment was finished. The results presented in the following section thus need to be treated as preliminary, yet already allow for valuable insight. A single L-shaped specimen was coated at FZJ following two different procedures: One side was coated using a vertical up-down meander movement between specimen and spray gun (left side of Fig. 6a, indicated by arrows). This movement was chosen to avoid a jump in coating thickness at the transition between straight and curved sections, when a translational movement of the sample holder needed to be switched to rotational movement. The other side was coated using a horizontal meander movement (right side of Fig. 6a). For this first test, only a mixture of 50% W – 50% EUROFER was applied instead of the FGM. Coating thicknesses > 1 mm were achieved with both horizontal and vertical meander movements.

While for the vertical movement cracking and delamination of the coating was observed (Fig. 6b), no defects were found in the middle of the sample where the two coatings ended in steps (Fig. 6c). In Fig. 6c only the edge of the right coating is clearly visible since the edge of the left coating had a lower angle towards the substrate. The absence of cracking here indicates that a partial coating with edge may be created successfully despite the expected stress concentration at the edge (Fig. 5b). The delamination observed on the experiment with vertical meander movement was most pronounced in the vicinity of the bearing hole (Fig. 6b) where the sample was connected to a metallic sample holder. At this position the sample may quickly lose heat during the coating experiment. It is thus assumed that this region became too cold for successful bonding of the coating, similar to the above case of the corners of the flat 300×200 mm^2 plate (Fig. 4). For the horizontal meander movement, on the other hand, no cracking or delamination was found. Here a sound coating was achieved (Fig. 6d), its only discontinuity being a higher thickness at the transition between straight and curved section (left end of Fig. 6d) that could not be avoided here because of a switching delay between translational and rotational movement. The absence of delamination here indicates that the horizontal movement approach lead to a more homogeneous temperature distribution over the coated area, thus avoiding formation of weak spots. This successfully applied coating with a transfer from straight to curved substrate is a promising outcome for the first feasibility test. The remaining experiments to be conducted will aim for applying the entire coating with FGM and to further improve temperature management, so that a full coating can be applied onto curved segments in the future.

5. Conclusions

Joining tungsten and steel is a challenge in materials technology. The specifications for the future fusion reactor DEMO call for even more: a stable, thick coating of tungsten on steel that needs to be applied over areas in the square metres range and that needs to withstand substantial heat loads. Nevertheless, a suitable coating technology was developed over the past years. This contribution summarises the major development steps, ranging from early laboratory coating experiments via mock-ups for high heat flux tests towards a technology transfer to industry in order to achieve upscaling of the coated area. The design process was enhanced by combining findings from finite element simulation, processing and characterisation to allow for quick identification of optimised parameters. Temperature management was identified as an additional challenge when upscaling the components to be coated. Moreover, future First Wall panels will involve curved surfaces that need to be coated. The feasibility of coatings on fusion-relevant curvatures was demonstrated here, both by simulating the expected stress levels and by running a first feasibility test in laboratory. Future coating experiments for the First Wall of DEMO may build upon the experience collected here in order to fully master the coating of curved surfaces and to drive the coated area towards the size needed in fusion.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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