



# Laboratory measurements of RAFM steel erosion and H retention in RAFM steel

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## Content:

- Introduction: Sputtering of mixed materials
- Erosion of EUROFER and model samples
- What do we need?



EUROfusion

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## Introduction



### What do we need to answer the question:

Can we use RAFM steels at some areas of the first wall of a future fusion power plant?

Certainly, steel is not an option for areas receiving a high power load and high particle flux.

And probably also not for areas receiving a non-negligible ion (plasma) flux.

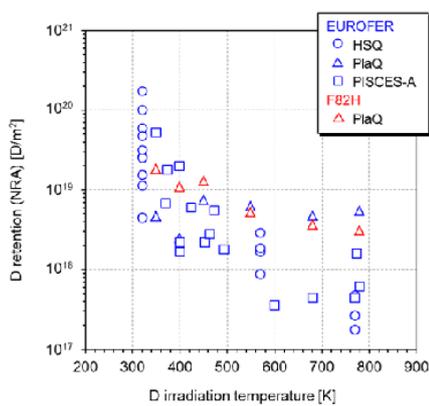


### Why should we use RAFM (reduced activation ferritic-martensitic) steel at all?

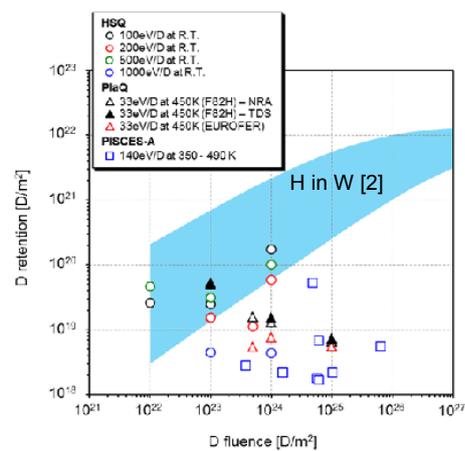
- Blanket modules for the first wall blankets are made of RAFM steel
- Technologically it would be much easier and less expensive
- H retention in RAFM steels is low, even lower than in W



### H isotope retention in steels even lower than in W



Temperature dependence of D retention in EUROFER and F82H [1] irradiated by D ion / exposed to D plasma under various conditions in HSQ, PlaQ and PISCES-A devices.



Comparison of fluence dependence of D retention between W [2] and RAFM steels (EUROFER and F82H).

[1] F82 H data: N. Ashikawa et al., Poster P2-020, ISFNT-12,2015

[2] ITPA SOL/Div topical group / B. Lipschultz et al., MIT report PSFC/RR-10-4



### Why should we use RAFM steel at all?

- Blanket modules for the first wall blankets are made of RAFM steel
- Technologically it would be much easier and less expensive
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### So what is the problem in using steel?



- Energy dependence of sputtering yield of Fe and W measured by weight loss & RBS (perpendicular ion incidence)
- Data fitted with Bohdansky formula
 
$$Y(E) = QS\eta^{KrC}(\epsilon) \left[ 1 - \left( \frac{E_{th}}{E} \right)^{2/3} \right] \left( 1 - \frac{E_{th}}{E} \right)^2$$

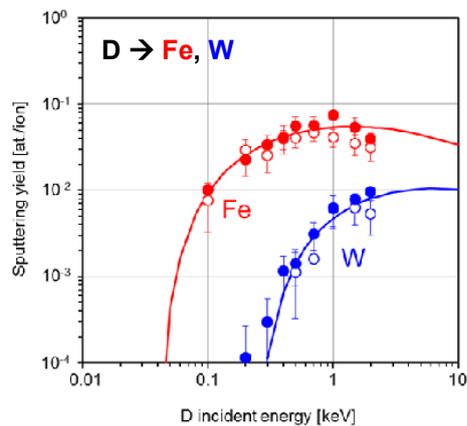
$$Q(D \rightarrow Fe) = 0.154 \text{ [at./ion]}$$

$$Q(D \rightarrow W) = 0.034 \text{ [at./ion]}$$

$$E_{th}(D \rightarrow Fe) = 37.5 \text{ [eV]}$$

$$E_{th}(D \rightarrow W) = 216 \text{ [eV]}$$
- Fe has lower sputter threshold and higher yield
- In relevant E region (50 to 1000 eV)  $Y_{Fe} > 10 * Y_W$

→ Fe (steel) not useable as PFM



Sputtering yields of Fe and W due to D bombardment as a function of D energy.

- Open circle: determined by weight-loss measurement.
- Closed circle: determined by RBS (Rutherford Backscattering Spectrometry).
- The curve is derived from the fitting by Bohdansky formula.



### Sputtering of pure Fe (the main component of steel) is too high!

But: steel is not pure Fe

**RAFM steels** (EUROFER, RUSFER, F82H)  
**contain small amounts (0.4 to 1.0 at.%) of W**

Sputter yield of W,  $Y_W$ , is much lower than  $Y_{Fe}$   
→ W enrichment / Fe depletion at the surface

This phenomenon is called “*preferential sputtering*”

Preferential sputtering will lead to a continuous change of the sputtering behavior



### The dynamic surface evolution due to preferential sputtering can be simulated by SDTrimSP

- SDTrimSP: dynamic version of TRIM.SP [1] (an earlier version was called TRIDYN [2])
- TRIM.SP describes the sputtering of surfaces due to impact of energetic species in the binary collision approximation
- TRIM.SP is well established and benchmarked with numerous experimental results
- SDTrimSP takes into account dynamic changes at the surface during sputtering, for example those due to preferential sputtering [3]
- Important for extrapolation to conditions not (easily) accessible to experiments (e.g. sputtering by tritium)

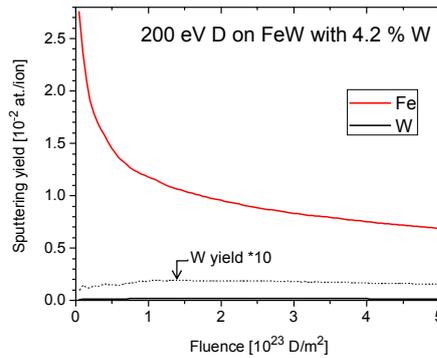
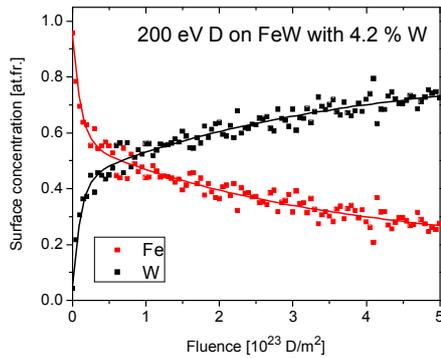
[1] W. Eckstein, Springer Series in Materials Science, Springer, Berlin, 1991  
[2] W. Möller, W. Eckstein, J. P. Biersack, Comput. Phys. Comm. 51 (1988) 355  
[3] Mutzke et al., IPP Report #12/8 “SDTrimSP, Version 5.00”, 2011



## SDTrimSP results: Dynamic Behaviour



- RAFM steels contain **W** which has a much lower sputter yield than Fe etc.
  - Preferential sputtering leads to W enrichment due to the difference of sputtering yields.
  - Erosion yield is reduced.



### Dynamic surface evolution due to preferential sputtering

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## Preferential Sputtering



The two most important factors for preferential sputtering:

- Max transferable energy for a given projectile/target combination
- Surface binding energy

Energy transfer in binary collisions: 
$$T_{max} = 4 \cdot \frac{M_1 M_2}{(M_1 + M_2)^2}$$

	$M_1$	$M_2$	$T_{max}$	$E_{Trans}$ at 200 eV (in eV)
D on W	2	184	0.043	8.6
D on Fe	2	56	0.133	26.6

Surface binding energy of W (in W!) = 8.7 eV

Fe (in Fe!) = 4.4 eV

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### Preferential sputtering

- Leads to enrichment of one component (transient phase until steady state)
- Reduces total sputter yield
- Effect increases with difference of sputter yield of the 2 components
- Occurs for all energies, but is strongest in the region between the 2 threshold energies

**SDTrimSP can simulate the dynamic surface evolution due to preferential sputtering**



### Preparation of EUROFER samples (W conc. = 0.42 at%)

- Specimens cut out from a EUROFER sheet (EUROFER 97-2 [heat 993 393])
- Surface polished to mirror-finish and pre-annealed at 800 K.

### Preparation of Fe/W binary system layers as "model" of RAFM steel

- Deposition by magnetron-sputtering from Fe and W targets
- Composition variable: Prepared W concentrations: 0.7, 1.5 and 4.2 at%.

**Fe/W model layers are used for benchmarking of SDTrimSP simulations.**



### D ion irradiation & plasma exposure

#### “High current ion source (HSQ)” ion-beam set-up (IPP-Garching)

- ❑ Conditions well-defined:
  - mass-separated mono-energetic  $D_3^+$  ion beam
- ❑ But relatively low D flux  $\leq 10^{19} \text{ m}^{-2}\text{s}^{-1}$

#### “PISCES-A” linear plasma device (UCSD) and Linear plasma device “PSI-2” (FZJ)

- ❑ High flux ( $D^+$ ,  $D_2^+$ ,  $D_3^+$ ) plasma  $\sim 10^{21} \text{ D}^+/\text{m}^2\text{s}$
- ❑ But possible influences of plasma impurities (e.g. O) and redeposition



### Post-irradiation analysis

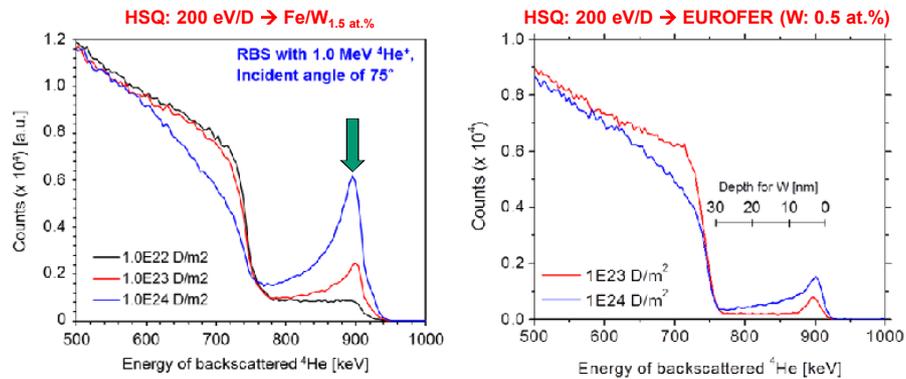
- ❑ **Weight-loss measurement**
  - the only applicable technique to determine the sputtering yield of bulk materials, e.g., EUROFER steel
- ❑ **Rutherford Backscattering Spectroscopy (RBS)**
  - with 1 MeV  $^4\text{He}^+$ : determination of surface composition and measurement of sputtered amount
- ❑ **Scanning Electron Microscopy (SEM)**
  - Surface morphology examination



## Experiment: Post-irradiation Analysis



- Surface composition changes with D irradiation fluence (HSQ exposure).
- W concentration at the top surface increases with fluence (for all D impinging energies).



RBS spectra obtained from Fe/W<sub>1.5</sub> at.% and EUROFER steel irradiated by 200 eV/D with different fluences: 1.0e22, 1.0e23 and 1.0e24 D/m<sup>2</sup>.

→ Predicted effect of surface enrichment experimentally proven

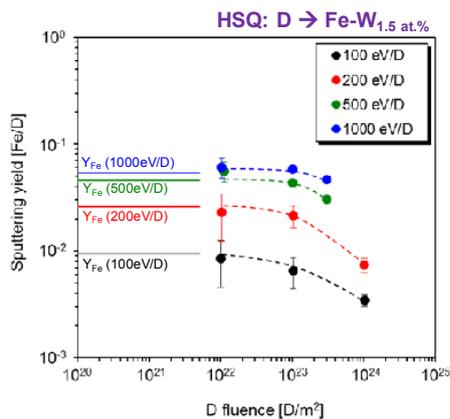


## Erosion of Fe/W Model Layers



### Decrease of sputter yield with increasing fluence

- Initial level similar to that for pure Fe ( $Y_{Fe}$ ) (solid lines)
- Clear decrease with fluence (in range of  $\geq 10^{23}$  D/m<sup>2</sup>)
- Yield reduction by 30 to 50% compared with that of pure Fe at  $10^{24}$  D/m<sup>2</sup>



Sputtering yield of Fe/W (W ~1.5 at.%) layer by D ion irradiation with different D energies as a function of D fluence (320 K)

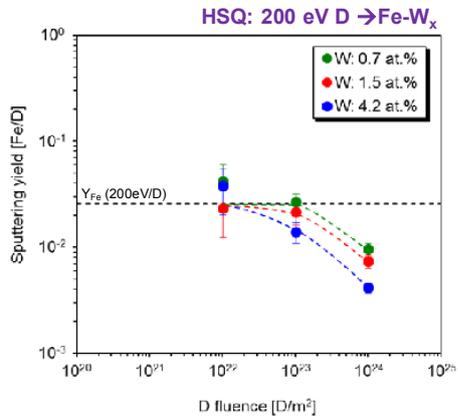


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- Yield reduction by 30 to 50% compared with that of pure Fe at  $10^{24}$  D/m<sup>2</sup>
- Yield reduction depends also on the initial W content in the Fe-W binary layer.



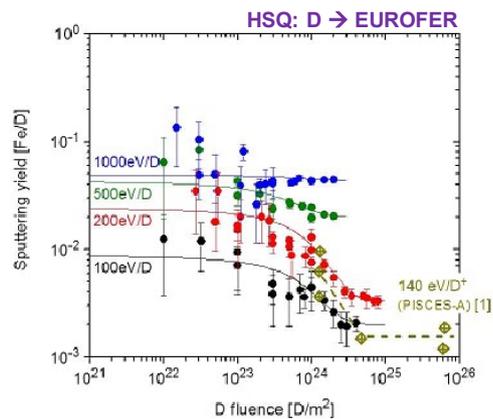
Sputtering yield of Fe/W layers with different W content by 200 eV/D ion irradiation as a function of D fluence (320 K)



## Erosion of EUROFER



- Yield reduction in the higher fluence range ( $\geq 10^{23}$  D/m<sup>2</sup>), as well as for Fe/W layer.
- For 200 eV/D steady state seems to be reached for fluence  $> \sim 5 \times 10^{24}$  D/m<sup>2</sup>.
- PISCES-A data<sup>[1]</sup> at very high fluence and 140 eV/D also indicate steady state for fluence  $> \sim 5 \times 10^{24}$  D/m<sup>2</sup>.



Sputtering yield of EUROFER steel by D ion irradiation with different D energies as a function of D fluence (320 K)

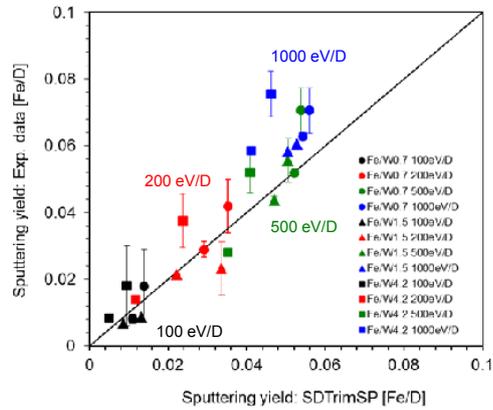
[1] J. Roth et al., J. Nucl. Mater. **454** (2014) 1



## Comparison SDTrimSP – Experiment



- Fe/W binary layers: Experimental data and SDTrimSP result agree within ~ 30 %



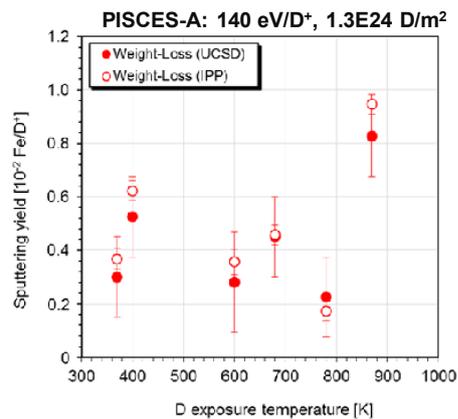
Comparison of sputtering yields between SDTrimSP calculation result and experimental data obtained for Fe/W binary layers with different W content.



## Temperature Dependence



- Exposure of EUROFER to low-energy (140 eV/D<sup>+</sup>) / high-flux (~10<sup>21</sup> D<sup>+</sup>/m<sup>2</sup>s) plasma at various temperatures (370 - 870 K).
- Sputtering yield varies within a limited range at < ~800 K, while it clearly increases at 870 K.
  - consistent trend with the numerical prediction.
  - No clear temperature dependence of sputtering in the DEMO FW working temperature range (< 800 K)



Sputtering yield of EUROFER steel by 140 eV/D<sup>+</sup> exposure as a function of exposure temperature (measured at PISCES-A)

[1] J. Roth et al., J. Nucl. Mater. **454** (2014) 1

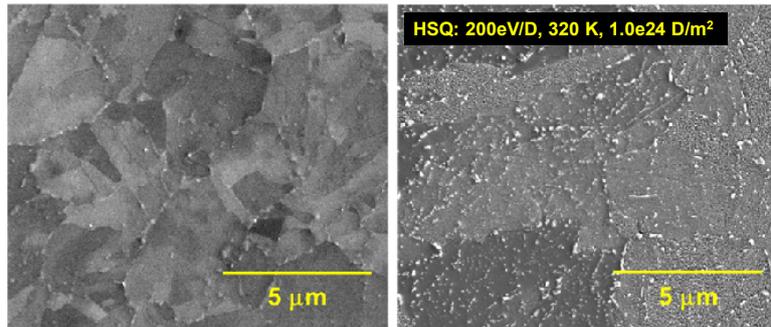


## Surface Morphology



### Surface morphology change of EUROFER

- EUROFER surface sputtering is not homogeneous...:
  - grain-dependent erosion.
  - high-Z precipitates.
  - nano-scale roughness.



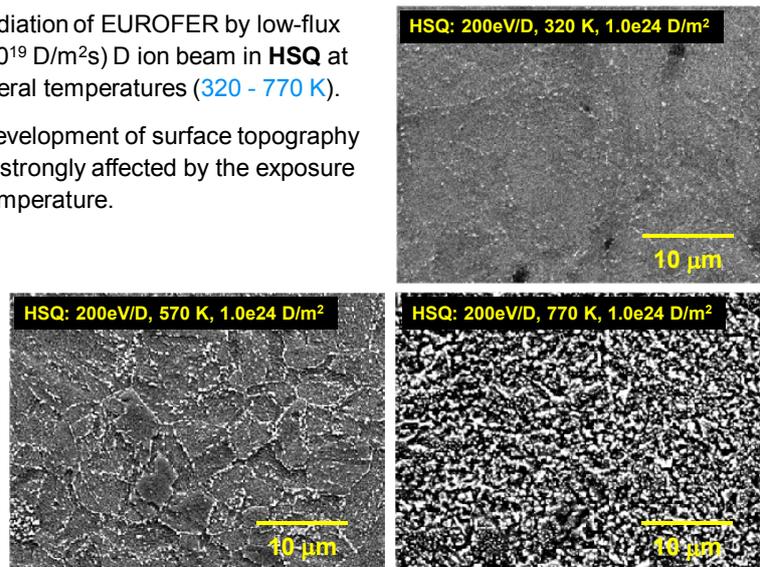
SEM images of EUROFER steels: as-deposited and after D irradiation with 200 eV/D, 1.0e24 D/m².



## Surface Morphology



- Irradiation of EUROFER by low-flux ( $\sim 10^{19}$  D/m²s) D ion beam in HSQ at several temperatures (320 - 770 K).
  - Development of surface topography is strongly affected by the exposure temperature.

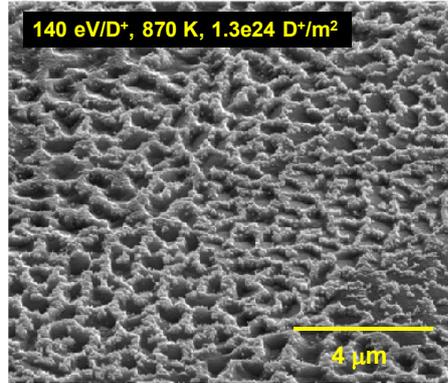
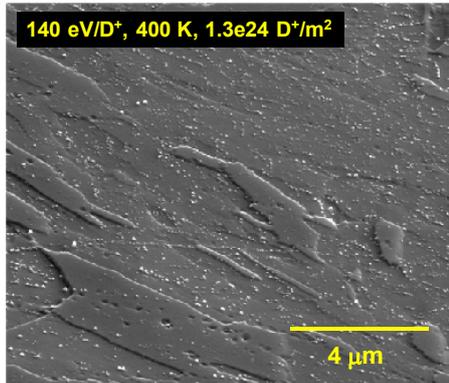




## Surface Morphology



- EUROFER exposed to PISCES-A plasma: Development of surface topography is strongly affected by the exposure temperature as well.



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## Surface Morphology



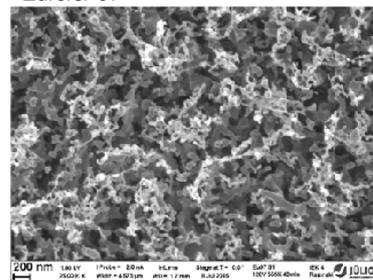
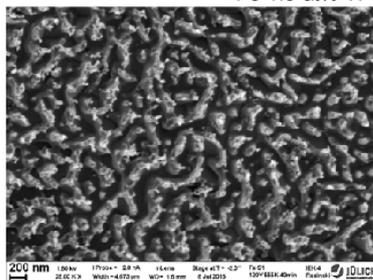
PSI-2: 555 K, 90 eV,  $2.6 \times 10^{25}$  D m<sup>-2</sup>

Fe 1.5 at% W

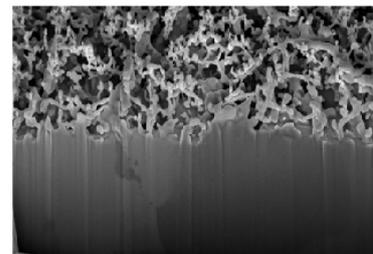
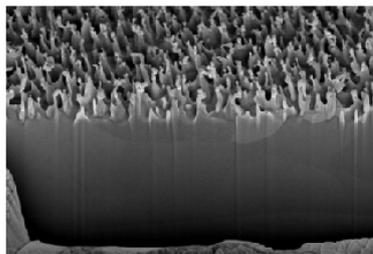
Eurofer-97



surface



cross section



IAEA CRP "Steel"



### Temperature Dependence

- T dependence of sputter yield
- Onset of diffusion (counteracting enrichment?)
- T dependence of surface morphology

### Impurity sputtering

- Higher mass → higher sputtering of W
- Ions: higher energy due to sheath acceleration



- Erosion of RAFM steel and model systems was investigated in ion beam experiment and in linear plasma devices
- Surface enrichment of W and reduction of sputter yield were experimentally proven
- For the model layers reasonable agreement with theoretical predictions (SDTrimSP)
- Reduction of EUROFER sputter yield by factor up to 8 (at 200 eV)
- Reduction possibly strongly influenced by surface morphology development
- H retention in steel is low (even lower than in W)





## Summary



### Where could RAFM steel be used?

- First wall in areas without plasma contact
- Impinging flux “only” CX neutrals
- CX neutrals have a very wide E distribution, but dominantly low-E (< 200 eV) hydrogen isotopes
- Under such conditions W enrichment (and the corresponding reduction of the sputter yield) might be effective

### Why “no plasma contact”?

- Impurity ions (higher mass and higher energy)
- Higher mass → better E transfer → higher sputtering of W
- Ions, sheath acceleration → higher E → higher sputtering
- Under such conditions W enrichment probably not effective



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## What is still needed?



### What do we need to answer the question:

Can we use RAFM steels at some areas of the first wall of a future fusion power plant?

### What is still needed?

- From lab results: better understanding of T dependence and surface morphology effects
- Improved surface diagnostics
- Influence of impurities
- From the fusion plasma side: mass and energy distribution of impinging particle fluxes



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