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Hot electron electrochemistry at silver activated by femtosecond laser pulses

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The fabrication of the extremely small silver working microelectrode with a diameter of 30 μm required an elaborate procedure. They were fabricated by embedding a purchased polycrystalline silver wire (Johnson Matthey, >99.5% Ag) with a diameter of approximately 30 μm in epoxy resin. A graphical step-by-step instruction of the fabrication process is given in Fig. S1.

The orifice of a discardable 5 ml syringe (DB Discardit II) was cut off. The plunger was removed and the formed opening was widened with a round file to such an extent, that a 2 mm socket (RS Components, 102-866) could be inserted comfortably. A head-less match was fixed across the handle plates of the syringe with hot glue. The corpus of the socket was then coated with silicone rubber to prevent resin from infiltrating it during the filling process. The silver wire was then lead through the syringe opening and soldered on the rubber-free contact of the socket. The socket was subsequently fitted in the opening and fixed with super glue. The wire was carefully tightened and fixed to the match on top

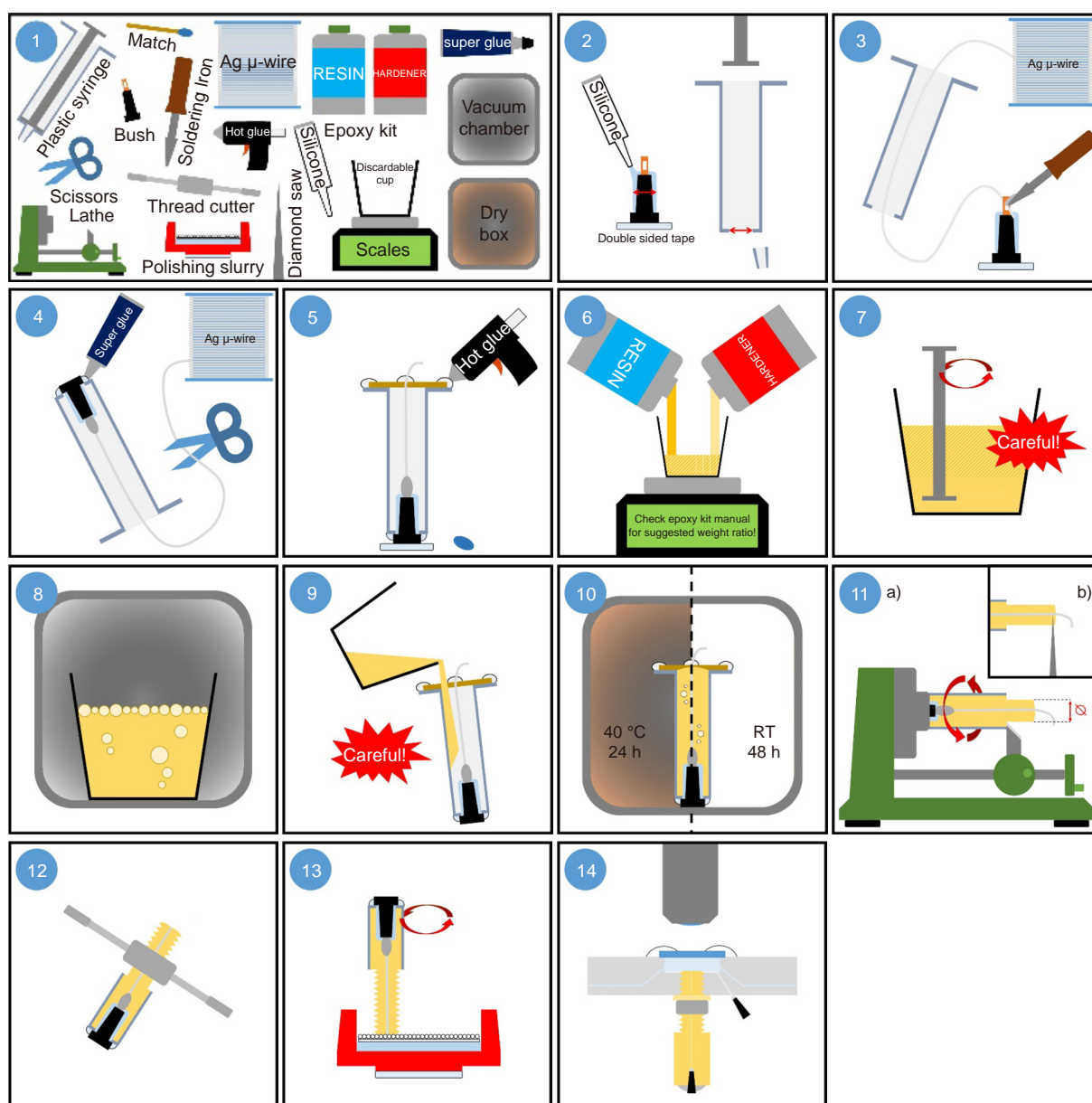


Fig. S1 | (1) Required materials and devices. (2) Bush sealing and syringe preparation. (3) Soldering of silver wire to bush. (4) Fixing of bush to syringe base. (5) Tightening of the silver wire. (6) Mixing of resin and hardener according to the suggested weight ratio. (7) Careful stirring to avoid excessive bubble formation. (8) Degassing of the mixture in a vacuum chamber. (9) Careful casting to avoid rupturing of the wire. (10) Curing of the casting in a dry box according to the resin/hardener manual. (11a) Lathe cutting to the wanted diameter. (11b) Top clipping with a diamond saw. (12) Thread cutting. (13) Surface polishing with Al_2O_3 slurries. (14) Quality inspection and measurements.

of the syringe. The needed amounts of casting resin and hardener were weighted and cautiously mixed to avoid bubble formation.

For electrode casting, Araldite DBF (Huntsman) resin and Aradur HY 951 (Huntsman) hardener was used. The viscosity of this compound is very low at room temperature and therefore suitable for cast filling, the pot life is long and degassing in vacuum was easily achievable. The carefully stirred and degassed liquid epoxide mixture was slowly poured into the prepared syringes, which were then left for curing for several days as specified by the manual. After hardening, the upper part of the syringe featuring the handling plates, the match and protruding epoxy resin were cut away with a diamond saw. The diameter of the electrode was then adjusted on a lathe to approximately 8.16 mm for subsequent M10 thread cutting. The exposed surface was finally gently abraded on a 4000 mesh SiC grinding paper (Struers) and then stepwise polished with Al₂O₃ slurries as described in Table S1. After each polishing step, the microelectrode was thoroughly rinsed with distilled water and shortly cleaned in an ultrasonic bath to remove residual polishing grains. Before being inserted into the electrochemical cell, the electrode was cleaned by briefly dipping it into 2M HNO₃ and spectroscopic grade methanol and rinsing it with distilled water.

Table S1 | Conducted polishing steps. The polishing powders and cloths were provided by Buehler GmbH.

Powder	Particle size (µm)	Polishing cloth	Lubricant
Al ₂ O ₃	9	Nylon	H ₂ O dest.
Al ₂ O ₃	5	Nylon	H ₂ O dest.
Al ₂ O ₃	1	Nylon	H ₂ O dest.
Al ₂ O ₃	0.3	Nylon	H ₂ O dest.
Al ₂ O ₃	0.05	Microcloth	H ₂ O dest.