

Procedure for the Preparation of Mandrels for Hardness Analysis

The following procedure is recommended for preparing mandrels intended for hardness measurement via metallographic indenter. This procedure is most commonly used to prepare test specimens of electrodeposited nickel and may be used as is, or adapted for other metallic deposits. Please refer to the notes and tables for specific information for your application.

Required Equipment

1. 1,000 ml tall form beaker
2. 1,000 ml sample of plating solution
3. Copper or brass rod (typical 0.375 inches diameter, 3 inches long)
4. Laboratory size rectifier and platers tape
5. Two small pieces of anode and material to bag the anode by using woven polypropylene or Whatman filter paper # 541 (185 mm diameter).
6. Hot plate and stir bar
7. Ring stand and miscellaneous clamps

Mandrel Preparation Procedure

1. Measure 1 inch from the rod end and apply platers tape with two or three wraps leaving the top end uncovered for clip connection. Using a micrometer accurately measure the starting diameter of the rod.
2. Electroclean the rod for one minute
3. Rinse well with D.I. water
4. Deoxidize with dip in 10 % sulfuric acid for 30 seconds
5. Rinse briefly with D.I. water
6. Clip the negative rectifier lead to the upper end of the copper rod and clamp to the ring stand. Attach the positive leads to the anodes and then lower the ring stand bar with the rod into the solution covering approximately half the tape.
7. Turn the rectifier and stir plate on at 0.25 A (20 ASF), for two hours for a deposit of 0.002" minimum thickness. (See notes on calculations and minimum thickness requirements)
8. Rinse well with D.I. water
9. Over plate with acid copper to protect the nickel during cross sectioning, i.e. 0.20 amps (20 AFS) for 60 minutes at room temperature in a similar plating set-up.
10. Rinse and dry the plated rod.
11. With a micrometer measure the thickness of the foil to verify the thickness 0.002 +/- 0.0005 inches for the nickel plus the copper over plate.
12. Send the plated rod for micro hardness testing.

Notes:

1. To calculate the effective surface area of one end of any rod, take
Area of the rod cylinder section = $3.14 \times \text{Diameter} \times \text{Length}$
Plus the area of the end circle $0.785 \times \text{Diameter} \times \text{Diameter}$

Or

Circumference x length plus twice the cross-sectional area
 $3.14 \times \text{Diameter} \times \text{Length} + 0.785 \times \text{Diameter} \times \text{Diameter}$

- Refer to Table 1 and 2 to calculate the time at current to produce the required minimum thickness for nickel to avoid the anvil effect when making the indenter measurement.

Table 1 - Minimum Deposit Thicknesses to Avoid Anvil Effect in Hardness Measurements with Knoop Indenter

Knoop Hardness	Minimum Thickness in Mils		
	25 gram Load	100 gram Load	200 gram Load
100	1.1	2.2	3.0
300	0.6	1.2	1.8
500	0.5	1.0	1.4
700	0.4	0.8	1.2
900	0.4	0.7	1.0
1100	0.3	0.7	0.9

Table 2 - Plating Rates for Electrodeposited Nickel
 (Based on 96.5% Cathode Efficiency)

Thick- ness in Inches	Oz. per sq. ft.	Grams per sq. ft.	Amp. Hrs. needed	Amp. Min. needed	Minutes for Obtaining Coating at Various Amperes per Sq. Ft.												
					5 a.s.f.	8 a.s.f.	10 a.s.f.	12 a.s.f.	15 a.s.f.	20 a.s.f.	25 a.s.f.	30 a.s.f.	40 a.s.f.	50 a.s.f.	75 a.s.f.	100 a.s.f.	125 a.s.f.
.00001	.0072	.204	.199	11.9	2.4	1.5	1.2	1	.8	.6	.5	.4	.3	.3	.2	.12	.1
.00002	.0144	.408	.398	23.84	4.8	3	2.4	2	1.6	1.2	1.0	.8	.6	.5	.4	.24	.2
.00003	.0216	.612	.597	35.8	7.2	4.5	3.6	3	2.4	1.8	1.5	1.2	.9	.8	.5	.36	.3
.00004	.0288	.816	.795	47.7	9.6	6	4.8	4	3.2	2.4	1.9	1.6	1.2	1.0	.7	.5	.4
.00005	.0361	1.020	.995	59.6	12.0	7.6	6.1	5.1	4.1	3.1	2.4	2.0	1.5	1.2	.8	.6	.5
.00006	.0433	1.224	1.19	71.6	14.3	9	7.2	6	4.8	3.6	2.9	2.4	1.8	1.5	1.0	.8	.6
.00007	.0504	1.428	1.39	83.5	16.7	10.5	8.4	7	5.6	4.2	3.4	2.8	2.1	1.7	1.2	.9	.7
.00008	.0578	1.632	1.59	95.6	19.2	12	9.6	8	6.4	4.8	3.9	3.2	2.4	1.9	1.3	1.0	.8
.00009	.0649	1.836	1.79	107.3	21.5	13.5	10.8	9	7.2	5.4	4.3	3.6	2.7	2.2	1.5	1.1	.9
.0001	.0721	2.04	1.99	119.2	23.9	14.9	12	10	8	6	4.8	4	3	2.4	1.6	1.2	1.0
.0002	.144	4.08	3.98	238.4	47.7	29.8	23.9	19.9	15.9	11.9	9.6	8	6	4.8	3.2	2.4	2
.0003	.216	6.12	5.97	358	72.0	44.7	35.8	29.8	23.9	17.9	14.3	12	9	7.2	4.8	3.6	2.9
.0004	.288	8.16	7.95	477	95.3	59.6	47.7	39.9	31.8	23.9	19.1	15.9	11.9	9.6	6.4	4.8	3.8
.0005	.360	10.20	9.95	596	120	74.5	59.6	49.7	40.0	30.0	23.8	20.0	15.2	12.2	8.2	6.1	4.9
.0006	.433	12.24	11.9	716	143	89.5	71.6	59.6	47.7	35.8	28.6	23.9	17.9	14.3	9.6	7.2	5.8
.0007	.504	14.28	13.9	835	167	104.4	83.5	69.5	55.6	41.8	33.4	27.8	20.9	16.7	11.2	8.4	6.7
.0008	.578	16.32	15.9	956	191.2	119.6	95.6	79.7	63.8	47.8	38.3	31.9	23.9	19.2	12.8	9.6	7.7
.0009	.649	18.36	17.9	1073	215	134.3	107.3	89.5	71.6	53.7	43	35.8	26.9	21.5	14.3	10.8	8.7
.001	.721	20.40	19.89	1192	239	149	119.2	99.4	79.5	59.6	47.7	39.8	29.8	23.9	15.9	12	9.6
.002	1.44	40.80	39.78	2384	477	298	238.4	198.3	158.7	119.0	95.3	79.5	59.6	47.7	31.8	23.9	19.1