Supplementary Material for: High-solar-absorptance CSP coating characterization and reliability testing upon isothermal and cyclic loads for service-life prediction

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Adhesion testing

he release behaviour was measured with a standard pull-off test, using a metallic punch with a diameter of 2 cm, initially pressed into contact with the coating surface after applying a thin layer of adhesive. The adhesive used was a two-component epoxy glue (LOCTITE® EA 9466™).

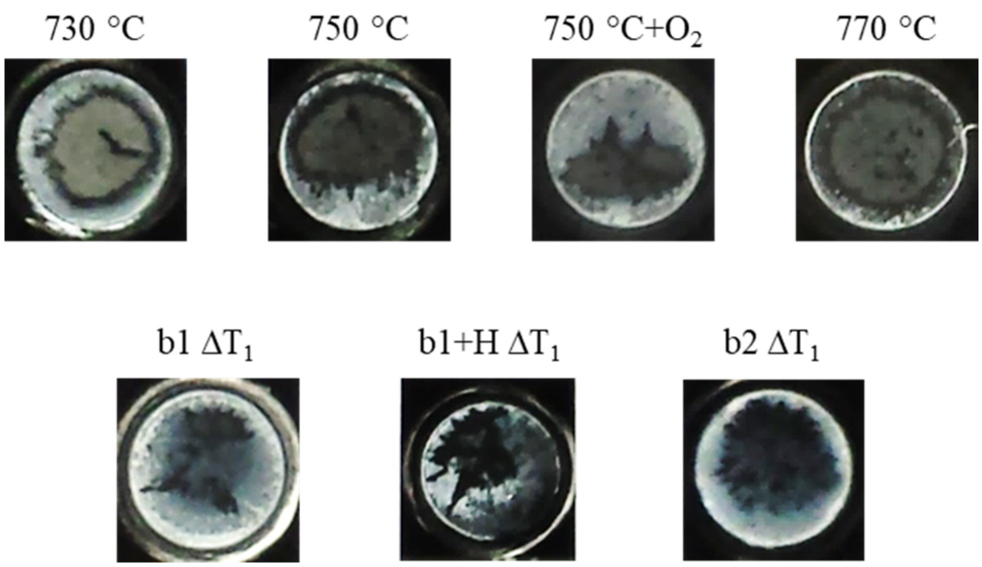

Figure S1: Photograph of the testing surface by pull-off test after 18 weeks of different thermal loads.

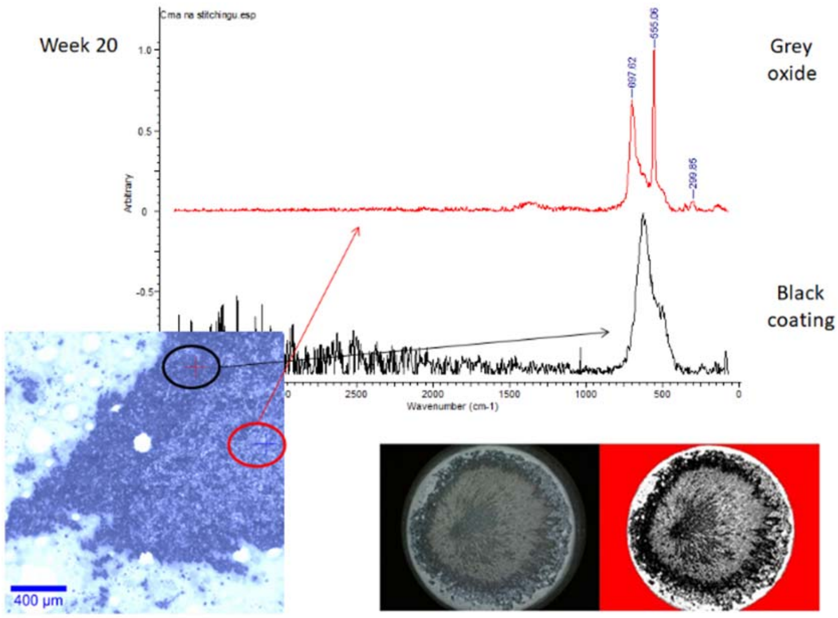

Figure S2: The evaluation of the delamination layers was obtained from the optical images (A) presented above. The B&W images were evaluated by WolframAlpha software, where black pixels were determined as the coating (B444), grey pixels as the oxide and white pixels as the delamination along the glue. As support for a proper evaluation of the delamination layer (optical image of delaminated surface with corresponding areas where spectra were collected) confocal Raman microscopy was used with representative Cr_2O_3 and coating spectra. The ratio between the delamination surfaces is presented in Fig. S3.

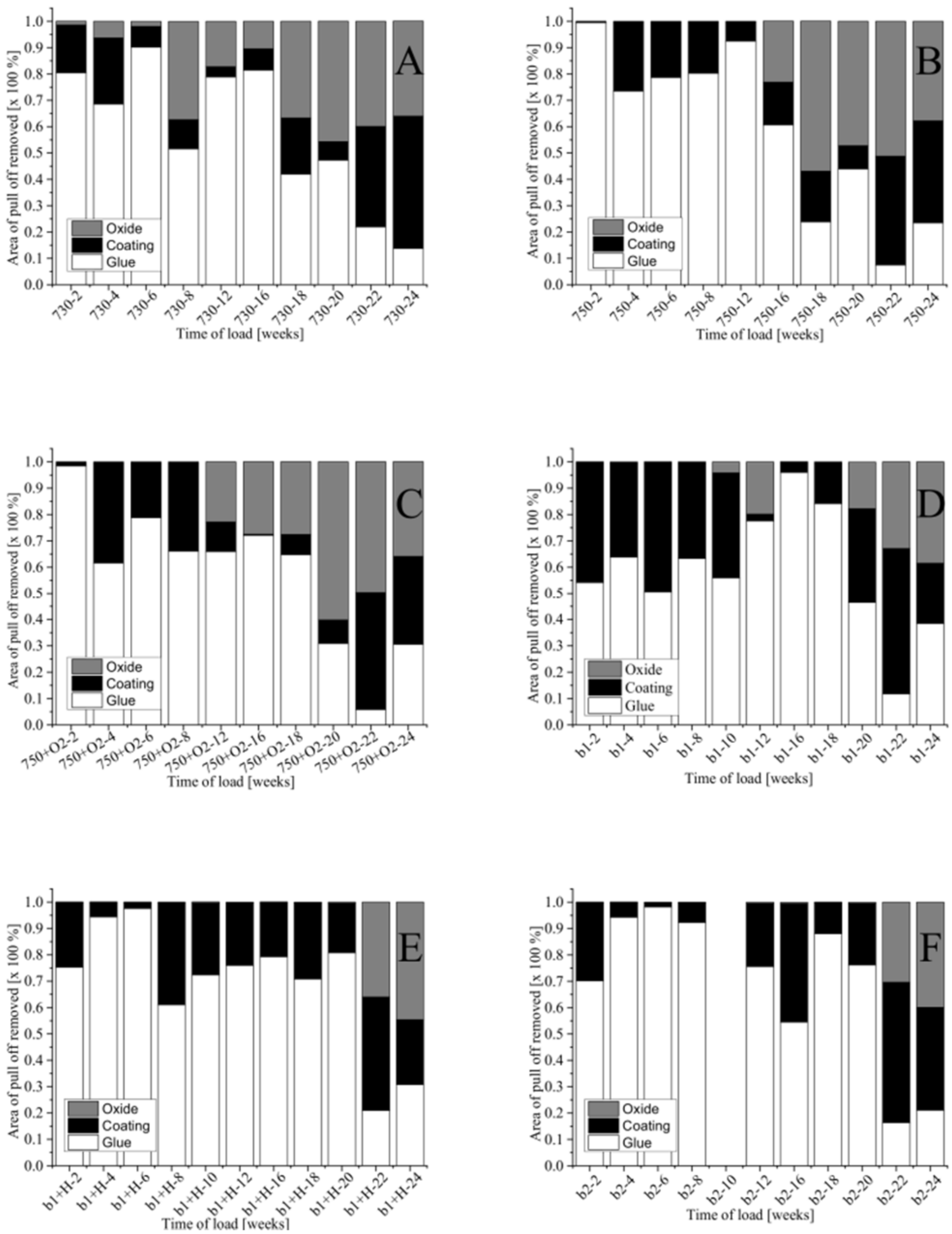

Figure S3: Ratio of areas for three different species (oxide, coating, glue) after pull-off testing vs. isothermal loading time at 730 °C (A), 750 °C (B), 750 °C +O_2 (C), b1 ΔT_1 (D), b1+H ΔT_1 (E) and b2 ΔT_1 (F).

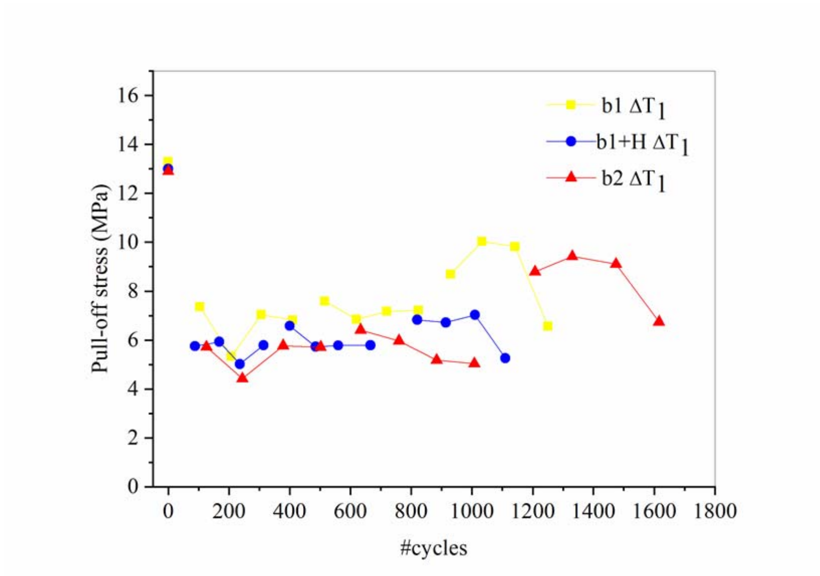

Figure S4: Pull-off stress as a function of the loading time for a cyclic load.

Oxide-thickness evaluation

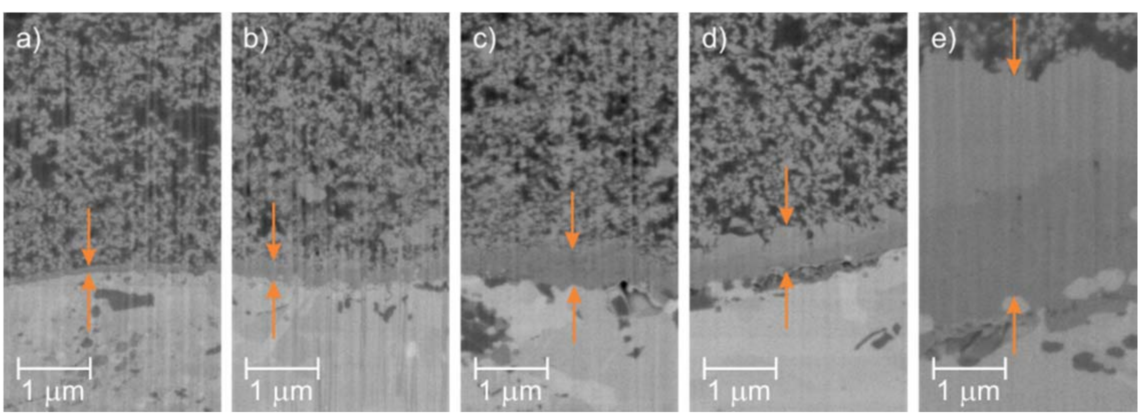

Figure S5: FIB-SEM cross-sectional image of oxide-layer formation on Inconel support material for the case of: a) Initial sample B444 [average 110 nm], b) sample b1 ΔT_1/12 weeks (620 cycles) [average 320 nm], c) sample b1 ΔT_1/24 weeks (1142 cycles) [average 590 nm], d) sample 750/2 weeks [average 670 nm], e) sample 770/14 weeks [average 3,1 µm].

FIB coating lamella preparation

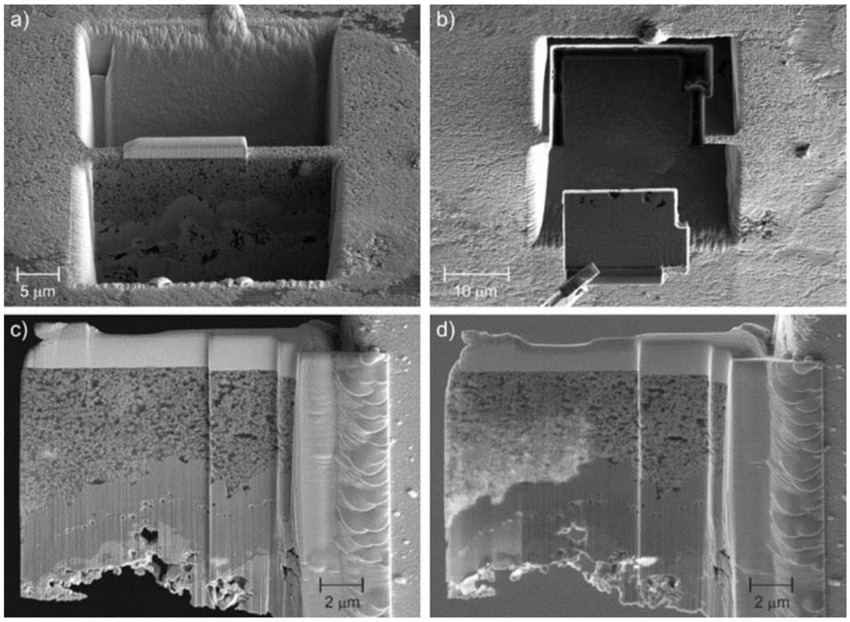

Figure S6: FIB lamella TEM sample preparation: a) FIB lamella bulk milling, b) rough lamella lift out, c) sequential lamella thinning using high-energy focused ion beam, d) thinned lamella polishing using low-energy focused ion beam.

FIB 3D coating evaluation

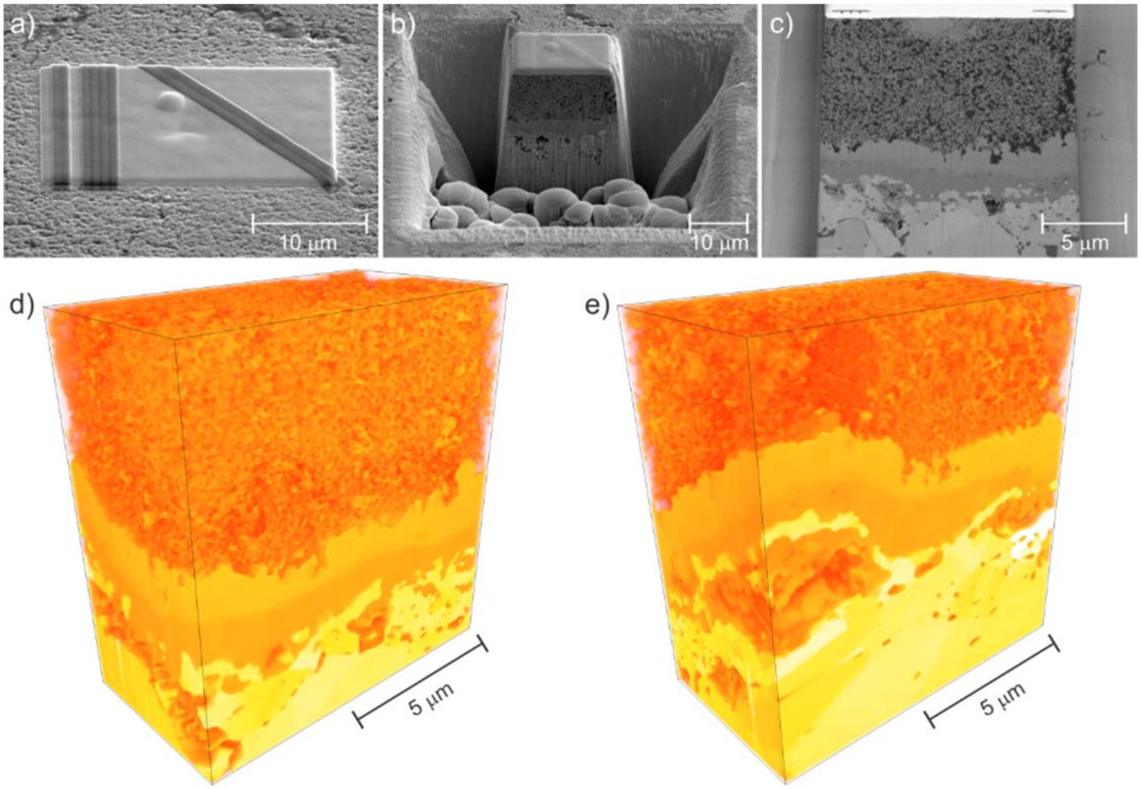

Figure S7: FIB 3D analysis of sample 770-14W: a) fabrication of Pt protective layer with embedded reference markers, b) U-Shaped region of interest preparation by FIB milling, c) Z- contrast cross-sectional SEM image of the first acquired slice-image, d) three-dimensional sample visualization using volume rendering (front view), e) three-dimensional sample visualization using volume rendering (back view).

Incorporation of Cr in the coating layer1 [1]

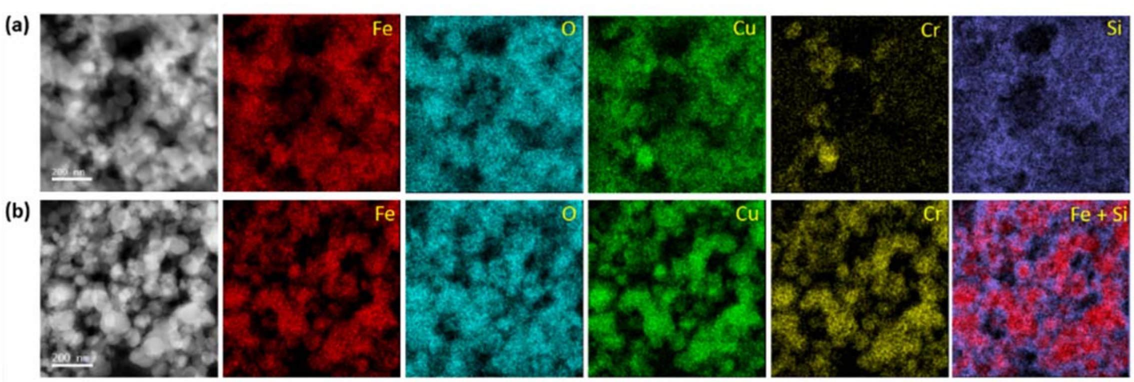

Figure S8: (a) EDX elemental maps acquired from the mid-section of the B444 coating, where Cr segregation is observed: only some particles imaged on the mapped area show a Cr signal. (b) EDX elemental maps acquired from a section of the B444 coating located very close to the interface with the oxide layer. The Cr signal, in this case, is uniformly distributed along the particles. To visualize the Si coating on the particles, a compositional map of Fe and Si is displayed as well .

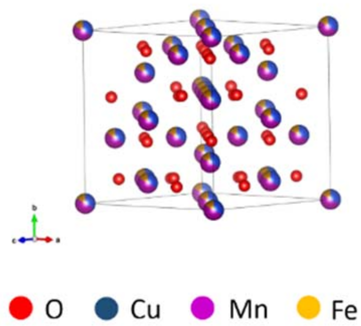

Figure S9: $CuMn_{1.5}Fe_{0.5}O_4$ spinel structure.

Table S1: Boiler temperatures in Ivanpah, USA facility. Additional data for the total annual number of cycles is 330 +100=430 (including cloudy days).

Load case	Start up	75%-MCR	Shutdown	After SD	
Pass III	440	682.6	460	250	
Pass VI	460	706.9	480	250	
Pass V	480	716.7	500	250	**Total**
Tot. Yr. hrs	**144**	**2300**	**162**	**660**	**3753**

Table S2: Fitting constants for Eq. 16.

load	D(T)	τ(T)
730	0.0261	371.4
750	0.0273	196.7
750+O2	0.0294	281.1
770	0.0380	178.2
b1 ΔT_1	0.0152	1003.1
b1+H ΔT_1	0.0139	1043.7
b2 ΔT_1	0.0145	1041.0

Table S3: Measured values of the selected pull-off tests (after at least 18 weeks of exposure). Oxide-layer thickness is calculated using Equation 16. Individual delamination rations are obtained from the pixel evaluation in the bitmaps of recorded photos.

load	time (weeks)	S_g/S_0	S_P/S_0	S_{ox}/S_0	d(t)	F(t)/S_0
b1 ΔT_1	20	0.465	0.357	0.177	1.0	10.03
b1 ΔT_1	22	0.118	0.553	0.329	1.0	9.82
b1 ΔT_1	24	0.385	0.23	0.385	1.1	8.56
b1+H ΔT_1	18	0.708	0.289	0.003	0.9	6.83
b1+H ΔT_1	22	0.21	0.43	0.36	1.0	9.03
b1+H ΔT_1	24	0.308	0.246	0.446	1.0	8.26
b2 ΔT_1	20	0.762	0.233	0.005	1.0	9.42
b2 ΔT_1	22	0.164	0.532	0.304	1.0	9.10
b2 ΔT_1	24	0.211	0.39	0.399	1.0	8.74
730	18	0.42	0.213	0.367	1.5	8.70
730	20	0.473	0.0701	0.457	1.6	7.66
730	24	0.138	0.502	0.361	1.7	6.55
750	18	0.239	0.192	0.57	1.6	8.94
750	20	0.44	0.0879	0.472	1.6	8.45
750	24	0.235	0.387	0.378	1.8	7.66
750+O_2	18	0.648	0.0764	0.276	1.7	8.15
750+O_2	22	0.058	0.445	0.497	1.8	7.80
750+O_2	24	0.306	0.335	0.36	1.9	7.70
770	18	0.337	0.232	0.431	2.2	6.36
770	22	0.062	0.433	0.504	2.4	5.30
770	24	0.254	0.293	0.453	2.5	5.72

Reference:

1. Noč, L., Ruiz-Zepeda, F., Merzel, F. & Jerman, I. High-temperature ion baseball for concentrating solar power efficiency enhancement (Submitted to Solar Energy Materials and Solar Cells).