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THERMAL NEUTRON FILTER CADMIUM ELECTROPLATE PROPERTIES

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Idaho Falls, Idaho — 83401

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ABSTRACT

The properties of thick cadmium electrodeposited on steel plate and tubing are compared for three principal baths - a standard bath, an Owen's bath, and a sulfamate bath. Some baths produced a brittle plate, e.g., the standard bath, while the Owen's and sulfamate baths produced a ductile plate that can experience a strain of about 20% without cracking. The plate was heated to 400°F without roughening or blistering from release of absorbed gas. The properties investigated were the bend and tensile, x-ray diffraction, hydrogen and impurity content, and microstructure.

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THERMAL NEUTRON FILTER CADMIUM ELECTROPLATE PROPERTIES

1. INTRODUCTION

The Sodium Loop Safety Facility (SLSF) program alters the neutron spectrum of the facility in the Engineering Test Reactor by the presence of a cadmium thermal neutron filter. The cadmium filter, Figure 1, surrounds the stainless steel in-pile tube in the core region and is retained by a stainless steel sheath. In the fabrication of a prototype thermal neutron filter (TNF) an apparent lack of ductility of the electrodeposited cadmium on the sheath resulted in formation of cracks during or after assembly from the interface fit stresses. The prototype TNF produced by Pyromet Industries incorporated a 0.040 in. thick cadmium plate on the inside diameter of a stainless steel sheath. The sheath was assembled to the secondary tube with a 0.003 to 0.006 in. diametral interference fit. The prototype TNF sheath was heated to 400°F and the tube cooled to 30°F. After cooling to room temperature, the internal stresses in the cadmium layer are apparently relieved by cracks. The cracking is undesirable in the operation of the facility because the cracks allow streaming of thermal neutrons into the experiment. If the cadmium were ductile, the cadmium layer would plastically deform rather than crack, but during machining operations the cadmium did not form a chip but powdered off during single point turning operations, and in bend samples the cadmium fractured in a brittle manner. Some possible causes or combination of causes for the lack of ductility in the thick (0.040 in.) electrodeposited cadmium are (1) hydrogen occlusion, (2) grain boundary absorption of impurities, (3) plating stresses, (4) preferred orientation, (5) formation of phases or phase transformation during or after assembly.

To overcome the cracking problem methods for producing low stress, thick cadmium deposits were investigated. The purpose of this report is to present the properties measured to characterize the plated cadmium and to evaluate the success of the plating methods in producing ductile cadmium.

2. MATERIAL AND PROCEDURE

2.1 Material

Three kinds of samples of plated cadmium were examined: (1) heavy deposits of cadmium on the inside of 6 in. stainless steel tubes (sheath), (2) small carbon steel plates approximately 2 in. square, cadmium plated on both sides, and (3) stainless steel plates 4 in. square, cadmium plated on both sides.

In the production of the cadmium plates, baths of different types were used. Three of these baths are as follows:

#1 Standard Cadmium Bath

Composition - Cadmium Metal	3oz/gal
Sodium Cyanide	15.3 oz/gal
Sodium Hydroxide	2 oz/gal
ROCO 20 XL (Brightener)	0.6%
Current density - 15 ASF	
Agitation - Magnetic stirrer	

#6 Owen's Bath⁽¹⁾

Composition - Cadmium Metal	5.2 oz/gal.
Sodium Cyanide	15 oz/gal.
Sodium hydroxide	3.5 oz/gal.
Nickel Sulfamate	1.0 gram/gal.
Triethanolamine	90 ml/gal.
Current density - 15 ASF	
Agitation - Magnetic stirrer	

#12 Sulfamate Bath

Composition - Cadmium Sulfamate	315 g/l
Ammonium Sulfamate	30 g/l
Nickel Sulfamate	1.8 g/l
Cetyltrimethyl	
Ammonium Bromide	5 g/l
Stannous Sulfate	1 g/l
Current density - 15 ASF	
Agitation - Magnetic stirrer	

2.2 Procedure

2.2.1 Tube Samples - Samples from the 6 in. tube were cut approximately 1/4 in. wide and bent to produce a fracture surface. The fracture surface was examined by scanning electron microscopy. Other samples were used for hydrogen analysis and for x-ray diffraction. Samples were from the standard cadmium bath.

2.2.2 Small Plate Samples - Duplicate samples (one set for controls, one set for annealing) from the small plates were prepared by cutting the plate into strips. Bend samples were about 1/2 in. wide and the surfaces were hand polished to produce flat specimens.

Metallography samples were prepared on planes perpendicular and parallel to the plating surface. X-ray diffraction samples were 0.4 in. by 0.8 in.. The plates were identified as follows:

- #1 Standard Bath, deposit thickness .023 in.
- #6 Owen's Bath, without periodic reverse deposit thickness .027 in.
- #10 Owen's Bath with periodic reverse, deposit thickness .037 in.
- #12 Sulfamate Bath, deposit thickness .018 in.

2.2.3 Large Plate Samples - Duplicate samples (one set for controls, one set for annealing) from the stainless steel 4 in. plates were cut about 1/2 in. wide. Tensile tests as well as bend, x-ray diffraction, and metallography were performed. The plates with .050 in. stainless steel in the center were identified as follows:

- #13 Standard Bath
- #14 Owen's Bath
- #15 Standard Bath with periodic reverse
- #16 Sulfamate Bath

The tensile specimens were hand polished to flats and shaped on a Tensil-cut machine with a 1 in. gage length. The ratio of grip-to-gage section was 2 to 1.

3. RESULTS

3.1 Bend and Tensile Tests

The three point bend tests were conducted on a self-aligning rolling-fixture as described in ASTM E-399-72 on an Instron with CCT load cells and ranges between 5 and 200 lb. The start of cracking was detected with a cathetometer sighted on the surface. The deflection at the time of the start of cracking or the start of checking for the ductile specimens was taken to determine the strain or ductility. The deflection was measured and the strain in the outer fiber in the bend test was calculated. When the cadmium deforms plastically the calculated strain will be low, since elastic conditions are assumed as follows:

The beam of thickness, h , is elastically bent to a radius, ρ . In pure bending the deflexion of the beam is approximately given⁽²⁾ by

$$y_o = \frac{L^2}{8\rho} \text{ where } L = \text{length of beam.}$$

Since the elongation in the outer fiber is $C' - C$, where C is the length of the circular segment, and

$$\frac{C'}{C} = \frac{\rho + \frac{h}{2}}{\rho} = 1 + \frac{h}{2\rho}$$

then the strain $\epsilon = \frac{d\ell}{\ell} = \frac{C' - C}{C} = \frac{C'}{C} - 1 = \frac{h}{2\rho}$ or the strain,

$$\epsilon = \frac{4h y_o}{L^2}$$

In the bend tests the crosshead loading rate was 0.002 in/min. The data is given in Table I for the as-plated (control) and the annealed specimens.

TABLE I
BEND AND TENSILE TESTS ON TNF SPECIMENS

Specimen	Bend Specimen Thickness in.	Outer Fiber Bend Strain%	Tensile Specimen Thickness in.	Tensile Strain %
#1 Control	.0076	0.5		
#1 Annealed, 550, 3 hrs	.0776	0.08		
#6 Control	.0866	1.2		
#6 Annealed, 550, 3 hrs	.0866	0.59		
#10 Control	.1022	0.61		
#10 Annealed, 550, 3 hrs	.1034	0.86		
#12 Control	.0614	2.1		
#12 Annealed	.0634	2.9		
#13 Control	.147	1.0	.130	1.7
#13 Annealed, 550°F, 3 hrs	.132	0.6	.133	3.5
#14 Control	.132	4.0	.122	~19.0
#14 Annealed, 550°F, 3 hrs	.139	6.6	.132	~23.0
#15 Standard Bath with P.R.,Control	.123	0.7	.116	0.7
#15 Standard Bath with P.R.,Annealed	.117	0.6	.120	Failed outside gage
#16 Sulfamate, Control	.098	9.0 ⁽¹⁾	.107	32.0 ⁽²⁾
#16 Sulfamate, Annealed, 550°F for 3 hrs, blistered on surface	.122	4.5	.117	12

(1) Multiple strain lines after taking out of fixture and bending further in vice.

(2) Multiple strain lines.

The tensile specimens which consisted of a sandwich of stainless steel .050 in. thick between cadmium plate were pulled on an Instron at 0.02 in. per minute crosshead speed. An extensometer at 200X magnification was used to measure the elongation at which cracking or checking occurred. This value of elongation is given in Table I. When the cadmium plate is ductile, the specimen elongates and thins down. The influence of the stainless steel on the elongation is more pronounced. The process of strain checking is progressive; hence, the higher strain values tend to be subjective.

Thus for the ductile specimens in the bend tests the measured strain is low while in the tensile tests it is high, because the plastic flow of the cadmium is influenced by the flow of the stainless steel across the metallurgical bond.

3.2 X-Ray Diffraction

X-ray diffraction was performed on polished surfaces parallel to the plating surface. The specimens, except that for the ASTM card, were examined at the Chemical Processing Plant on a Norelco X-ray diffractometer. The specimens were mounted flat on the axis of the goniometer and analyzed between two-theta angles of 30 and 80 degrees at 1 degree per minute. Specimens 1, 6, 10 and 12 were examined for variations in relative intensities after polishing, after etching, after specimen rotation of 90 degrees, and during rotation. Any differences were not considered significant for the intended analysis.

The purpose of the analysis was to get information on the preferred orientation (not a complete description as by pole figures) and the effect of annealing on the structure. The information can be obtained from a comparison of calculated diffraction line intensities with those obtained on a diffractometer. Diffracted line intensities are given by equations involving the structure factor, Lorentz-polarization factor, multiplicity factor, absorption and temperature factors. Relative line intensities are given accurately by an equation involving the structure factor, multiplicity factor, and the Bragg angle only when the crystals of the specimen have completely random orientations. Therefore, any radical disagreement between observed and calculated intensities is evidence of preferred orientation. The cadmium ASTM Card 5-0674 for powder approximates completely random crystal orientation and is used in Table II as a comparison basis for determining preferred orientation. Changes in relative intensities after annealing are used as indicators of recovery or atom movement.

TABLE II

RELATIVE X-RAY INTENSITIES OF TNF SAMPLES

hkℓ	ASTM Card 5-0674	#1		#6		#10		#12	
		Control	Annealed	Control	Annealed	Control	Annealed	Control	Annealed
002	65	100	100	100	100	100	100	100	100
101	100	26	13	52	31	43	28	85	91
103	26	nd	3	3	7	3	5	19	19
102	32	nd	1	3	5	3	3	16	15
112	17	nd	<1	2	3	nd	2	7	6
100	32	32	12	30	10	14	4	17	18
003	3	3	5	3	4	2	4	4	3
201	13	4	3	12	3	5	2	4	5
110	19	nd	<1	nd	2	<1	4	40	29
104	4	nd	nd	nd	nd	nd	nd	1	1
200	2	3	4	2	nd	nd	nd	nd	nd

hkℓ	#13 Standard Bath		#14 Owen's Bath		#15 S.B.P.R.		#16 Sulfamate		#838 AS	#848
	Control	Annealed	Control	Annealed	Control	Annealed	Control	Annealed	Plated	Cast
002	100	100	32	27	100	100	55	38	100	52
101	31	11	100	100	42	8	100	100	40	100
103	1	2	13	12	2	2	21	24	4	13
102	2	1	17	9	2	nd	21	21	4	13
112	1	nd	14	9	2	nd	16	21	2	11
100	36	24	20	16	33	33	24	28	10	23
004	5	4	2	<1	4	4	3	nd	2	1
201	6	3	10	6	11	4	11	10	2	8
110	<1	<1	11	14	nd	<1	14	13	2	9
104	nd	nd	nd	nd	nd	nd	3	3	nd	--
200	5	5	1	2	3	7	1	2	nd	--

The comparison of relative intensities is shown in Table II. There is a difference in the texture of samples #14 and #16 and the other plated samples. The texture of these samples more closely resembles that of the sample cast as a 1/2 in. diameter rod, and approximates that of ASTM card 5-0674. Upon annealing the relative intensity of the (101) line of the samples with the lower ductility i.e. samples #1, 6, 10, 13, and 15 decreases indicative of recovery or atom movement.

X-ray diffraction was also done on selected samples of the standard bath #838 heated for three hours at 350, 400, and 450°F. Recovery or atom movement did not affect the relative intensity of the (101) line at 350 or 400°F but the relative intensity increased about 30% at 450°F, while after 165 hours at 590°F it had increased to nearly the value of the cast sample. The change between 400 and 450°F was also evidenced in annealing tests on sample #14 as described in Section 3.5.

3.3 Hydrogen Analysis

Selected samples were taken for hydrogen analysis. The hydrogen analysis was done by hot gas extraction (Leeco apparatus), and by vacuum-fusion. Both methods agree within 3 ppm. Most of the results were by hot gas extraction. (Analysis performed through Anamet Laboratories by Atlas Testing Laboratory.) The results were as follows:

1. As cast sample #848 - 0.0057% by weight
2. As electrodeposited Sample #838 - 0.0055% by weight
3. Electrodeposited plus 350°F for 3 hrs. - 0.0065%
4. Electrodeposited plus 400°F for 3 hrs. - 0.0038%
5. Electrodeposited plus 450°F for 3 hrs. - 0.0068%
6. Electrodeposited plus diffusion bonded at 590°F for 165 hrs. - 0.0071%

The precision of the analysis was 3 ppm which indicates there is considerable variability in the hydrogen content in the electrodeposited cadmium. This variability may be real since a reference (3) states that cadmium hydride becomes unstable at near room temperature. Pockets of hydrogen gas may account for this variability.

3.4 Spectrographic Analysis

Three samples were analyzed by spectrographic analysis (Anamet Laboratories) that had been prepared as metallographic samples mounted in Formvar, ground and polished and etched in 1% nitric.

The samples were identified as follows:

#7-Owen's bath

#9-Owen's bath with periodic reverse. Direct 20 sec.
15 ASF. Deplete 5 sec. 25 ASF.

#12-Sulfamate bath

Sample No.	Cd %	Al %	Cu %	Fe %	Pb %	Ni %	Sn %
7	Bal	Nil	0.0004	0.003	0.0002	0.0004	0.0002
9	Bal	Nil	0.005	0.001	0.0002	Nil	0.0001
12	99.98	.0003	0.0005	0.008	0.0004	0.0006	0.0003

3.5 Visual Appearance and Weight Loss

The annealing specimens were encapsulated in an inert atmosphere glove box with <3 ppm oxygen plus water vapor. After annealing for three hours at 550°F as measured with a TC on the stainless can, the specimens had a bright appearance. The weight loss as measured to the nearest milligram was nil on the 5 to 9 gram bend samples.

Samples which blistered during the anneal were #10, 12, and 16, while #14 had a roughened appearance from the extrusion of grains and some gas release. The appearance is shown in photographs of selected samples in Figures 2 through 4. Figure 2, A and B, shows both sides of samples 1, 6, 10, and 12. In Figure 3A can be seen a blister which cracked along with heat etching of the surface of sample #10. Figure 3B shows blisters in the cadmium of sample #12. Figure 4, shows the roughened surface of sample #14 as a function of annealing temperature. The specimens were held at 400, 440, 480, and 510°F for three hours using the same encapsulation procedure as previously. The results indicate that at 400°F heat etching starts; at 440°F heat etching is more pronounced and grain extrusion starts; at 480°F grain extrusion is more pronounced and microtearing at the grain boundaries starts; and at 510°F extrusion is prominent and microtearing is evident.

3.6 Metallography

Metallography was done on sections of the as-plated (control) and annealed specimens on planes perpendicular and parallel to the plating surface to determine the microstructure of the samples from various baths. For the Standard Bath and the less ductile cadmium plate, the grain size is finer near the steel plating surface and

the grains become larger as the thickness increases, Figures 5 through 24 and Figures 29 through 32. The grains are columnar in the perpendicular plane and equiaxed in the plane parallel to the plating surface. For the ductile cadmium plate samples #14 and #16, Figures 25 through 28 and Figures 33 through 36, the columnar grains in the perpendicular plane are broken up and smeared out, although they tend to get larger away from the steel plating surface.

The annealing does have a slight effect on the microstructure. In some there is a refinement of grains and decrease in boundary width while in other samples there is grain growth. Although care has to be exercised in the interpretation of grain size effects it is apparent that in general the samples with the lower ductility undergo refinement of the grains upon annealing for three hours at 550°F, while the samples (#14 and #16) with the higher ductility undergo grain growth.

The polished specimens were etched with 5% ferric chloride and 2% HCl in ethyl alcohol by swabbing.

3.7 Scanning Electron Microscopy (SEM)

Fracture surfaces were examined by scanning electron microscopy to characterize the mode of failure, identify defects, and possible hydrogen entrapment sites. Selected fractographs are shown in Figures 37 through 41 from sample #876 Heat 28, the Standard Bath, Sample #14, Owen's Bath, and Sample #16, Sulfamate Bath. The fracture surfaces were prepared by bending the cadmium sandwich sample until the cadmium cracked. In the case of the brittle standard bath cadmium, Figure 37, and the annealed cadmium, Figures 39 and 41, when the crack reached the stainless the cadmium would tear away from the stainless leaving flakes attached to the stainless steel. The ductile as-plated cadmium had to be pried away from the stainless.

The fracture surface of the brittle cadmium, Figure 37, has a columnar structure composed of deposited filaments connected by low energy boundaries, pores, or voids. Low energy tearing of the filaments and spallation occurs as the crack progresses.

The fracture surface of the Owen's Bath sample #14, Figure 38, has a tangled structure with ductile tearing and dimpling. This same structure is also seen after annealing, Figure 39.

The fracture surface of the Sulfamate Bath, Sample #16, Figure 40, has a porous tangled structure with ductile tearing and dimpling. After annealing the fracture surface is more porous and gas pockets appear to be present with ductile tearing and dimpling, Figure 41.

4. DISCUSSION

The bend and tensile data indicate that ductile cadmium plate 30 to 40 mils thick on stainless steel can be produced by depositing from the Owen's or Sulfamate baths. The Sulfamate bath cadmium will contain entrapped gas and impurities which upon annealing at 550°F for three hours will cause the unconfined cadmium to blister or produce microcracks. The Owen's Bath cadmium upon annealing at 550°F for three hours undergoes grain extrusion and microtearing at grain boundaries due to gas evolution, but the ductility does not decrease as with the Sulfamate Bath. The grain extrusion is evidently due to anisotropic expansion in the lattice directions. If the annealing temperature is reduced to less than 440°F roughening of the surface or microtearing from gas evolution is not evident in the Owen's Bath cadmium.

The x-ray diffraction, metallography, and fractography were consistent in indicating that the plated cadmium in the Owen's and Sulfamate baths had a more random texture than in the Standard bath samples. It should be noted that the grain size and orientation appears to change with thickness of the cadmium plate, hence a thickness effect may change the properties measured as the plate thickness changes.

5. CONCLUSIONS

1. Plated cadmium 30 to 40 mils thick can be produced which can experience a strain of about 20% without cracking. The cadmium can be heated to 400°F without roughening or blistering.

2. X-ray diffraction indicates that texture of the ductile cadmium plate is similar to that of ASTM Card 5-0674 for cadmium.

3. Hydrogen analysis of cadmium deposited from the Standard bath gave 55 ppm which was not released by heating at 450°F for three hours.

4. Cadmium can be heated in argon with less than 3 ppm oxygen plus water vapor to 550°F for three hours without appreciable loss in weight or discoloration.

5. Grain growth or grain refinement upon heating to 550°F appears to depend on the texture and presence of plating stresses.

6. Fracture of the Standard bath cadmium occurs by low energy tearing at the filament boundaries assisted by the texture, while fracture of the Owen's or Sulfamate bath cadmium occurs by ductile tearing and dimpling of the grains.

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FIGURE 1 -

THERMAL-NEUTRON FILTER DIMENSIONS

Figure 2A - General appearance of side A of annealed samples #1, 6, 10, and 12 (shortest to longest, respectively).

Figure 2B - General appearance of side B of annealed samples.

Figure 3A - Region around blister of sample #10
side A.

Figure 3B - Region around blisters of sample #12
Side B.

Figure 4A-Surface of annealed samples after 3 hrs.
at 400 and 510°F. Sample #14.

Figure 4B - Surface of annealed samples after 3 hrs
at 440 and 480°F. Sample #14.

100X

Figure 5 - Columnar grain structure in as-received sample #1, perpendicular to plating surface.

100X

Figure 6 - Columnar grain structure in annealed sample #1 note slight refinement of grains at outside surface.

Polarized light

250X

Figure 7 - Equiaxed grain structure on plane parallel to plating surface, sample #1, as-received.

Polarized light

250X

Figure 8 - Sample #1, annealed, apparent refinement of grains may be partially a thickness effect on planes parallel to plating direction.

100X

Figure 9 - Sample #6, as-received, columnar grain structure in perpendicular plane.

100X

Figure 10 - Sample #6, annealed, very subtle changes if any due to annealing.

250X

Figure 11 - Sample #6, as-received, plane parallel to plated surfaces.

250X

Figure 12 - Sample #6, annealed.

100X

Figure 13 - Sample #10 with periodic reverse (P.R.). This plane appears to have some equiaxed grains not present in Figure 9 or Figure 14.

100X

Figure 14 - Sample #10, annealed. Note the apparent decrease in the columnar grain boundary width when compared with Figure 13.

250X

Figure 15 - Sample #10, as-received, some voids at the columnar grain boundaries visible.

250X

Figure 16 - Sample #10, annealed, some apparent refinement of the grains.

100X

Figure 17 - Sample #12, as-received, finer columnar grains due to thickness effect.

100X

Figure 18 - Sample #12, annealed, grain boundary effect.

Polarized light

250X

Figure 19 - Sample #12, as-received.

Bright Field

250X

Figure 20 - Sample #12, annealed. Grain boundary effects.

100X

Figure 21 - Sample #13, control, plane perpendicular to plating surface, composite photograph.

100X

Figure 22 - Sample #13, annealed, plane perpendicular to plating surface, some refinement of grains composite photograph.

100X

Figure 23 - Sample #13, control, plane parallel with plating surface.

100X

Figure 24 - Sample #13, annealed, plane parallel with plating surface, refinement of grains.

100X

Figure 25 - Sample #14, control, plane perpendicular to plating surface, composite photograph.

100X

Figure 26 - Sample #14, annealed, plane perpendicular to plating surface, composite photograph.

100X

Figure 27 - Sample #14, control, plane parallel with plating surface.

100X

Figure 28 - Sample #14, annealed, plane parallel with plating surface, grain growth.

100X

Figure 29 - Sample #15, standard bath periodic reverse, (S.B.P.R.) control, plane perpendicular to plating surface. Composite photograph.

100X

Figure 30 - Sample #15 S.B.P.R., annealed, plane perpendicular to plating surface, composite photograph.

100X

Figure 31 - Sample #15 S.B.P.R., control, plane parallel to plating surface.

100X

Figure 32 - Sample #15, S.B.P.R., annealed, plane parallel to plating surface some grain refinement.

100X

Figure 33 - Sample #16, sulfamate, control, plane perpendicular to plating surface.

100X

Figure 34 - Sample #16, sulfamate, annealed, plane perpendicular to plating surface. Grain growth.

100X

Figure 35 - Sample #16, sulfamate, control, plane parallel to plating surface.

100X

Figure 36 - Sample #16, annealed, plane parallel to plating surface. Grain growth.

1000X

Figure 37 - Sample #876, assembled at 350°F. Fracture surface perpendicular to plating surface showing typical filament structure.

1000X

Figure 38 - Sample #14, as-plated. Fracture surface perpendicular to plating surface showing ductile tearing and dimpling.

1000X

Figure 39 - Sample #14, annealed at 550°F. Fracture surface perpendicular to plating surface showing ductile tearing.

1000X

Figure 40 - Sample #16, as-plated. Fracture surface perpendicular to plating surface showing ductile tearing and a more porous structure than in Sample #14.

1000X

Figure 41 - Sample #16, annealed at 550°F. Fracture surface perpendicular to plating surface showing porous structure with ductile tearing and presence of gas pockets.