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Development of coatings for fusion power applications

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Abstract

Coatings have been proposed as the solution to critical materials constraints for most of the blanket concepts under development for fusion power applications. However, the international programs on coating development are focused primarily on electrically insulating coatings to mitigate the magneto-hydrodynamic pressure drop in self-cooled lithium/ vanadium blanket concepts, and on tritium permeation barriers to reduce tritium permeation from Pb–Li into the water coolant in water-cooled Pb–Li concepts. Emphasis of the insulator coating development is on CaO and AlN coatings formed on vanadium alloys either in situ in lithium or by vapor deposition processes. The tritium barrier coating development is focused on Al₂O₃ formed on aluminized martensitic steels by several processes. This paper presents an overview of the fundamental materials issues associated with the various coatings and the status of coating development for the various applications.

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1. Introduction

Coatings are proposed as the solution to critical materials constraints in most of the leading first-wall/ blanket concepts considered for magnetic fusion power systems [1–15]. Coatings are considered essential for some first-wall/blanket concepts and they are needed to enhance the performance limits for other concepts. Proposed coating applications include:

- electrically insulating coatings to mitigate magneto hydrodynamic (MHD) effects in self-cooled liquid metal systems,
- tritium barriers within the blanket system to reduce tritium permeation into water coolants,
- tritium containment to reduce tritium release to the environments,
- corrosion barriers to permit higher temperature operation,

• helium containment to reduce helium leakage into the plasma chamber.

The international programs on development of coatings are focused in two specific areas. These areas involve electrically insulating coatings to mitigate the MHD pressure drop in self-cooled lithium systems with a vanadium-alloy structure, and tritium permeation barriers (TPB) in water-cooled Pb-Li (WCLL) systems to reduce the cost of the tritium recovery from the water coolant and to enhance the safety. Other specific applications identified include: (1) to improve tritium containment in high-temperature helium-cooled systems, (2) to improve tritium containment and/or serve as a corrosion barrier for the flibe molten salt systems (depending on the flibe chemistry and the structural materials), (3) for helium containment in a helium-cooled SiC_f/SiC system to reduce leakage into the plasma chamber, and (4) for tritium containment in high-temperature ceramic breeder systems.

In Europe, the emphasis of the coating development program is on an alumina/Al–Fe coating on reduced activation martensitic (RAM) steels as a tritium barrier in the WCLL concept. The research programs have

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included effort at FZK in Germany, CEA in France and both ENEA and ISPRA in Italy. In Japan, the emphasis of the program is on electrically insulating coatings for the self-cooled lithium/vanadium system; however, some effort is also focused on coatings for the flibe breeder systems. The research is conducted primarily at the National Institute for Fusion Science (NIFS), the University of Tokyo and Tohoku University. In Russia and the United States the research effort is focused on the development of electrically insulating coatings for the lithium/vanadium system. Coating research programs involve Red Star, Efremov and the Institute of Physics and Power Engineering in Russia, and Argonne and Oak Ridge Laboratories in the US. Coatingrelated activities for the other applications have been limited to analyses conducted as part of system design studies.

2. Considerations and requirements for coatings

Electrically insulating channel walls are required to reduce the pressure drop to acceptable levels in a selfcooled lithium system. Thin insulating coatings on the vanadium alloy coolant channels are the proposed approach [1-7]. Based on analyses of conceptual designs for a Li-V system, the product of the electrical resistivity of the coating and the coating thickness must exceed a value of approximately 100 Ω cm² [16,17]. This value corresponds to an electrical resistivity of $10^6 \Omega$ cm for a 1 µm coating. To assure complete coverage, a coating thickness of 5-10 µm is considered desirable. Therefore, an electrical resistivity of $10^5-10^6 \Omega$ cm would be adequate. In addition, the coating for the Li-V system must be chemically compatible with both the lithium and the vanadium alloy substrate at temperatures of interest, viz., 400-750 °C. As discussed later, this will require control of the lithium chemistry.

Design studies for the WCLL blanket concept with a martensitic steel structure indicate that a permeation barrier is required to reduce the permeation of tritium from the Pb–Li breeder material into the water coolant [8–11]. It is both difficult and expensive to recover tritium from water at levels considered necessary for safety reasons. The design studies indicated that a tritium permeation reduction factor (TPRF) of >100–1000 compared to the permeation through a bare steel wall is required for the typical wall temperature of about 300 °C. A coating on the martensitic steel channel wall is the proposed solution to reduce the tritium permeation to acceptable levels. In addition, the coatings must be compatible with the Pb–Li breeder material.

Tritium and helium permeation barriers are also considered for other blanket concepts; however, the requirements have not been quantitatively defined since they depend on the specific design.

Table 1

Considerations/requirements for coatings

Electrical insulator for LilV system

- Resistivity X thickness > $100 \ \Omega \ cm^2$
- Compatibility with Li

Tritium barrier for WCLL system

- TPRF > 100 compared to bare RAM steel wall
- Compatibility with Pb-Li

General requirements/considerations for all systems

- Potential for coating large complex configurations
- Potential for in situ self-healing on defects that might occur
- Processing parameters compatible with materials and capabilities, e.g., temperatures and times
- Thermal expansion match/bonding with substrate
- Acceptable neutronic properties
- Materials availability/cost
- Safety/environmental characteristics
- Radiation damage resistance

In addition to the specific considerations/requirements discussed above, several other general considerations/requirements relevant for all coating applications are listed in Table 1. In all cases, the coating procedures must be applicable to coating large complex configurations. In most cases, the coating must be applied after assembly of the component so as not to affect welding/ joining and to assure effective coverage. Because of the severe consequences of a coating failure, it is widely accepted that self-healing of any defect that might occur during operation is a requirement. Process parameters, e.g., temperatures and times, must be compatible with the substrate materials and engineering constraints. A thermal expansion match between the coating and the substrate is important to enhance the bonding. Other considerations include acceptable neutronic properties, materials availability/cost, safety/environmental characteristics, and radiation damage resistance.

3. Electrically insulating coatings for the V–Li system

3.1. Candidate coatings

Only a limited number of materials offer a potential for meeting the most basic requirements for the MHD coatings, viz., electrical resistivity and chemical compatibility with lithium. Most carbides exhibit low electrical resistivity, many nitrides also exhibit low electrical resistivity and only a limited number of oxides are stable in lithium. Early assessments of various materials for the insulator coating application identified CaO and AlN as leading candidates based on the criteria discussed above [1,3]. The focus of the coating development for this application has been on these two materials with CaO receiving the most attention. Other candidate materials that have been considered include: Y_2O_3 , BeO, MgO, Er_2O_3 , Sc_2O_3 , $CaZrO_3$, BN and Si_3N_4 .

The electrical resistivities of potential candidate insulator coating materials are plotted in Fig. 1 as a function of temperature. CaO exhibits very high resistivity while that of AlN is much lower but still appears to be adequate. Fig. 2 indicates the relative thermodynamic stabilities of several oxides with respect to Li/ Li₂O equilibria and the oxygen activity in liquid lithium with oxygen concentrations of 1, 10 and 100 wppm. CaO and Y_2O_3 should be stable in lithium with only a few wppm oxygen. Fig. 3 is a similar plot indicating the thermodynamic stability of several nitrides with respect to Li/Li₃N and lithium with nitrogen concentrations of 10, 100 and 1000 wppm. TiN is the most stable of these nitrides but it has a relatively low electrical resistivity.



Fig. 1. Electrical resistivity of ceramic materials as a function of temperature.



Fig. 2. Thermodynamic stability of candidate oxide coating materials with respect to Li/Li₂O and Li with low oxygen concentrations.



Fig. 3. Thermodynamic stability of candidate nitride coating materials with respect to Li/Li_3N and Li with specified nitrogen concentrations.

AlN is stable in lithium with low nitrogen concentrations at temperatures of interest. These results are supported by the calculations of Hubberstey [18,19].

The oxide (or nitride) coating must not only be stable in lithium with specified oxygen (or nitrogen) concentrations, but must be stable with respect to other compounds that could form by reactions with other impurities in the lithium. Fig. 4 shows an example of complex interactions involving both oxygen and nitrogen in lithium that affect the AlN/ LiAlO₂ equilibria. AlN will be stable in lithium only if the nitrogen concentration in lithium exceeds the specified oxygen concentrations indicated by the curves. In a cold-trapped (e.g., 200 °C) lithium system, fortunately a region of AlN stability exists above \approx 7 wppm oxygen and below the \approx 2000 wppm nitrogen. Note that the Al activity in Li can also affect these equilibria. Similar considerations



Fig. 4. Calculated regions of stability for AlN and LiAlO_2 in Li.

must be given to other compounds such as carbides, nitrides and hydrides that might affect the stability of the desired coating material.

3.2. Approaches to insulator coating development

The insulator coating development has focused on two general approaches, viz., in situ formation during exposure to lithium with controlled chemistry, and various vapor deposition processes.

3.2.1. In situ formation of insulating coatings

In situ formation of coatings on V-alloys by exposure to lithium with controlled chemistry is regarded as the most ideal approach for meeting the requirements. In situ formation of the coating would indicate compatibility with lithium, would provide for self-healing of defects that might occur during operation and would be applicable for coating large complex configurations. The in situ coating development has focused on CaO and AlN and has involved primarily US [20–25] and RF [26– 28] efforts with recent involvement by Japan.

For the case of in situ formation of CaO, Ca dissolved in lithium can react with O dissolved in the Valloy to form a CaO coating as indicated schematically in Fig. 5. The thermodynamics and kinetics of this reaction will be controlled by the oxygen concentration and mobility in the V-alloy and the Ca concentration in the lithium. Only about 2 at.% Ca in Li appears adequate to form CaO on the V-alloy substrate at temperatures of interest. Oxygen charging to about 1 mg/cm² of the V-alloy surface regions is adequate to form the de-

Li (Ca)

V-alloy

O,

sired CaO coating thickness of $\approx 10 \ \mu\text{m}$. It is important in the oxygen charging process to avoid formation of vanadium oxide, e.g., VO₂, which would react with Ca in Li to form a complex Ca–V–O compound, since these complex oxides typically exhibit low electrical resistivity.

Fig. 6 shows a typical hardness profile of a V-4Cr-4Ti alloy after an oxygen pre-charge of $\approx 0.92 \text{ mg O/cm}^2$ (solid symbols). The higher surface hardness indicates that the added oxygen remains near the surface (≈ 100 μm). Also shown in the figure is the hardness profile (open symbols) of a similar specimen ($\approx 1.23 \text{ mg O/cm}^2$) after exposure to Li-2.8at.%Ca at 700 °C for 50 h. The reduction in the surface hardness indicates that much of the pre-charged oxygen has diffused to the surface and reacted to form CaO. Fig. 7 shows an EDS profile indicating a CaO coating thickness of ${\approx}18~\mu m$ after exposure to Li-2.8at.%Ca at 700 °C for 50 h. Measurements performed on similar coatings indicate electrical resistivities of $\approx 10^8 \Omega$ cm at 500 °C, exceeding the required value by approximately three orders of magnitude. Fig. 8 shows a low magnification SEM of a uniform in situ formed CaO coating on V-5Cr-5Ti after exposure to Li-2.8at.%Ca at 600 °C for 120 h and a backscattered electron image micrograph of the same (\approx 15 µm) coating.

Similar procedures have been used to form AlN coatings in situ by exposure of V-alloy specimens to lithium with controlled concentrations of Al and N [28]. In one example, V-alloys are exposed to Li–(5–15)wt%A1–(0.5–5)wt%N at 600–800 °C. AlN coatings 2–4 µm thick were formed that exhibited an electrical resistivity of \approx 4000 Ωcm. Fig. 9 presents results of compatibility tests conducted at 700 °C for 100 h, which indicate very little weight change when exposed to Li–0.05wt%N–0.5wt%A1.





Fig. 6. Hardness profile of V–4Cr–4Ti alloy after oxygen precharge and after exposure to Li–2.8at.%Ca at 700 $^\circ$ C for 50 h.





Fig. 7. EDS profile of \approx 18 µm CaO coating formed on V–4Cr– 4Ti by in situ exposure to Li–2.8at.%Ca at 700 °C for 50 h.





Fig. 8. (a) Low magnification SEM and (b) backscattered electron image of $\approx 15 \ \mu m$ CaO coating on V–4Cr–4Ti formed in situ after exposure to Li–2.8at.%Ca at 600 °C for 120 h.



Fig. 9. Weight change of V–4Cr–4Ti alloy samples and alloy with AlN coating after exposure to flowing Li with various concentrations of N and Al at 700 $^{\circ}$ C for 100 h.

3.2.2. Insulator coatings formed by vapor deposition processes

Electrically insulating coatings on V-alloys have also been formed by several vapor deposition processes including thermal/chemical vapor deposition (T/CVD), chemical vapor deposition (CVD) and RF sputtering techniques. Development of T/CVD of CaO coatings and CVD of CaO and Y₂O₃ coatings has been conducted [29–34] as well as RF sputtered AlN coatings [33]. This research has involved investigations of process parameters in addition to electrical properties, microstructural characterization and Li compatibility of candidate coatings formed by these methods. Fig. 10 presents an EDS profile of \approx 35 µm T/CVD CaO coating on V–4Cr–4Ti. Coatings formed by this technique have exhibited good compatibility with Li–2.8 at.%Ca at temperatures to 700 °C. Fig. 11 shows the measured



Fig. 10. EDS profile of ${\approx}35~\mu m$ CaO produced by T/CVD process.



Fig. 11. Measured electrical resistance of T/CVD CaO coating after exposure to Li–2.8at.%Ca at 500 °C for 68 h.

electrical resistivity of a T/CVD CaO coating as the temperature in increased to 700 °C (and subsequent cooling) after exposure to Li–2.8at.%Ca at 500 °C for 68 h. The electrical resistivity exceeds the required value by over two orders of magnitude up to 700 °C.

3.2.3. Compatibility of bulk ceramics with Li

Scoping tests have been conducted to assess the chemical compatibility of potential candidate coating materials with liquid lithium. Park et al. [22] investigated a large number of candidate materials in the early phase of the insulator-coating program and reported that the compatibility was sensitive to the purity of the ceramics and the chemistry of the lithium. This is consistent with a vast database on liquid metal corrosion. More recent investigations conducted to assess the long-term high-temperature compatibility of bulk ceramics confirm the earlier conclusions [35]. These results indicate that oxygen impurity in AlN significantly affects the compati-



Fig. 12. Weight loss of bulk AlN after exposure to Li contained in V-alloy and Mo capsules.

bility with Li as predicted since Al₂O₃ is not stable in Li. Similar results on CaZrO₃ with excess ZrO₂ are obtained which indicate that the corrosion of CaZrO₃ increases with the ZrO₂ content as expected [36]. The importance of Li chemistry is also shown in Fig. 12 where the effects of the capsule material on the compatibility of AlN in Li is significant at temperatures above ≈ 600 °C. In this case, the nitrogen activity in the Li is affected by the container material.

4. Tritium permeation barrier coatings for WCLL systems

The second major effort on coatings development for fusion power applications is related to TPB for the WCLL system to reduce the tritium permeation into the water coolant [8-12]. Use of TPB coatings is the proposed approach for reducing the tritium permeation from the Pb-Li into the water coolant. This effort, which is conducted within the European Union, is focused exclusively on Al₂O₃ formed on aluminized, RAM steels [37-40]. The relatively high tritium partial pressure produced in a Pb-Li breeder blanket and the large surface area of the coolant tubes in the WCLL concept contribute to the issues associated with tritium permeation into the water coolant. Tritium buildup in the water coolant presents a safety issue and recovery of tritium from water at the low levels desired is a significant economic issue.

4.1. Requirements for the TPB coatings

Specific requirements for the tritium barrier coating application for the WCLL system include [41]:

- Produce a TPB on the RAM steel without adversely affecting the metallurgical state of the martensitic steel structure, e.g., temperature limits.
- Produce a coating with a TPRF of >100 under reactor conditions compared to the permeation rate for a bare RAM steel coolant-channel wall.
- The coating procedures must be compatible with the ITER test module geometry and fabrication sequence.
- The coating must provide for self-healing of the alumina scale for acceptable reliability.

In addition to these specific requirements, the general considerations/requirements discussed in Section 2 also apply.

4.2. Development approach for TPB coatings

Several fabrication approaches for the TPB coatings have been under development in various European laboratories [37,42–44]. These include:

- Hot-dip aluminizing (HDA) at FZK, Germany.
- CVD at CEA, France.
- Vacuum plasma spray (VPS) at JRC, Ispra, Italy.
- Detonation jet, low pressure plasma spray (LPPS) and Air Plasma Spray (APS) at ENEA, Italy.

Evaluations of the TPRFs for coatings produced by all of the above methods have been conducted at Ispra and at ENEA. In addition, corrosion testing of the most attractive coatings has been conducted in a flowing Pb–Li loop at FZK. After a first coating evaluation conducted in 1998, development efforts for the VPS process at Ispra and the detonation jet spray process and the plasma spray processes at ENEA were terminated. Only development efforts on the HDA process at FZK and the CVD process at CEA were continued after 1998.

The HDA process under development at FZK involves dipping of RAM steel specimens in liquid aluminum at 700 °C for 30 s followed by heat treatment under conditions typically used for the martensitic steels [45]. Initial heat treatments investigated involved temperatures of 950-1075 °C for periods of 0.5-2 h followed by air cooling. The purpose of this heat treatment is to transform the brittle intermetallic Fe₂Al₅ scale formed during hot-dipping to more ductile phases of FeA1 and α -Fe(A1). The current reference heat treatment is 1040 °C for 0.5 h with air cooling followed by a normalizing heat treatment at 750 °C for 1-2 h with air cooling. An EDS profile of the surface region of an F82H-mod steel with an HDA coating after heat treatment (1040 °C/0.5 h; 750 °C/1 h) is shown in Fig. 13. During the development it was shown that HIPing during the hightemperature heat treatment at a pressure of 250-1000 bar increased the thickness of FeAl, decreased the thickness of α -Fe(Al) and suppressed subsurface pore formation as shown in Fig. 13.



Fig. 13. EDS of hot-dip aluminized coating on F82H-mod after heat treatment (1040 °C/0.5 h; 750 °C/1 h) under 750 bar pressure.

The CVD process under development at CEA, France involves pack cementation on FeAl with an NH₄Cl activator and an Al₂O₃ inert filler to form an FeAl/Al₂O₃ TPB on the RAM steel substrate [42]. This technique has the advantage of a lower temperature process, typically 650–750 °C, which avoided any modification of the martensitic steel properties. The CVD approach also lends itself to a potential for in situ coating formation. Typical coating thickness of the aluminized layer is 3–7 µm with an Al₂O₃ layer \approx 1 µm thick. Possible improvements in the CVD process development include the use of CrAl instead of FeAl, and consideration of the use of CO₂ for the oxidation process.

The VPS process, which was under development at Ispra, involved a plasma spray coating of Al followed by a high-temperature heat treatment to transform the aluminum layer into an aluminide coating [41]. The initial investigation of the VPS process (Series 1), utilized the final tempering treatment of MANET-II (750 °C/2 h) to produce the aluminized phase. The aluminized coating produced under these conditions appeared to be uniform and dense in regions near the steel but slightly porous near the outer surface. Based on these results, an alternative heat treatment of the aluminizing procedure with higher temperatures similar to the austenitizing treatment for MANET-II was used; viz., 965 °C/2 h plus 1075 °C/0.5 h/fast cool plus 750 °C/2 h. This series 2 approach yielded lower Al compositions in both the outer layer (\approx 30 at.%Al) and the inner layer (25–4 at.%) corresponding to the more ductile FeAl and α -Fe(Al) phases.

Three processes, viz., detonation jet, APS and LPPS were investigated at ENEA, Italy for producing the tritium barrier coatings [43]. The detonation jet process involved deposition of pure Al on MANET followed by a 950 °C heat treatment for 16 h in a vacuum of 10^{-2} Pa. Two layers consisting primarily of α -Fe(Al) and FeAl with a total thickness of \approx 300 µm were formed by this techniques. The APS approach involved spray of FeAl metal-based powders, which resulted in coatings up to 150 µm thick with a lamellar structure. These coatings were highly porous and brittle and exhibited rather poor adhesion. The LPPS process also involved spray of an FeAl layer followed by a spray of a thin layer of Al₂O₃. These coatings also suffered from poor adhesion.

4.3. Characterization of tritium permeation barrier coatings

Two primary factors for evaluation of the TPB coatings are the TPFR and the compatibility of the coatings with Pb–Li alloy. The measurements of the TPRF for the various candidate coatings described above were conducted at Ispra [37]. The proposed criteria was a TPRF > 1000 for a gas-phase permeation test and a TPRF > 100 for tests in liquid Pb–Li compared to the reference permeation rates through bare RAM steel. Only the HDA and CVD coatings met the required TPRF > 1000 for measurements in hydrogen gas at a temperature of \approx 300 °C. Preliminary results obtained by bubbling hydrogen through Pb–Li indicated that the TPRF for the HDA and CVD coatings exposed to the Pb–Li were below the acceptable value of 100. Further evaluation of these results is in progress in an attempt to understand the reasons for the low TPRF's in Pb–Li.

The second criterion used for evaluation of the TPB coatings involved compatibility with Pb-Li [46]. The corrosion tests were conducted in the PICOLO loop at FZK in flowing Pb-Li at 480 °C for periods up to 10000 h. Tests were conducted on HDA, CVD and VPS coated specimens provided by the various laboratories. Results obtained for the HDA and VPS coatings exposed up to 10000 h in Pb-17Li at 480 °C indicated no observable corrosion attack. However, a significant interaction between the CVD coatings and the Pb-Li was observed. The thickness and Al concentration in the Fe-Al layer decreased significantly with time of exposure. The reason for the differences between the compatibility of the CVD coating and that of the other two coatings with Fe-Al layers is uncertain at this time. One possibility is the formation of the more stable α -Al₂O₃ formed at the higher temperatures of the HDA and VPS processes compared to the lower temperature CVD process for which the γ -Al₂O₃ is the meta-stable phase. Further investigations are required to confirm the basis for the differences in compatibility of the various coatings with Pb-Li.

5. Other coating applications

Coatings for the flibe system are also under consideration either for corrosion protection or as a tritium barrier; however, no significant development of coatings for this system has been conducted to date. Coatings for corrosion protection is currently considered as a backup position until more detailed requirements and specifications of the flibe chemistry are developed. Possible coatings considered for the flibe system are Mo plating or Al_2O_3 coatings. The corrosion protection requirements will depend on the selection of the structural material. Preliminary assessments indicate that coatings for a tritium barrier may not be adequate for this system because the tritium partial pressure in flibe may be too high. More complex approaches such as double-wall containment may be required for this system.

6. Conclusions

Coatings are proposed as the primary solution to critical materials constraints in several fusion first-wall/

blanket systems. Coating development conducted by the international fusion community is currently focused on two systems; viz., electrically insulating coatings to mitigate excessive MHD pressure drops in self-cooled lithium systems with a vanadium alloy structure, and as a TPB to reduce tritium permeation into the water coolant in the WCLL system with a RAM steel structure.

Development of electrically insulating coatings has focused on CaO and AlN coatings on vanadium alloys to mitigate MHD pressure drop effects in self-cooled lithium systems. Both CaO and AlN coatings have been formed in situ by exposure of vanadium alloy specimens to lithium with controlled chemistry and by vapor deposition processes. CaO coatings 10-30 µm thick formed in situ during exposure to Li with small additions of Ca at temperatures of 600-700 °C exhibit electrical resistivities about three orders of magnitude greater than required. AlN coatings exhibit good compatibility with Li containing specific additions of Al and N; however, AlN coatings formed in situ during exposure to Li with Al and N additions exhibit marginal electrical properties while the electrical properties of the vapor deposited coatings appear adequate. The stability of ceramic coatings exposed to Li is dependent on the purity of the ceramic and the Li chemistry.

Development of coatings as TPB for the WCLL system has focused on Al₂O₃ formed on aluminized RAM steels. Coatings have been produced by HDA, CVD, plasma spray and detonation jet processes. Coatings produced by the HDA and CVD processes provided the goal tritium barrier reduction factors of >1000 during H₂ gas exposure, but failed to meet the TBRF > 100 when exposed to Pb–Li with hydrogen charging. Both the HDA and VPS coatings exhibited good compatibility with Pb–Li at 480 °C; however, the CVD coatings exhibited significant degradation. High temperature (\approx 1000 °C) heat treatments appear necessary to produce high quality coatings on RAM steels that are compatible with Pb–Li.

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