Investigation of the R20 Experiment of Tadahiko Mizuno and Jed Rothwell

 Last revision 25 May, 2023

The work presented in this document explores the materials and processes described in a paper published in July 2019 by Mizuno and Rothwell (“the Report”), titled ”Increased Excess Heat from Palladium Deposited on Nickel” [1]. Links to that document and a Supplement [2] with further details are included in the references at the end of this report. Please refer to those documents for fuller details of the experimental context not included here.

1. A quantity of the Nickel mesh identical to that used by Mizuno was provided by Rothwell for evaluation and replication attempts. A sample of the material 50 x 200 mm was removed from a full sheet for investigation of the treatment protocol as described on pg.16 of the Report. Individual pieces ~25 x 50 mm were then cut from that sample for testing. The described procedure was followed incrementally, and the material was examined by SEM/EDS after each step.

2. The material received was as described in the Report, a 2-2 twill weave of 0.055 mm wire and ~1.6 mm pitch. It was found to be ~99% Ni, with very few foreign particles and inclusions visible.

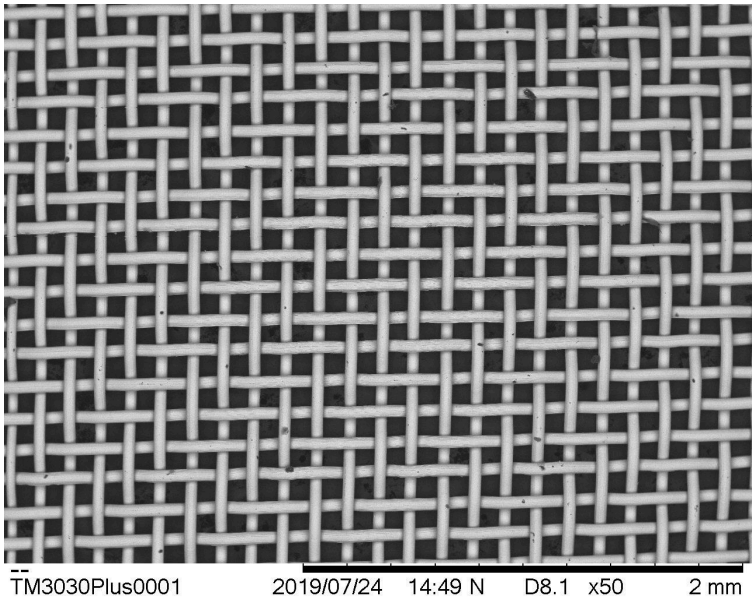


Image 1. Nickel mesh as received.

3. The treatment procedure shown in the Report was followed. Locally available supplies equivalent to those described were used, specifically Dawn Platinum dish detergent and 3M Wet/Dry SiC sandpaper. Next the 1-hour soak in tap water at 90°C was done. Methanol was substituted for the Ethanol specified in the Report for the final rinse. The sample was then air-dried for about 10 minutes and examined by SEM/EDS.

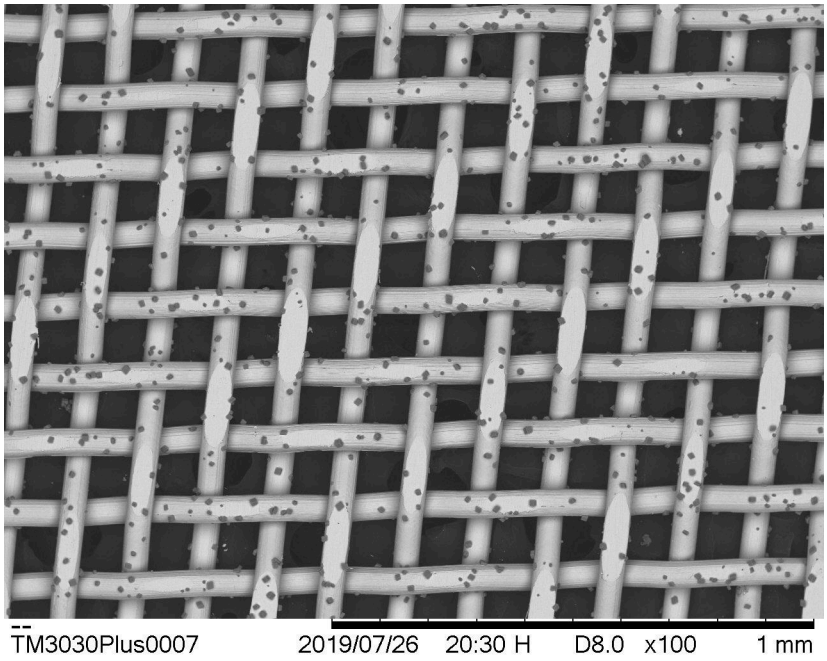


Image 2. After treatment by sanding and 60 minute soak in 90°C tap water.

Many small crystals had appeared on the surface of the mesh. Further analysis by EDS showed these crystals to be composed of Calcium, Oxygen, and a bit of Carbon. The composition and predominant cubic morphology(5) suggest that these are composed of Calcite, the stable crystal form of Calcium Carbonate $CaCO_3$.

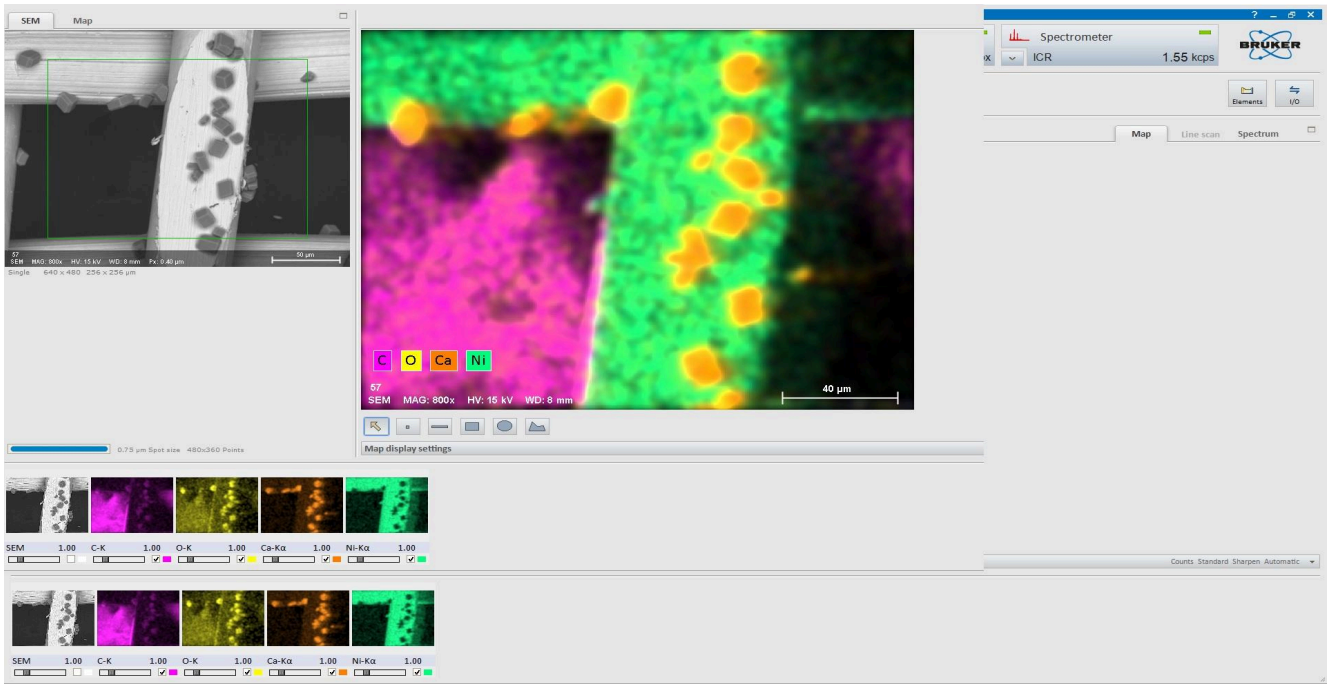


Image 3. SEM/EDS showing $CaCO_3$ crystals deposited by soaking in tap water after sanding.

4. Since neither the sandpaper nor the detergent contain any Calcium, the most likely source is the tap water. To test this theory, the treatment was repeated with a new sample of mesh, using deionized water for the 60 minute soak. The resulting image shows a clean surface with complete absence of the crystal deposits.

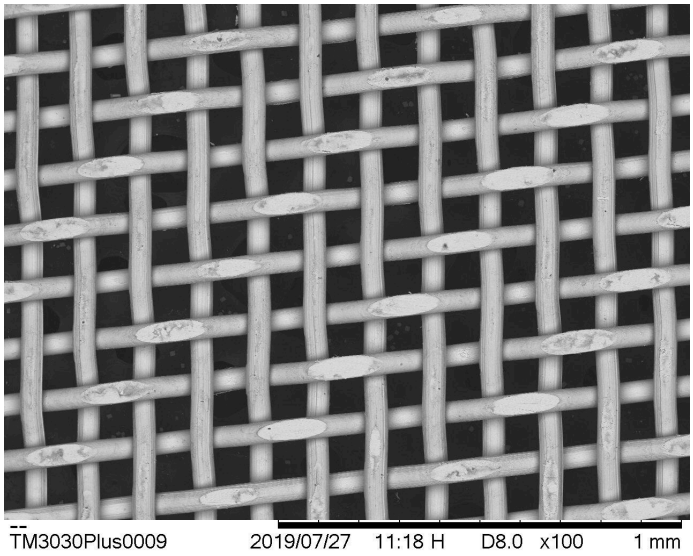
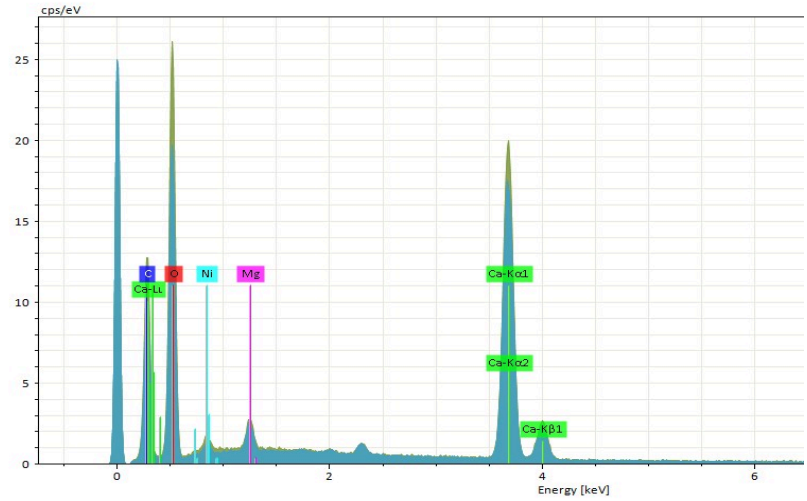


Image 4. Following sanding and soak in deionized water: no crystal deposits are seen.

5. Analysis of crystals

The sample above was soaked in tap water at 90°C for 60 minutes, then rinsed in Methanol. The crystals appeared as previously described. EDS analysis of a single crystal confirmed the composition as $CaCO_3$.



	Ca	C	O_3	Total mass %
Element percentage per $CaCO_3$ molecule	20.00	20.00	60.00	100.00
Element percentage by normalized mass from EDS	21.85	18.62	56.36	96.83

6. A 1g Pd chip (Valcambi, 99.95%) was annealed with an oxygen-rich Propane flame, then sanded flat, carefully cleaned and attached with cyanoacrylate adhesive to an Aluminum sample holder.

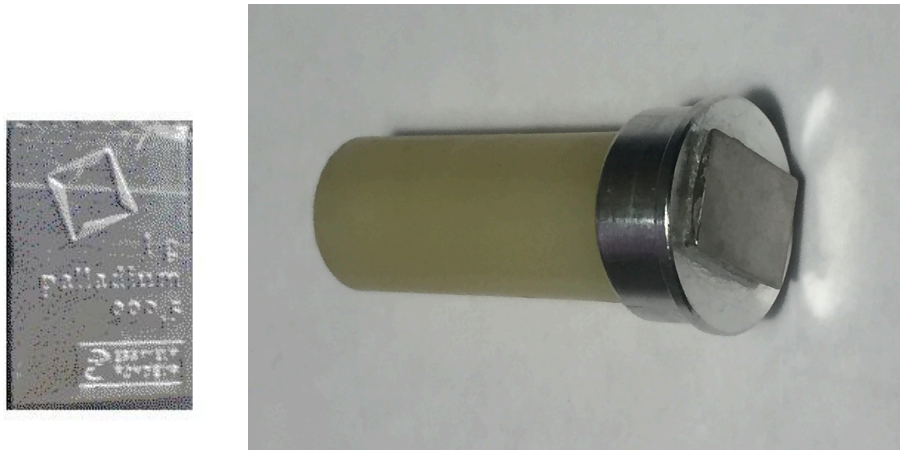


Image 5. Pd chip and attachment to a sample holder.

The Pd was rubbed on the prepared mesh sample on a piece of plate glass, with about 30 strokes in each of two directions at moderate (~1-2 kg) pressure. SEM examination showed Pd deposited primarily at the edges of the spots flattened by previous sanding. This seems to result from the sharp burrs left at the edges of those areas. Smaller deposits of Pd are seen at other high spots of the mesh.

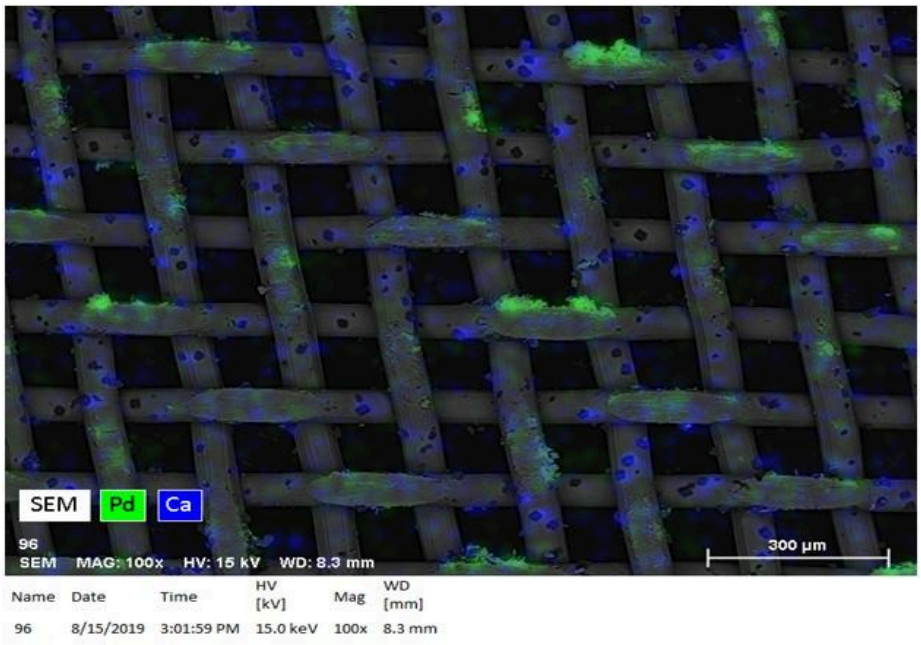


Image 6. Pd deposited by brief rubbing with annealed Pd.

In some of the deposits, fragments of the Calcite crystals could be seen attached to the Pd. In a few of those, the crystals appeared to have been broken up, leaving sub-micron size particles of $CaCO_3$ embedded in the Pd, as shown by SEM/EDS in Image7 below.

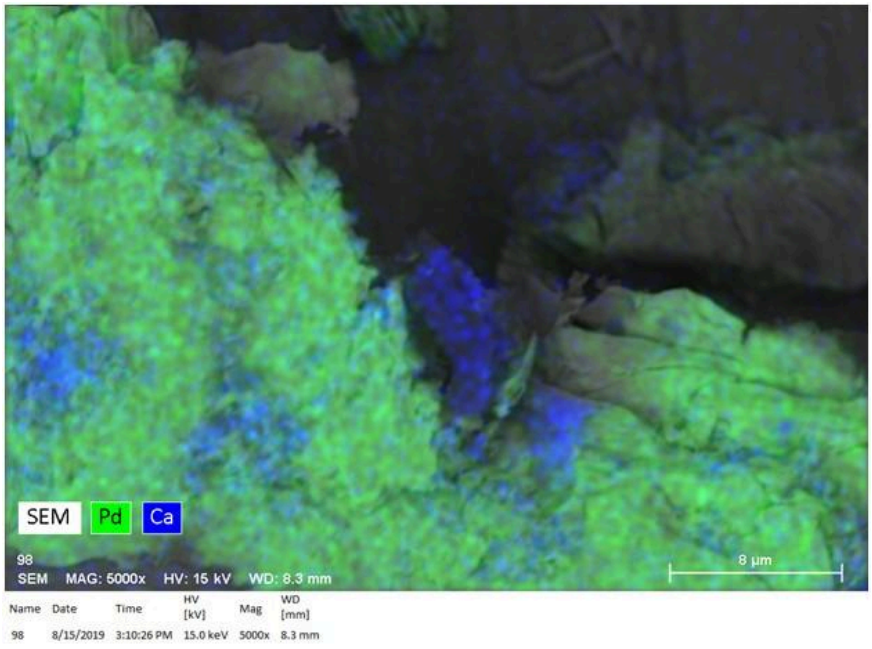


Image 7. Pd deposited by rubbing on the mesh, showing embedded $CaCO_3$

7. Tests of Nickel Foil

It was suggested by Ed Storms that a similar mechanical process can be applied to a flat plate or foil of Ni. I tried this on a piece of polished Ni foil and initially saw very little deposition of Pd. Then I sanded the foil with 1000, 400 and 100 grit applied to different test areas, followed by the usual 1 hr soak at 90°C in tap water.

These images show that the size and thus mass of deposited Pd is greater with the coarsest (100) grit sanded surface. The rubbing also crushed the CaCO3 crystals and mixed the resulting ~1um particles into the deposits of Pd. The SEM/EDS image 9 below shows this effect.

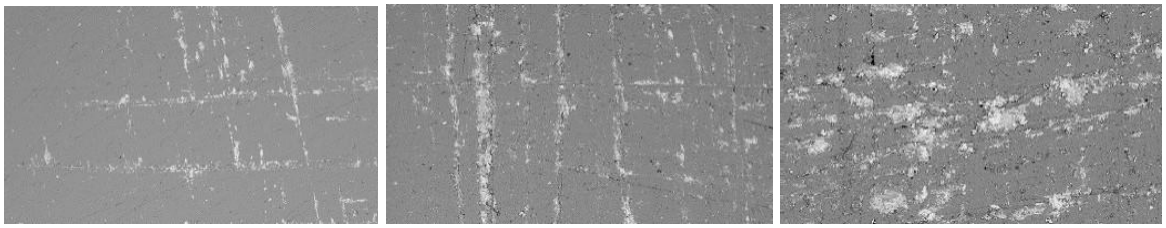


Image 8. Results of sanding with 1000, 400 and 100 grit, followed by rubbing with Pd

These tests show that sharp edges are created at the perimeter of the sanded flats on mesh, and at the sanding scratches on the Ni foil. These features are where most of the metal is deposited by subsequent rubbing with annealed Pd. There is little evidence of a thin layer being deposited on the unsanded Ni mesh or on the surface of sanded flats, though there are occasional spots where that occurred.Therefore, the deposition process is primarily scraping rather than burnishing.

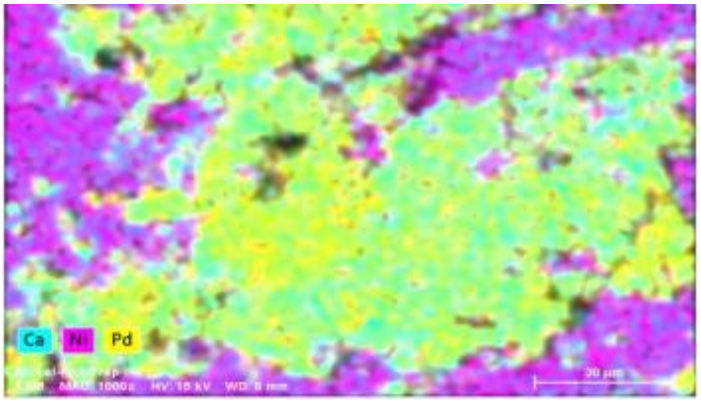


Image 9. SEM/EDS detail of the Ni foil after sanding with 100 grit and rubbing with Pd

8. Discussion of the Calcite deposits

Palladium on $CaCO_3$ is known as a Lindlar catalyst, used for hydrogenation in organic chemistry. It seems likely that the procedure as described in the Report creates catalytically active areas of this type on the mesh.

Work by Amy Kan et.al. at Rice University [6] suggests that Methanol promotes the precipitation of Calcite, and its substitution for Ethanol may therefore be promoting or increasing the appearance of the crystals observed. $CaCO_3$ is generally insoluble in alcohols, so this substitution is probably not a factor.

Analysis of local tap water [3] shows Calcium content of 50-100 mg/L. For comparison, a published analysis of municipal water in Sapporo Japan [4] where Mizuno's lab is located shows 39-44 mg/L of Calcium .

The Mizuno/Rothwell report specifies use of "tap water" for the wash and soak steps of the mesh preparation. Subsequent rubbing to apply Palladium incorporates the crystals into the intermetallic bond region where they might play an important role in the reaction.

Therefore if the formation of $CaCO_3$ crystals is a functional part of the mesh activation, use of water with low or zero Calcium ions for the mesh preparation may not yield an active material.

References:

(1) http://lenr-canr.org/acrobat/MizunoTincreasede.pdf
(2) https://www.lenr-canr.org/acrobat/MizunoTsupplement.pdf
(3) http://www.cityofsantacruz.com/home/showdocument?id=72995
(4) http://www.city.sapporo.jp/suido/overview/suishitu/result/documents/suishitsukekkah31-04.pdf
(5) See image of precipitated Calcite at https://soils.wisc.edu/facstaff/barak/temp/soil8510/images/mini_calcite_x1250.jpg
(6) https://cfpub.epa.gov/ncer_abstracts/index.cfm/fuseaction/display.files/fileID/14335 pg.18