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COMPATIBILITY OF UNSUPPORTED PYROLYTIC-CARBON COATED URANIUM DICARBIDE PARTICLES WITH WATER VAPOR

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OAK RIDGE NATIONAL LABORATORY
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APRIL 1964

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COMPATIBILITY OF UNSUPPORTED PYROLYTIC-CARBON COATED URANIUM DICARBIDE PARTICLES WITH WATER VAPOR

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ABSTRACT

Five batches of pyrolytic-carbon coated uranium carbide particles were exposed to partial pressures of water vapor ranging from 20 to 635 mm Hg at temperatures of 700 to 1100°C. All batches of coated particles were prepared by Minnesota Mining and Manufacturing Company, had laminar type of coating, and contained cores consisting principally of uranium dicarbide. The rate of reaction of the pyrolytic-carbon coatings with water vapor was determined from weight losses. The extent of failure of the coatings during exposure was established by microscopic examination and leaching of the particles with 8M HNO_3. Differences in the protectiveness of coatings present on the various batches of particles are attributable, at least in part, to two types of attack by water vapor, a localized or pitting type and a more general type. Rapid failure of the coatings is indicated at 1100°C. The coatings on some batches of particles remained protective at temperatures of 800 to 1000°C.

I. INTRODUCTION

The feasibility of producing practical fuel elements containing particles of UC_2 coated with pyrolytically deposited carbon has been enhanced by improvements in the vapor deposition techniques. The availability of such fuel elements will assist in the development of gas-cooled reactors having cores which contain only ceramic material of high-temperature stability. Anticipated advantages arising from the application of such materials include high coolant temperatures, high burnups, and retention of fission products. Oxidizing gases, however, would react with the pyrolytic-carbon coating of the fuel particle as well as with the graphite or any other form of carbonaceous material used to support the coated particles in the fuel body. The inleakage of steam following a steam generator failure is of particular concern, since a rapid reaction with the hot fuel body would occur. This could lead to a mechanical failure of the fuel body and/or a failure of the pyrolytic-carbon coatings on the fuel particles. The latter would result in a release of fission products into the main coolant stream. The H_2 and CO produced by the steam-carbon reaction would entail an additional hazard.

The reactivity of unsupported pyrolytic-carbon coated fuel particles with water vapor has been studied experimentally as a part of the fuel element evaluation program of the Oak Ridge National Laboratory. The rates of reaction of the coatings with water vapor have been determined at various temperatures and partial pressures of water vapor for a number of batches of coated particles. The integrity of the coatings, following exposure to water vapor, has been evaluated by means of a HNO_3 leach and microscopic examination.

II. EXPERIMENTAL

A. Materials

The five batches of coated particles studied were supplied by the Minnesota Mining and Manufacturing Company. All batches had laminar types of pyrolytic-carbon coatings surrounding cores containing primarily UC_2. The conditions used for deposition of the pyrolytic-carbon coatings were not described by the manufacturer. General information related to some of the properties of the various batches of coated particles are listed in Table 1 and macrographs and photomicrographs of the material as received are shown in Figs. 1 to 5. All batches

Table 1. General Information on Pyrolytic-Carbon Coated Particles[a]

Batch No.	Uranium Content (%)	Average Core Particle Size (μ)	Average Coating Thickness (μ)	Density by Helium Pycnometer (g/cm^3)	Surface Area[b] (m^2/g)	Surface Contamination[c] (%)	Uranium Removed in HNO_3 Leach[d] (%)	After Leaching Surface Contamination[c] (%)
					x 10^{-3}	x 10^{-3}	x 10^{-3}	x 10^{-3}
3M-D	67.0	477	119	5.34	36	31	25	12
3M-F	73.0	265	53	5.47	e	3	240	1.6
3M-113	26.4	196	137	3.04	79	0.02	0.6	0.01
3M-114	50.0	188	84	3.20	120	0.05	0.1	<0.05
3M-117	44.1	176	99	2.51	98	<1.8	0.09	0.6

[a] All lots had UC_2 cores and laminar-type of coating.

[b] BET with krypton.

[c] Determined by alpha counting; values based on total U in particles.

[d] Leached in 8M HNO_3 at 95°C for 8 hr; values based on total U in particles.

[e] Not determined.

of the coated particles were leached with 8M HNO_3 at ~ 90°C for ~ 8 hr prior to exposure to water vapor. Experimental studies of some of the batches of coated particles were quite incomplete due to the limited quantity of coated particles available.

B. Procedures

A silica tube (~ 10 mm inner dia) containing a fritted Vycor disk was used to support the coated particles in the studies with batches 3M-D and 3M-F. A helium-H_2O mixture having a partial pressure of H_2O of 20 mm Hg was passed downward through the bed of coated particles at a flow rate of ~ 100 cm^3/min (linear flow velocity of ~ 2.5 cm/sec). Samples from the other batches of coated particles were supported in a nickel tube (~ 10 mm inner dia) containing a drilled nickel plate and platinum gauze. Again the helium-H_2O mixtures were passed downward through the bed of coated particles. The desired partial pressures of H_2O were obtained by saturating helium with H_2O at various controlled temperatures. The flow rate of the helium-H_2O mixtures ranged from ~ 100 to 300 cm^3/min which corresponds to linear velocities of ~ 2.5 to 7.5 cm/sec. A weighed sample (~ 2 g) of coated particles was exposed at the desired temperature and partial pressure of H_2O. Following the exposure, the sample was reweighed and leached with 8M HNO_3 at ~ 80°C for ~ 8 hr. The leach solution was analyzed for uranium by the Analytical Service Group. The extent of failure of the coatings was determined from the uranium content of the leach solution and the quantity of uranium present in the original sample. The average reaction rate of the pyrolytic-carbon coatings was determined from the weight change and reported values are based on the original weight of pyrolytic-carbon present on the particular batch of coated particles used. Microscopic examinations of the coated particles were made after the exposure to H_2O and a HNO_3 leach.

III. RESULTS AND DISCUSSION

A. Reactivity of 3M-D and 3M-F Coated Particles

The results of the experiments using coated particles from batches 3M-D and 3M-F are given in Table 2. These results show that the loss in weight of the coated particles was greater in all cases for batch 3M-D than for batch 3M-F. On the other hand, after the test exposures more uranium was leached from 3M-F particles than from 3M-D particles. These data indicate that the extent of the damage, which may be associated with a different mode of attack, actually was greater for batch 3M-F than batch 3M-D. Photomicrographs shown in Figs. 6 and 7 reveal only a shallow attack on the outer regions of the coatings. This attack appears to be less severe for batch 3M-F than for batch 3M-D in agreement with the weight-loss data. Figures 2b and 7a show that there were particles present in batch 3M-F with much thinner than average coating thicknesses. Figure 7a also shows that cracking of the laminar coating occurred for a number of the 3M-F particles. These observations could account for the higher incidence of failure found for batch 3M-F than found for batch 3M-D.

B. Reactivity of 3M-113 and 3M-114 Coated Particles

Data obtained for coated particles from batches 3M-113 and 3M-114 are given in Table 3. The results show, as anticipated, that the reaction rates increased with increasing temperature and partial pressure of H_2O. Data in Fig. 8 show that the apparent order of the reaction with respect to H_2O is $\sim$ 0.7 for 3M-114 particles at 1000°C. This value is in agreement with that found by Pilcher et al[1] over about the same pressure range at 1100°C. Although, the data are not as

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[1] J. M. Pilcher, P. L. Walker, Jr., and C. C. Wright, Ind. Eng. Chem. 47, 1712 (1955).

Table 2. Reactivity of Coated Particles from Batches 3M-D and 3M-F with Helium Containing H_2O at a Partial Pressure of 20 mm Hg (Total Pressure, 1 atm) at 900°C

Sample No.	Weight of Sample (g)	Time (hr)	Weight Loss (g)	Amount of Coating Removed (%)	Rate of Coating Removal (g/g-hr)	Uranium Removed by HNO_3 Leach (g)	
						(g)	(% of Total U)
3M-D-3	2.01	8	0.018	3.5	0.004	0.0005	0.04
-4	1.66	24	0.040	9.4	0.004	0.001	0.09
-2	1.85	50	0.070	14	0.003	0.008	0.6
-1	1.79	100	0.070	15	0.002	0.009	0.8
3M-F-3	1.61	8	0.002	0.8	0.001	0.001	0.08
-4	1.96	24	0.005	1.3	0.0006	0.004	0.3
-2	1.56	50	0.008	2.7	0.0005	0.030	2.5
-1	2.05	100	0.021	5.3	0.0005	0.027	1.8

Table 3. Reactivity of Coated Particles From Batches 3M-113 and 3M-114 With Helium Containing H_2O at Partial Pressures of 55-355 mm Hg (Total Pressure, 1 atm) in the Temperature Range 700 to 1000°C

Sample No.	Weight of Sample (g)	Time (hr)	Temp. (°C)	Partial Pressure of H_2O (mm Hg)	Weight Loss (g)	Amount of Coating Removed (%)	Rate of Coating Removal (g/g-hr)	Uranium Removed by HNO_3 Leach	
								(g)	(% of Total U)
3M-113-6	2.16	6	700	355	0.0025	0.2	0.0003	0.0001	0.02
-5	2.18	4	800	355	0.041	2.6	0.006	0.0232	4.0
-3	2.47	6	800	355	0.108	6	0.010	0.0021	0.3
-4	2.56	7	800	355	0.203	11	0.016	0.147	22
-2	2.51	6	1000	355	0.450	25	0.042	0.654	97
3M-114-4	2.50	6	800	233	0.036	3.2	0.005	0.0031	0.3
-1	2.45	6	800	355	0.036	3.3	0.006	0.0107	0.9
-9	1.99	6	900	233	0.127	14	0.024	0.001	0.1
-10	1.95	6	900	233	0.121	13	0.023	0.001	0.1
-3	2.41	4	900	355	0.159	15	0.037	0.002	0.2
-2	2.56	6	900	355	0.211	18	0.030	0.0177	1.4
-8	2.01	4	1000	55	0.099	11	0.027	0.001	0.1
-7	2.03	4	1000	92	0.149	16	0.041	0.002	0.2
-6	2.02	4	1000	149	0.193	21	0.053	0.001	0.1
-5	2.43	4	1000	355	0.424	39	0.097	0.016	1.3

precise as desired, a plot of the reaction rates obtained for 3M-114 particles at 800 to 1000°C and a partial pressure of H_2O of 355 mm Hg gave a reasonably straight line with a slope corresponding to an activation energy of ~ 38 kcal/mole. The reaction rate found at 1000°C and a partial pressure of H_2O of 149 mm Hg is in agreement with the value obtained by extrapolating Pilcher et al[1] data from 1100 to 1000°C at a comparable steam pressure. The single run made at 700°C showed that the reaction rate was extremely low at this temperature suggesting that prolonged exposure would be required to cause damage to the coatings at temperatures of 700°C or below.

The data in Table 3 indicate that batch 3M-113 particles were more susceptible to damage than batch 3M-114 particles. For example, in runs 3M-113-4 and 3M-114-6 the quantities of coating removed are comparable, but much more uranium was leached from 3M-113-4. The coatings on batch 3M-114 apparently were more protective after serious attack than were the coatings on batch 3M-113. Metallographic examination of a number of samples of these particles provides some explanation of this difference in behavior. In Fig. 9a the mode of attack on batch 3M-113 particles is shown to be quite localized. This same type of attack at a more advanced stage is shown in Fig. 9b, where pits are seen in many of the coatings. This type of attack would result in exposure of the uranium carbide core without extensive removal of the coating. It is to be noted that a number of the particles show signs of only a superficial attack.

Examination of batch 3M-114 coated particles revealed a different mode of attack, as illustrated in Figs. 10 and 11. This series of photomicrographs shows the results of exposing these particles to increasingly severe conditions. Figure 10a shows the superficial attack on the outer layer of the coating after 6 hr at 800°C, during which 3% of the coating was removed. The particles shown in Fig. 10b suffered a 14% loss of the coating after 6 hr at 900°C and show a significant attack of several particles, including separation of the outer layer. The effect was accentuated as the degree of attack increased, as seen in Fig. 11a after 4 hr at 1000°C, during which 21% of the coating was removed. All coatings were damaged and peeling of the outer layer was very evident, as illustrated in Fig. 11b after an exposure of 4 hr at 1000°C at a higher partial pressure of H_2O. These latter conditions resulted in removal of 39% of the coating, but the remainder still afforded considerable protection to the cores. In several cases most of the coating was removed but no path was opened to the core. Generalized attack on coated particles from batch 3M-114 probably accounts for the protection afforded these particles even after substantial amounts of the coating were removed.

C. Reactivity of 3M-117 Coated Particles

Data obtained for coated particles from batch 3M-117 are listed in Table 4. The reaction rates found at 900 and 1000°C and a partial pressure of H_2O of 355 mm Hg are in good agreement with those measured for 3M-114 particles under comparable conditions (Table 3). The values found at 1000°C and a partial pressure of H_2O of 635 mm Hg are in reasonable agreement with values found by other investigators[2,3] using steam at atmospheric pressure. The data given in Tables 3 and 4 suggest that the reaction rate increases with degree of burnoff of carbon although there is sufficient scatter in the data to preclude a forthright statement to this effect.

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[2] M. A. Mayers, J. Am. Chem. Soc. 56, 1879 (1934).

[3] W. T. Abel and J. H. Holden, "The Suspended Specimen Method for Determining the Rate of Steam-Carbon Reaction," U. S. Bur. Mines Rept. Invest. No. 6000 (1962).

Table 4. Reactivity of Coated Particles From Batch 3M-117 with Helium Containing H_2O at Partial Pressures of 355-635 mm Hg (Total Pressure, 1 atm) in the Temperature Range 800-1100°C

Sample No.	Weight of Sample (g)	Time (hr)	Temp. (°C)	Partial Pressure of H_2O (mm Hg)	Weight Loss (g)	Amount of Coating Removed (%)	Rate of Coating Removal (g/g-hr)	Uranium Removed by HNO_3 Leach (g)	Uranium Removed by HNO_3 Leach (% of Total U)
3M-117-20	2.07	4	800	355	0.069	6.4	0.016	0.004	0.4
-16	2.10	1	800	635	a	a	a	0.001	0.1
-23	1.98	4	800	635	0.163	15.9	0.040	0.011	1.3
-5	2.06	1	900	355	0.030	2.8	0.028	0.027	3.0
-6	2.10	2	900	355	0.075	6.9	0.035	0.057	6.3
-22	2.06	4	900	355	0.115	10.9	0.027	0.032	3.5
-13	1.83	4	900	355	0.074	7.9	0.020	a	a
-17	2.03	4	900	355	0.142	13.5	0.034	0.053	5.9
-21	2.01	4.5	900	355	0.117	11.3	0.025	0.024	2.7
-8	2.13	1	900	635	0.078	7.1	0.071	0.006	0.6
-24	1.61	1	900	635	0.052	6.2	0.062	0.030	4.1
-7	2.01	2	900	635	0.172	16.6	0.083	0.037	4.1
-3	2.56	1	1000	355	0.084	6.4	0.064	0.050	4.4
-2	1.98	2	1000	355	0.186	18.3	0.092	0.122	14
-14	2.07	2	1000	355	0.179	16.8	0.084	a	a
-19	2.05	2	1000	355	0.133	12.5	0.063	0.031	3.4
-12	2.01	4	1000	355	0.417	40	0.10	0.061	6.8
-1	2.34	4	1000	355	0.521	43	0.11	0.046	4.5
-10	1.92	1	1000	635	0.114	11.5	0.12	0.036	4.1
-25	1.78	1	1100	635	0.421	46	0.46	0.32	40

[a] Not determined.

Surface area measurements were made on several samples after exposure to H_2O. The limited data indicate that the surface area, after ~ 10% burnoff of carbon, was some 20 times greater than that of the original 3M-117 particles. No measurements were made at very low burnoffs where the reaction rate would be expected to change most rapidly.

The coatings on 3M-117 particles appear to be somewhat less protective than those on 3M-114 particles, but afford much better protection than was observed for 3M-113 particles. Photomicrographs shown in Fig. 12 indicate that the mode of attack was predominantly a cracking and peeling type. There is some evidence, however, that a limited amount of the pitting type of attack also occurred.

The single experiment run at 1100°C resulted in a large removal of carbon and a high incidence of failure of the coatings. Figure 13a shows a photomicrograph of the 3M-117 particles after exposure to steam. The residual uranium oxide from the cores is evident. Figure 13b shows the same material after leaching with 8M HNO_3. The coatings show the effect of the drastic exposure conditions. Peeling is very evident and some radial cracking is visible. The latter may be primarily an effect of the temperature. If so, this could account for the larger fraction of failures found at 1100°C than at 1000°C at comparable degrees of coating burnoff.

CONCLUSIONS

The coatings present on the various batches of particles reacted with water vapor at similar rates at comparable temperatures and partial pressures of water vapor. These reaction rates also are comparable to those given by some graphites.

The degree of protectiveness of the coatings at comparable degrees of burnoff varied considerably in the case of batches 3M-113, 3M-114 and 3M-117. The pitting type of attack that occurred in the coatings of 3M-113 particles probably was responsible for their high rate of failure.

Rapid failure of the laminar type of coatings is indicated at 1100°C; most coatings probably would be protective at 800°C. Testing of the coatings of each batch at 900 to 1000°C would be required to establish the degree of protectiveness afforded by the coatings in this temperature range.

ACKNOWLEDGMENTS

The authors wish to thank E. S. Bomar and J. L. Cook of the Metals and Ceramics Division for their assistance in providing the various batches of coated particles and information concerning their properties. The metallographic examinations performed by the Metallography Group of the Metals and Ceramics Division are gratefully acknowledged.

FIGURE CAPTIONS

Fig. 1. Coated Particles from Batch 3M-D. As received. a. Unmounted. Oblique illumination. 30X. b. As polished. 100X.

Fig. 2. Coated Particles from Batch 3M-F. As received. a. Unmounted. Oblique illumination. 30X. b. As polished. Note range of coating thicknesses. 100X.

Fig. 3. Coated Particles from Batch 3M-113. As received. a. Unmounted. Oblique illumination. 33X. b. As polished. 100X.

Fig. 4. Coated Particles from Batch 3M-114. As received. a. Unmounted. Oblique illumination. 33X. Note polygonal shape. b. As polished. 100X.

Fig. 5. Coated Particles from Batch 3M-117. As received. a. Unmounted. Oblique illumination. 33X. b. As polished. 100X.

Fig. 6. Coated Particles from Batch 3M-D After 100-hr Exposure at 900°C to Helium Containing H_2O at a Partial Pressure of 20 mm Hg. a. As polished. 100X. b. Etched. 200X.

Fig. 7. Coated Particles from Batch 3M-F After 100-hr Exposure at 900°C to Helium Containing H_2O at a Partial Pressure of 20 mm Hg. a. As polished. 100X. Note cracking in the laminar coating. b. Etched. 500X.

Fig. 8. Apparent Order of Reaction of Coated Particles from Batch 3M-114 with Steam at 1000°C.

Fig. 9. Coated Particles from Batch 3M-113 Exposed at 800°C to Helium Containing H_2O at a Partial Pressure of 355 mg Hg. As polished. 100X. a. Exposed for 4 hr. b. Exposed for 7 hr.

Fig. 10. Coated Particles from Batch 3M-114 After 6-hr Exposure to Helium Containing H_2O at a Partial Pressure of 233 mm Hg. As polished. 100X. a. Exposed at 800°C. b. Exposed at 900°C.

Fig. 11. Coated Particles from Batch 3M-114 After 4-hr Exposure at 1000°C to Helium Containing H_2O. As polished. 100X. a. Partial pressure of H_2O of 149 mm Hg. b. Partial pressure of H_2O of 355 mm Hg.

Fig. 12. Coated Particles from Batch 3M-117 After 4-hr Exposure to Helium Containing H_2O at a Partial Pressure of 355 mm Hg. As polished. 100X. a. Exposed at 800°C. b. Exposed at 1000°C.

Fig. 13. Coated Particles from Batch 3M-117 After 1-hr Exposure at 1100°C to Helium Containing H_2O at a Partial Pressure of 635 mm Hg. As polished. 100X. a. Before HNO_3 leach. b. After HNO_3 leach.

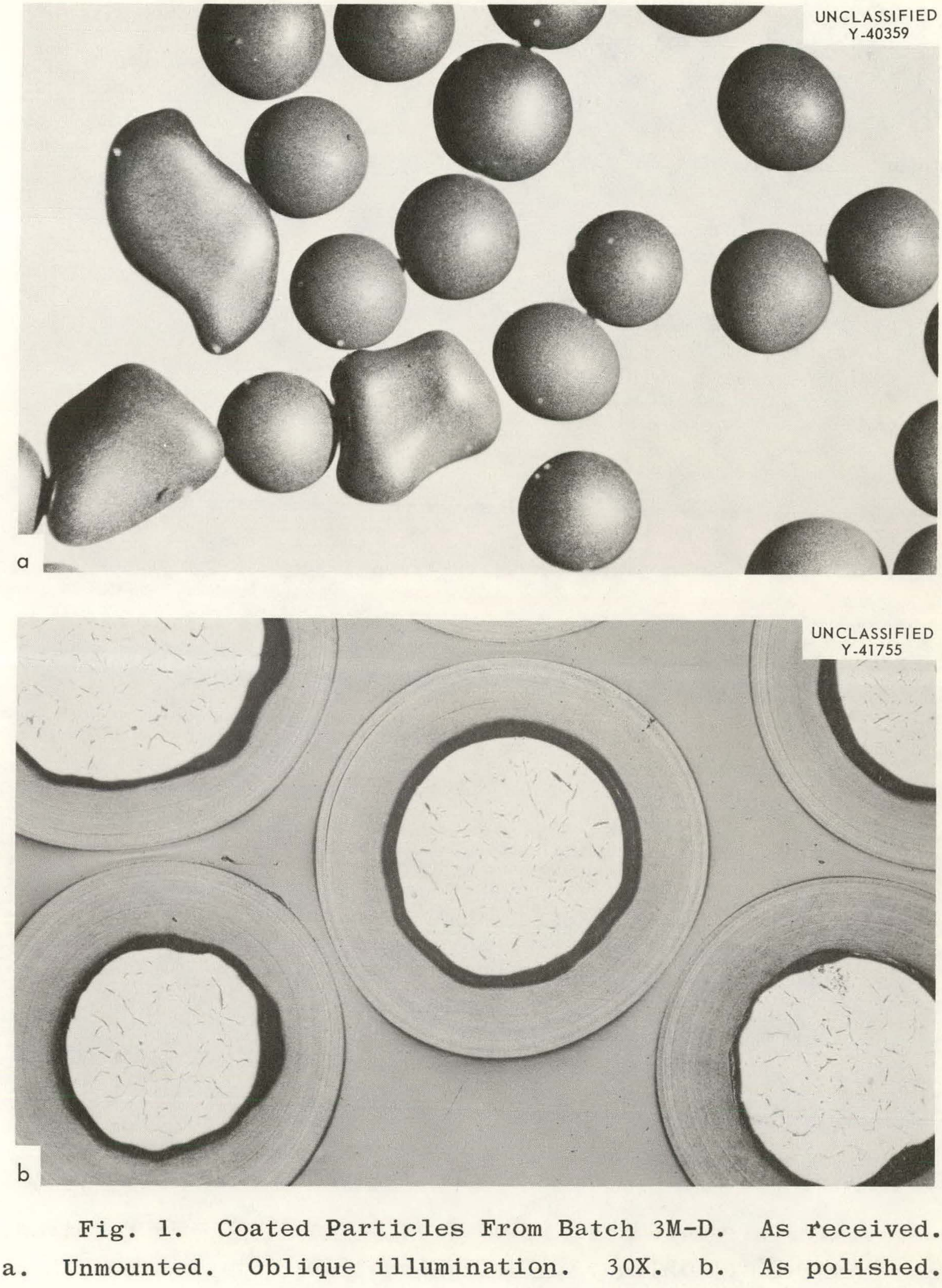


Fig. 1. Coated Particles From Batch 3M-D. As received. a. Unmounted. Oblique illumination. 30X. b. As polished. 100X.

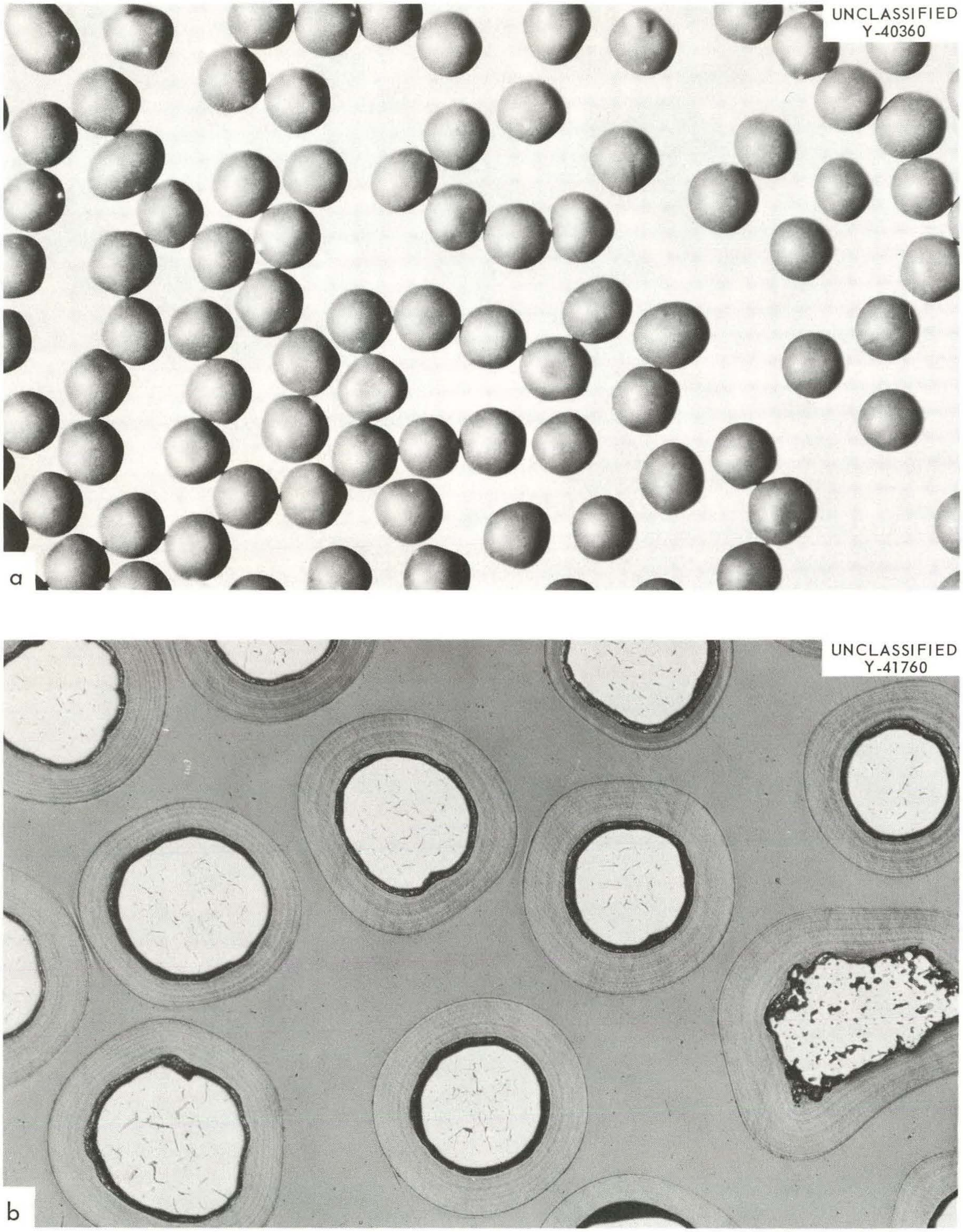
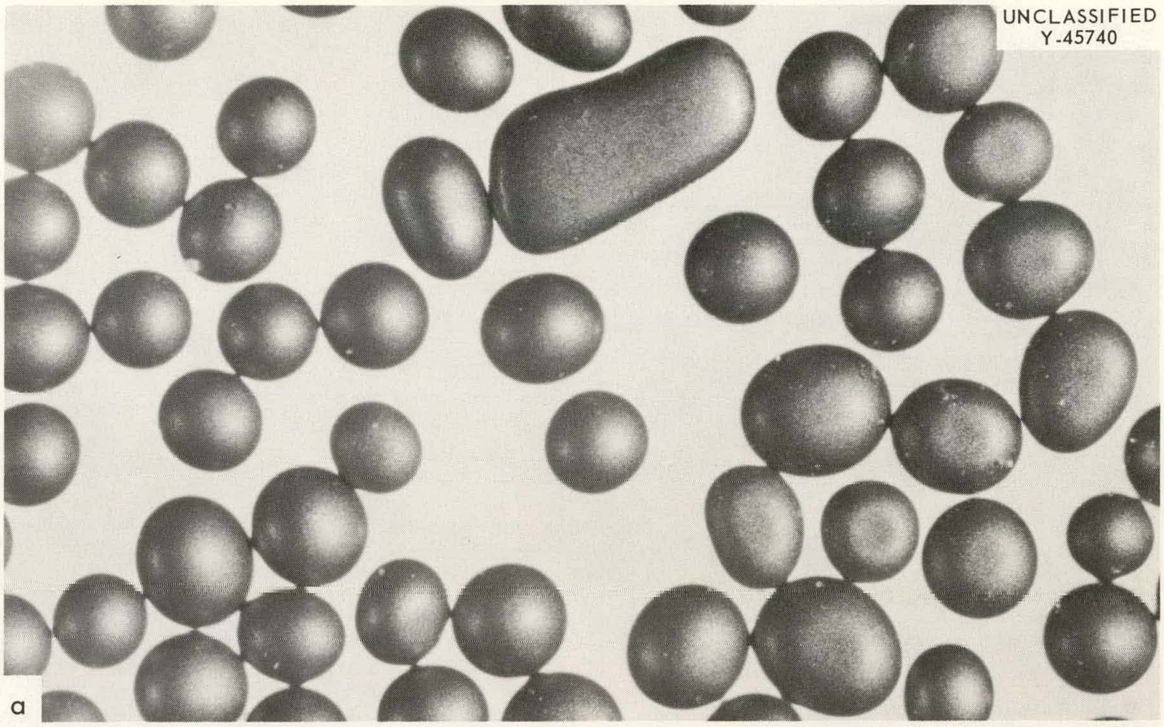


Fig. 2. Coated Particles from Batch 3M-F. As received. a. Unmounted. Oblique illumination. 30X. b. As polished. Note range of coating thicknesses. 100X.



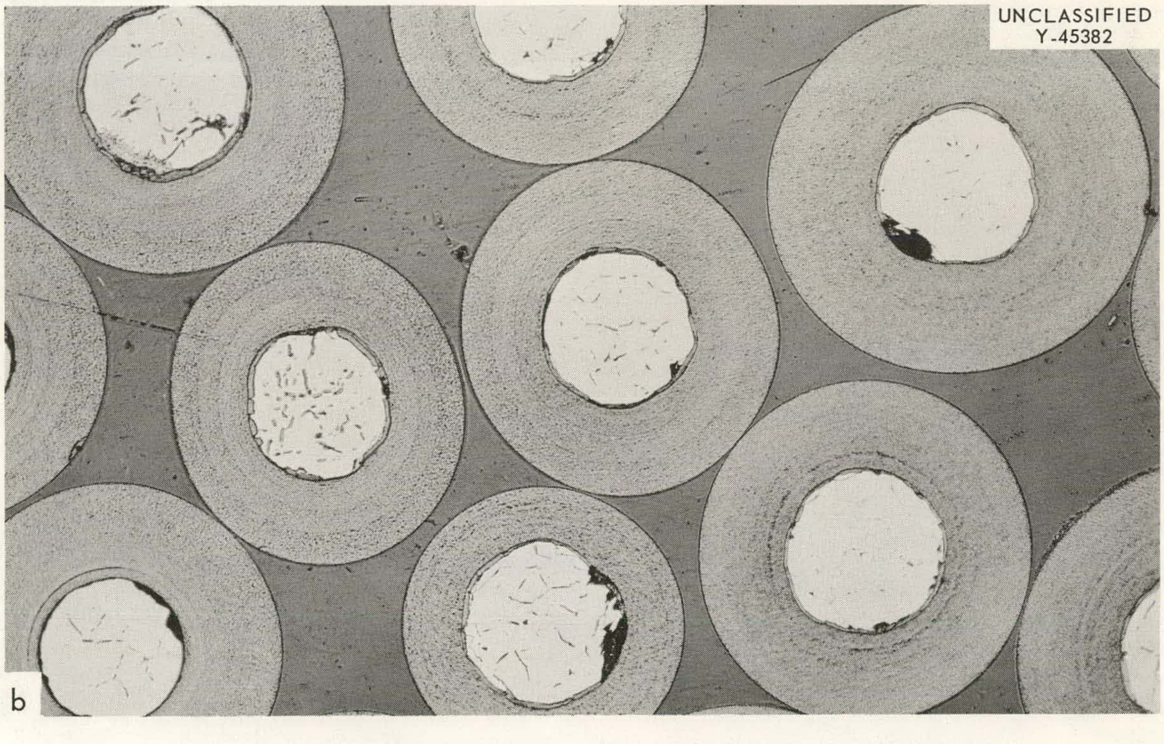


Fig. 3. Coated Particles from Batch 3M-113. As received. a. Unmounted. Oblique illumination. 33X. b. As polished. 100X.

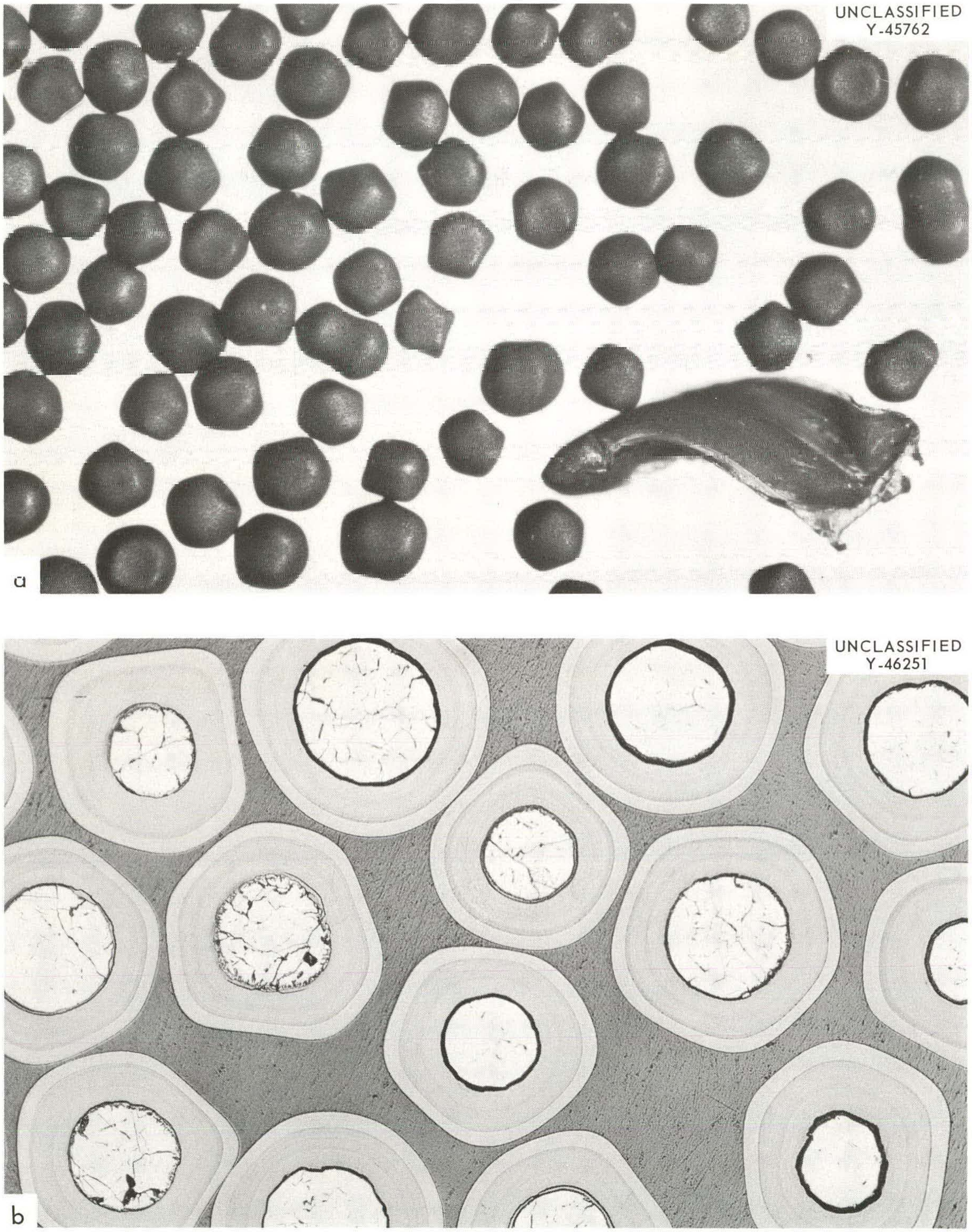


Fig. 4. Coated Particles from Batch 3M-114. As received. a. Unmounted. Oblique illumination. 33X. Note polygonal shape. b. As polished. 100X.

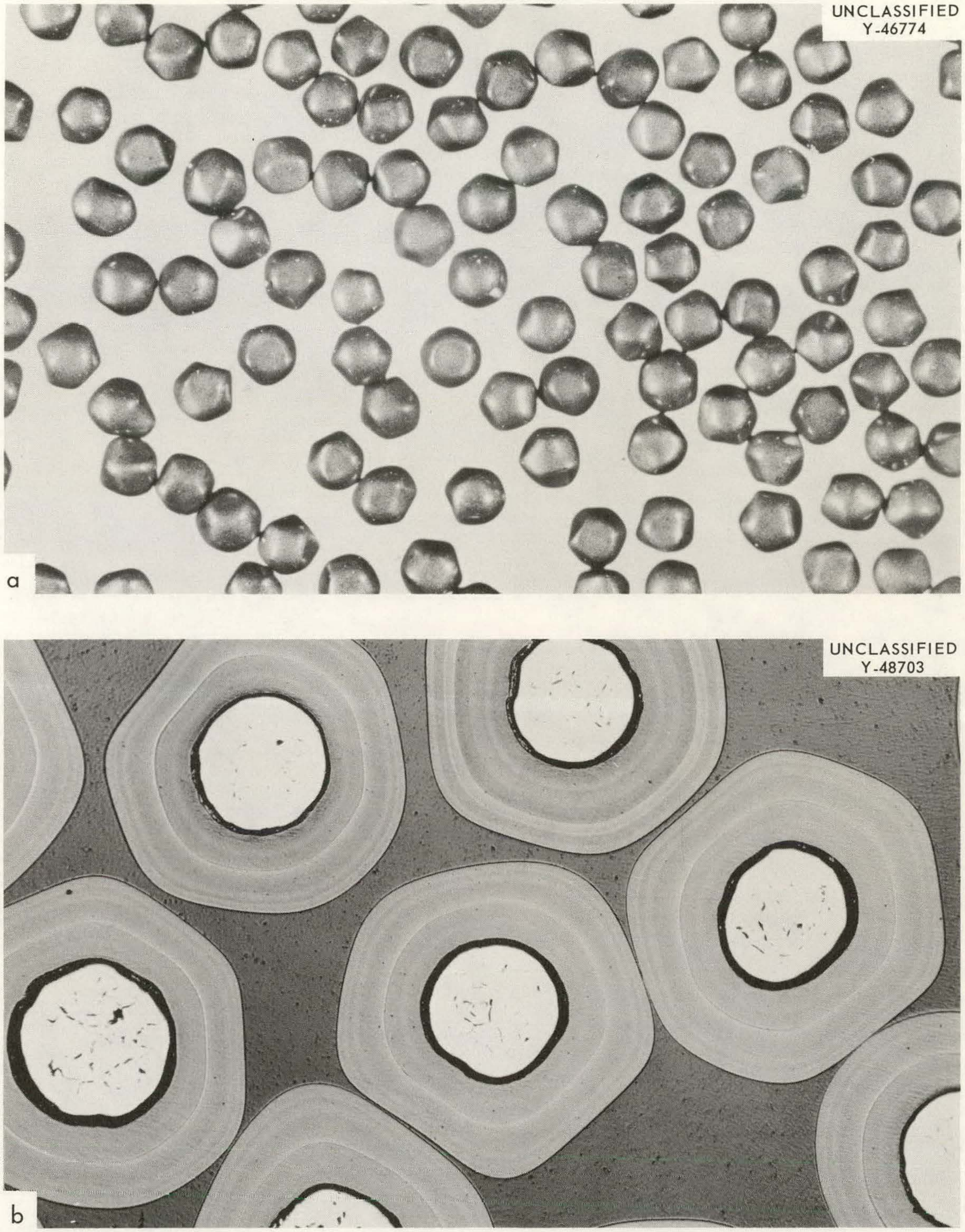


Fig. 5. Coated Particles from Batch 3M-117. As received. a. Unmounted. Oblique illumination. 33X. b. As polished. 100X.

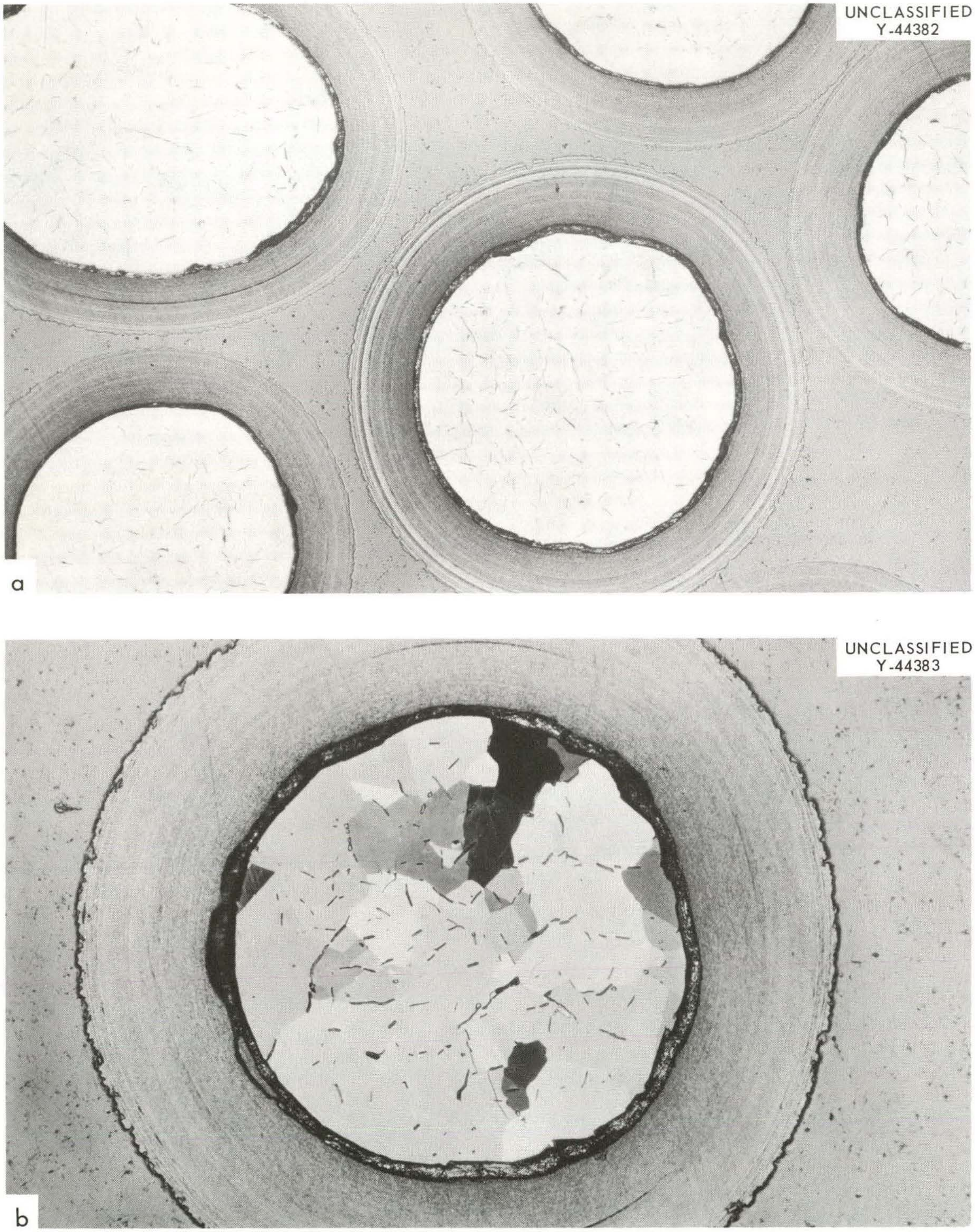


Fig. 6. Coated Particles from Batch 3M-D After 100-hr Exposure at 900°C to Helium Containing H_2O at a Partial Pressure of 20 mm Hg. a. As polished. 100X. b. Etched. 200X.

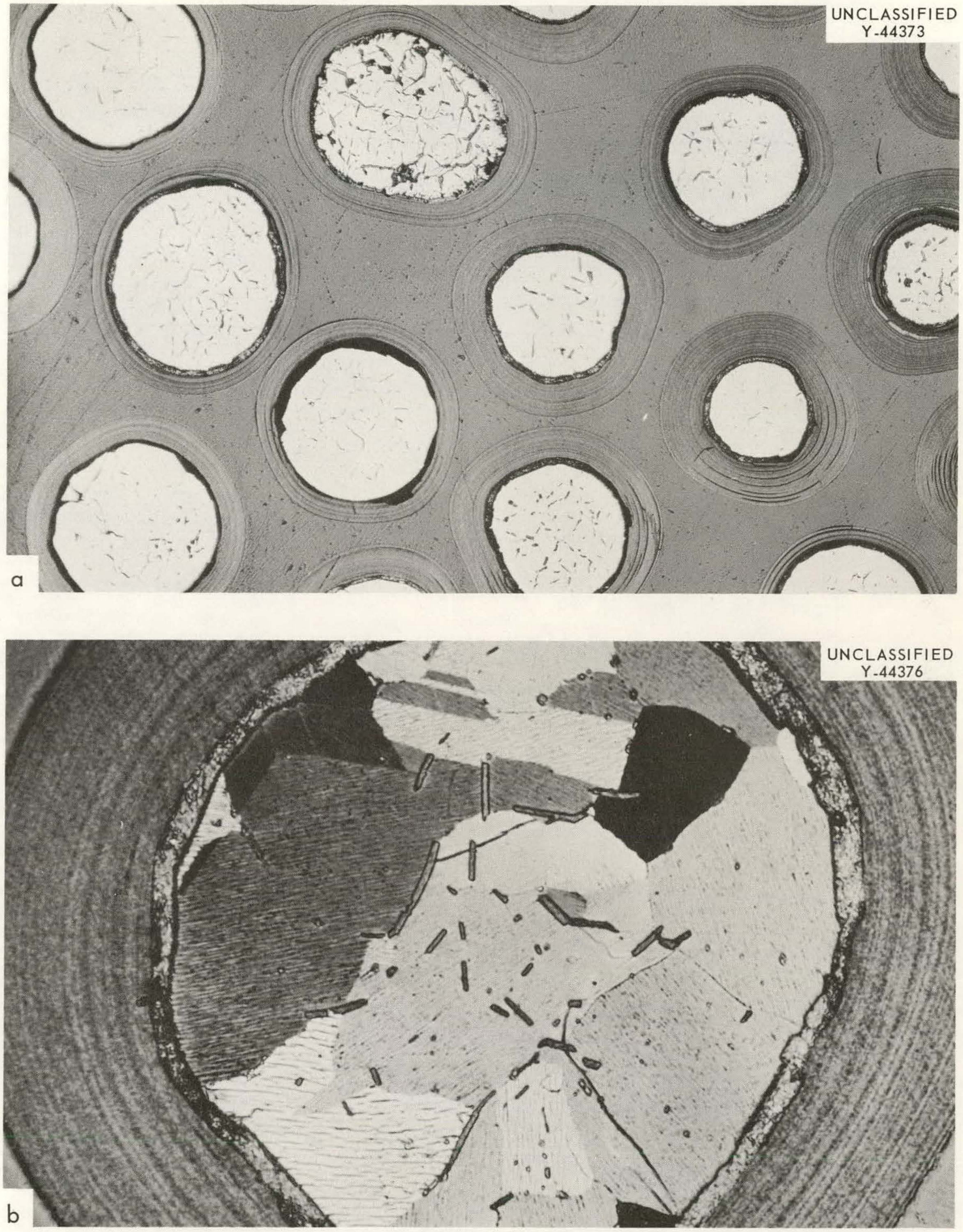


Fig. 7. Coated Particles from Batch 3M-F After 100-hr Exposure at 900°C to Helium Containing H_2O at a Partial Pressure of 20 mm Hg. a. As polished. 100X. Note cracking in the laminar coating. b. Etched. 500X.

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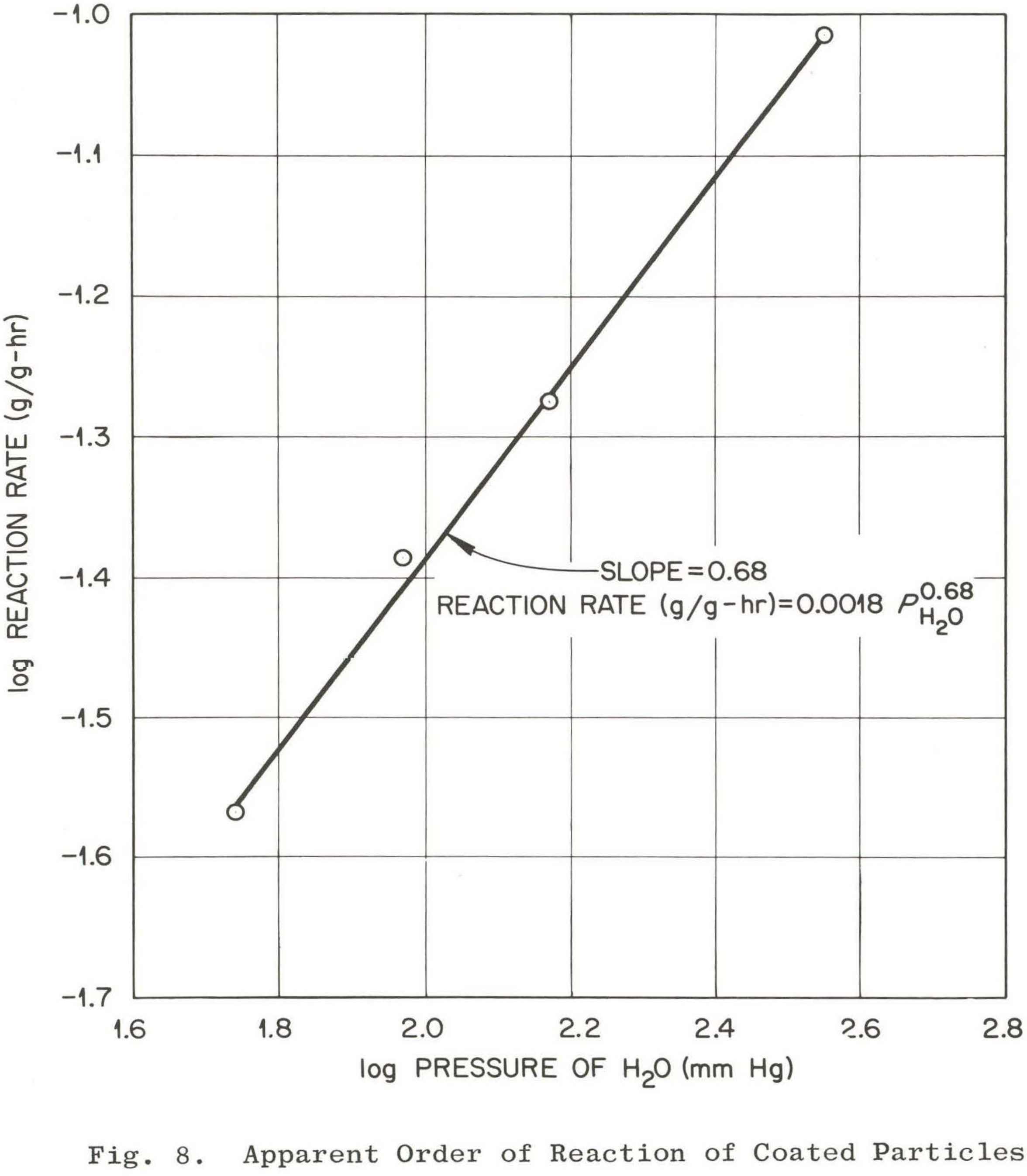

Fig. 8. Apparent Order of Reaction of Coated Particles from Batch 3M-114 with Steam at 1000°C.

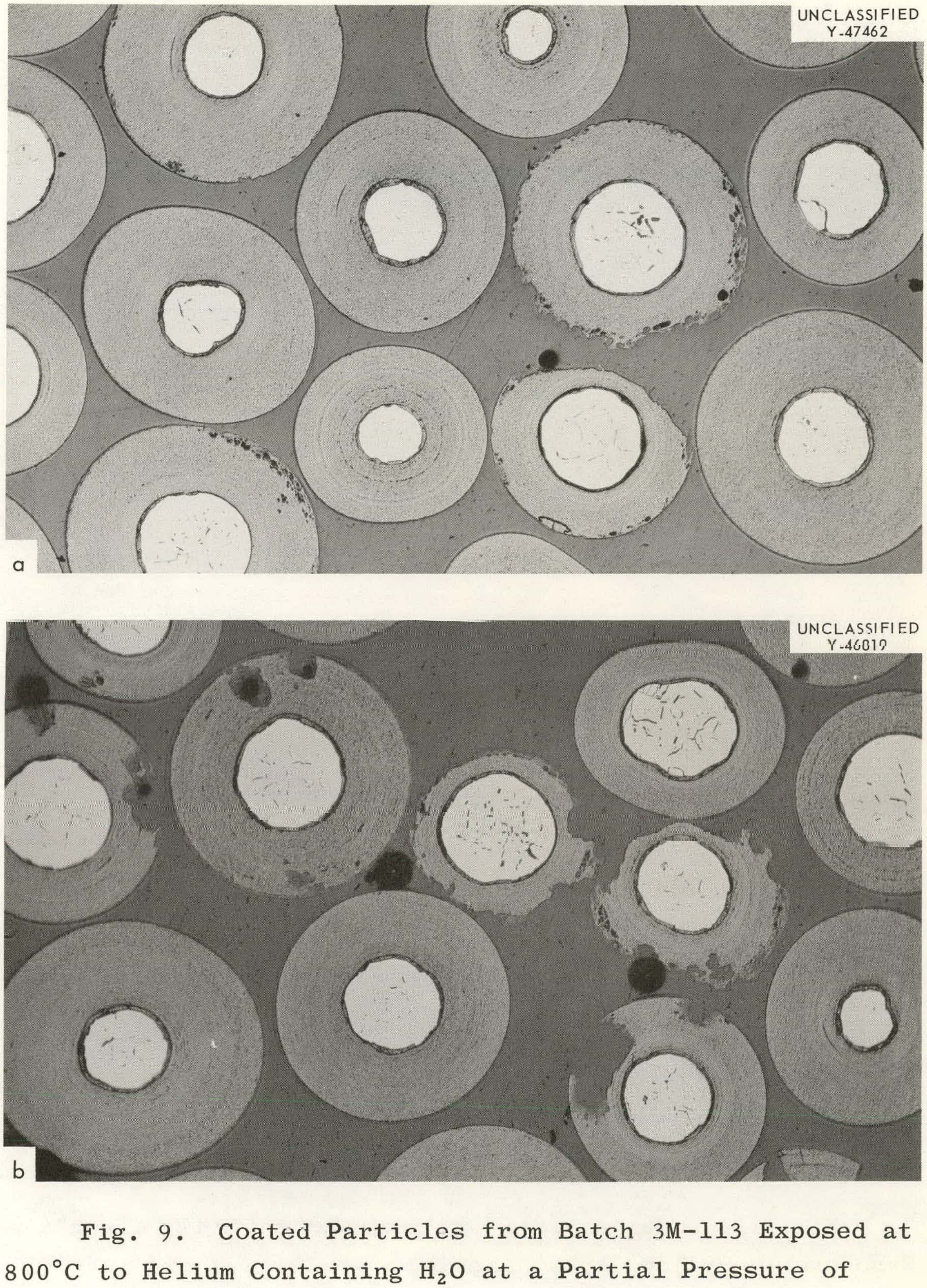

Fig. 9. Coated Particles from Batch 3M-113 Exposed at 800°C to Helium Containing H_2O at a Partial Pressure of 355 mg Hg. As polished. 100X. a. Exposed for 4 hr. b. Exposed for 7 hr.

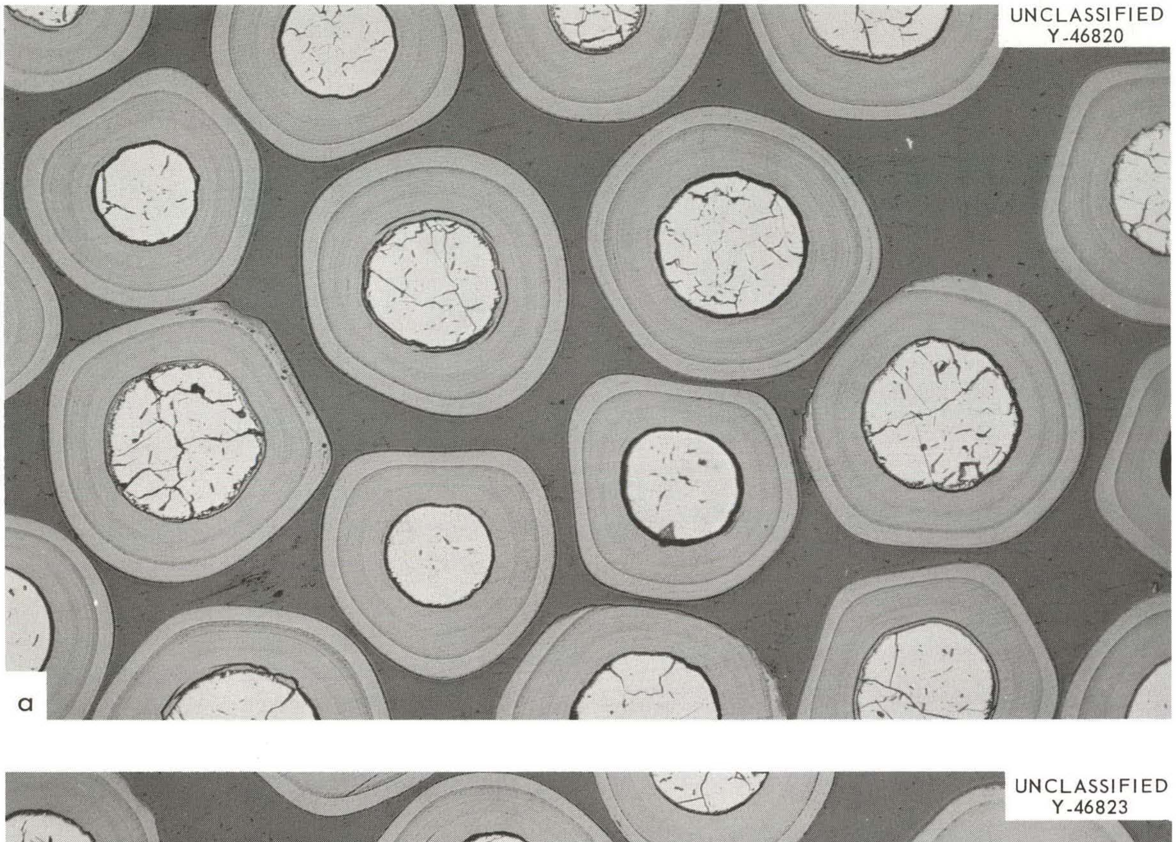

Fig. 10. Coated Particles from Batch 3M-114 After 6-hr Exposure to Helium Containing H_2O at a Partial Pressure of 233 mm Hg. As polished. 100X. a. Exposed at 800°C. b. Exposed at 900°C.

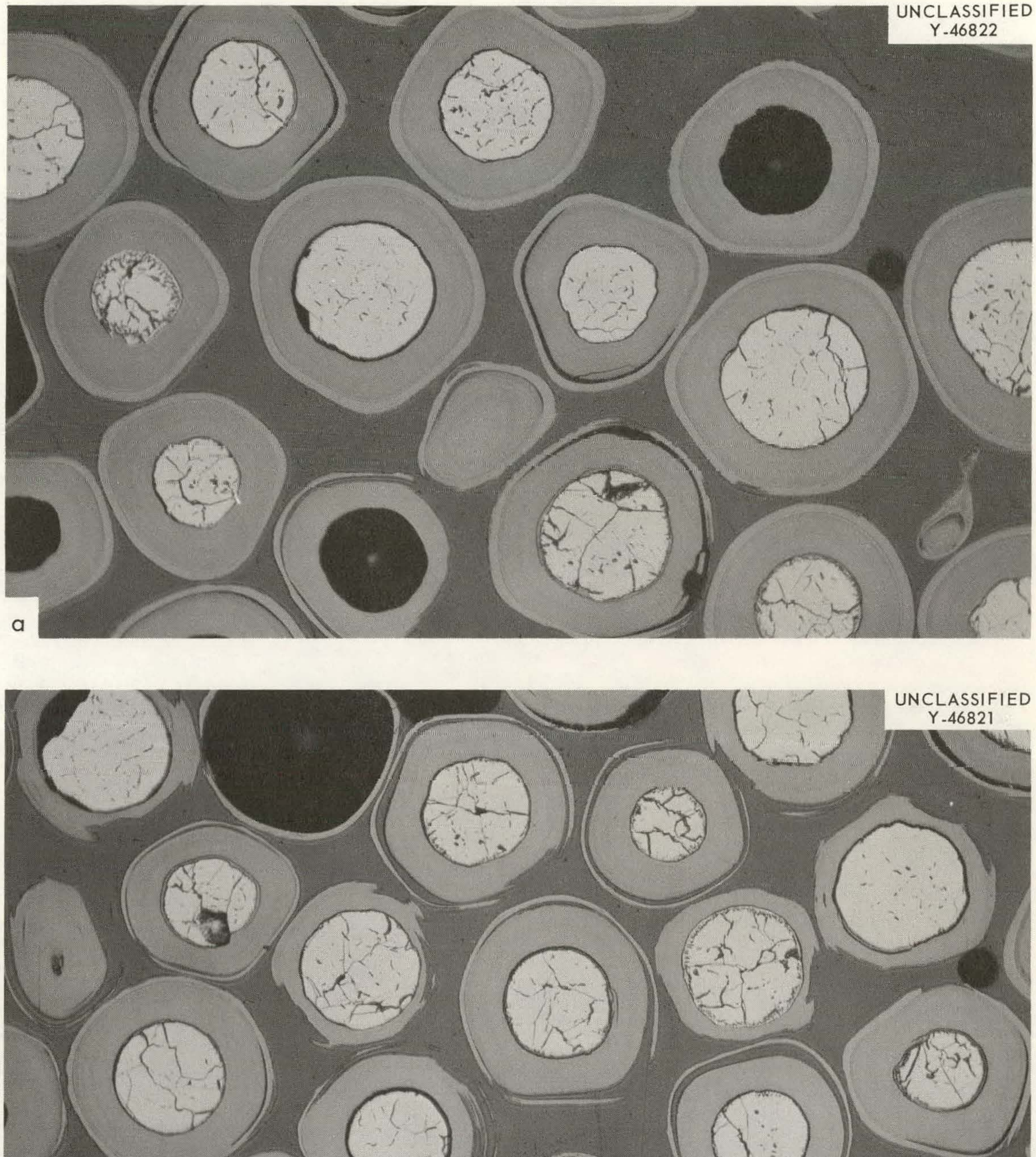

Fig. 11. Coated Particles from Batch 3M-114 After 4-hr Exposure at 1000°C to Helium Containing H_2O. As polished. 100X. a. Partial pressure of H_2O of 149 mm Hg. b. Partial pressure of H_2O of 355 mm Hg.

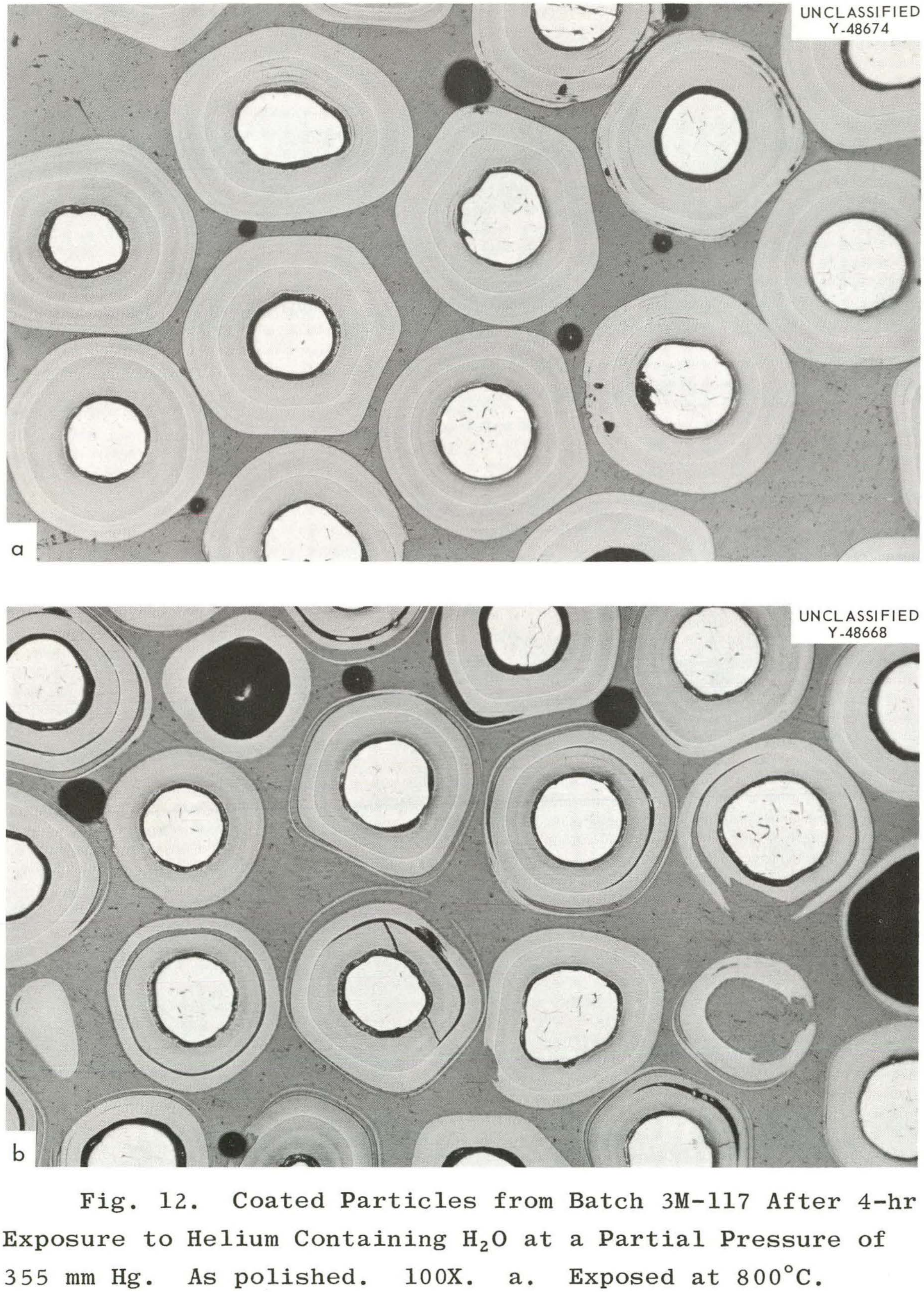

Fig. 12. Coated Particles from Batch 3M-117 After 4-hr Exposure to Helium Containing H_2O at a Partial Pressure of 355 mm Hg. As polished. 100X. a. Exposed at 800°C. b. Exposed at 1000°C.

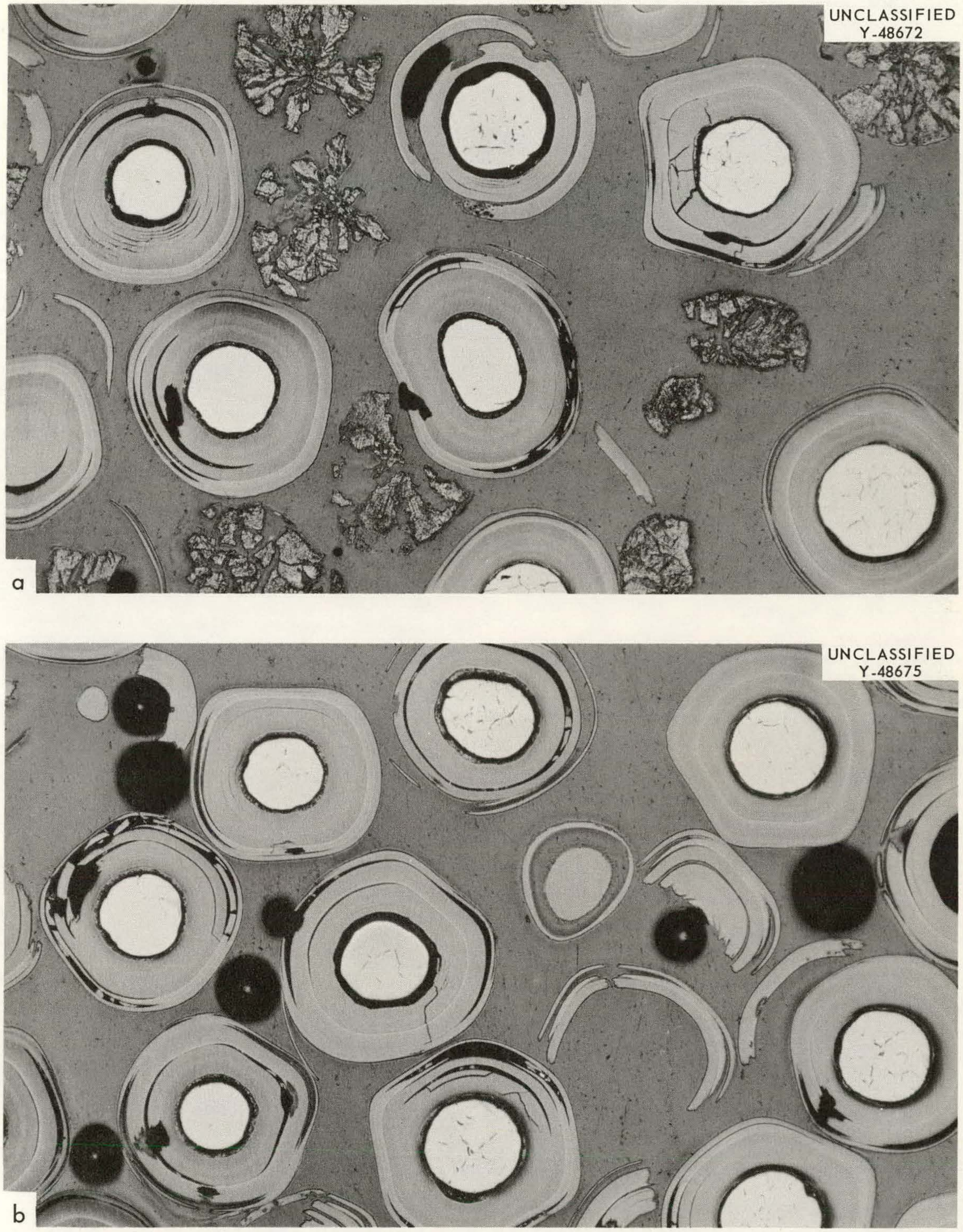

Fig. 13. Coated Particles from Batch 3M-117 After 1-hr Exposure at 1100°C to Helium Containing H_2O at a Partial Pressure of 635 mm Hg. As polished. 100X. a. Before HNO_3 leach. b. After HNO_3 leach.

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