SUPPORTING INFORMATION
for the paper
Towards CO_2 Electroreduction under Controlled Mass Flow Conditions: A Combined Inverted RDE & Gas Chromatrography Approach

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1. SEM and EDX Mapping of Alumina and Diamond Polished Silver RDEs

In a recent work (cited as Ref. 26 of the paper), Monteiro and Koper described an interesting phenomenon; namely, that the contamination of gold electrodes with alumina particles by electrode polishing leads to an enhancement in activity for hydrogen evolution (HER). In order to see whether alumina particles also exert an effect on the results of CO_2 electroreduction on silver RDEs (studied in our paper by the iRDE&GC hyphenation), we used both alumina and diamond suspensions (both of 50 nm particle size) for the polishing of an Ag RDE.

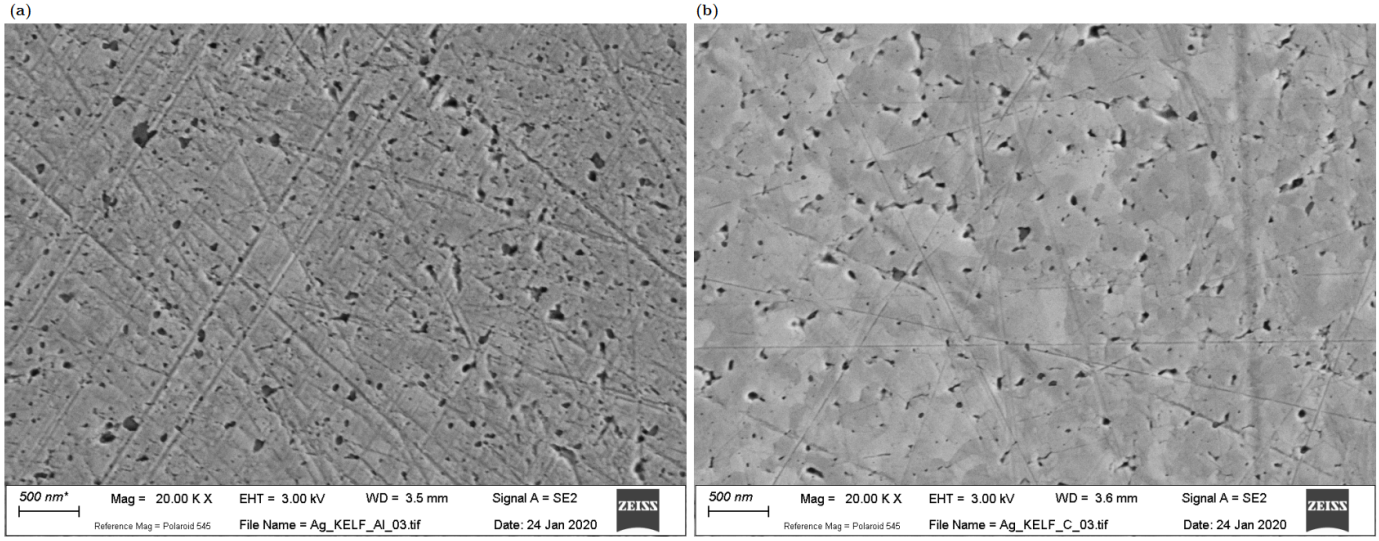

Figure S1: SEM micrographs of the surface of an Ag RDE polished by 50 nm alumina (a) and diamond (b) particles. (Zeiss Gemini 450 SEM, Germany; accelerating voltage: 3 kV, working distance: 3.5–3.6 mm.)

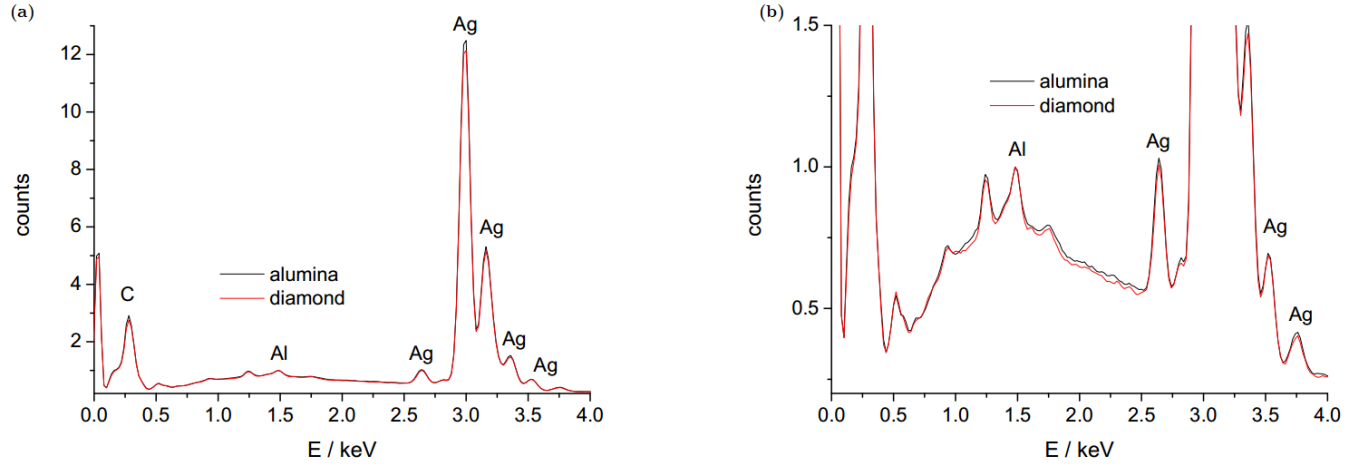

Figure S2: EDX spectra of an alumina and a diamond polished Ag RDE surface, overlapped in one plot, shown at different scaling in (a) and (b). The spectra were normalized to the Ka line of aluminum (1.48 keV). The main peaks are labelled (Ag, C and Al). An accelerating voltage of 10 kV and a working distance of 8.5 mm was applied.

SEM micrographs of the surfaces, shown in Figure S1, revealed no major differences between the two surfaces polished by different materials. The EDX spectra of the two surfaces, Figure S2, also showed minor if any differences. EDX revealed that the diamond-polished surface contains ~ 0.4 wt% Al, while the one polished by alumina (and then rinsed abundantly with MilliQ water) showed only a ~ 0.3 wt% (that is, even less) Al content.

We are aware that EDX may not be sensitive enough to indicate small Al contaminations that can already have a significant effect on the electrochemical measurements, thus we also repeated an iRDE&GC experiment under the same conditions that we applied for Figure 7 of the paper. By applying a current density of −2.6 mA and a rotation rate of 625 min^{-1} we detected a 93.8% ± 4.5% Faradaic efficiency for the production of CO on an Ag iRDE polished by a diamond suspension; for the alumina-polished electrode, this value was 90.5% ± 4.3%. Although there are some minor differences, pointing in the direction suggested by Ref. 26, it seems that the iRDE&GC hyphenation is not sensitive enough to point these out.