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Metals, Ceramics,
and Materials

MASTER

NICKEL COATING OF CERAMIC SPHEROIDS
USING FINE NICKEL POWDER AND OXIDE (U)

F. Lambdin

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OAK RIDGE Y-12 PLANT

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FINE NICKEL POWDER AND OXIDE (U)

F. Lambdin

Oak Ridge, Tennessee

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ABSTRACT

The phenomenon of the natural adhesion of a fine powder to a larger body has been applied to a method of coating ceramic spheres with a protective nickel coat. Coatings were applied and evaluated using the following powders: (1) air-dried, high-surface-area nickel oxide screened to -325 mesh, (2) nickel powder produced by the hydrogen reduction of the air-dried, high-surface-area nickel oxide, (3) combinations using the first two powders in alternate coating steps, and (4) vacuum-dried, high-surface-area nickel oxide.

All coatings were subsequently hydrogen reduced and sintered in place on the spheres. The resulting coats showed that the air-dried oxide produced serious cracking while the metal powder gave extremely thin coats with no appreciable cracking. The vacuum-dried oxide (low bulk density) showed good adhesion and produced fairly uniform, dense coatings in the thickness range of one mil. The method using the vacuum-dried nickel oxide could be applied to large-scale coating applications.

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INTRODUCTION

A great variety of methods have been studied and developed for coating small particles and spheroids to protect them from deterioration. Of some interest, because of relative simplicity and economy, is the direct application of the coating material in finely divided form, either as pure metal or as metallic oxide, relying on natural adhesion to retain the coating until it can be consolidated by sintering or (in the case of oxide powders) by reduction to metal followed by sintering. No very specialized equipment or involved techniques appear to be needed, and technology developed for one type of coating (nickel) may be adaptable to other coatings.

Nickel was selected for the study reported in this paper because of the large amount of information available about the technology of nickel coating and because nickel is protective in many different environments. Uranium dioxide was chosen as the particulate substrate because Y-12 has wide experience in the controlled production of this material and because the uranium provided a way to evaluate the coating by observing the extent to which alpha emission escaped from the coated particles. Furthermore, successful coatings on uranium dioxide might have reactor applications.

SUMMARY

Methods were investigated for applying a protective coating of pure nickel to uranium dioxide spheroids in the 60-mesh size range, utilizing fine nickel oxide and metal powder. These coatings used no binders and depended upon natural adhesion of the fine powder to the dioxide particle. First, air-dried, high-surface-area nickel oxide screened to -325 mesh and the metal powder made by its hydrogen reduction were used individually to achieve the coatings in the range of one mil in thickness. Second, the oxide and metal powders were used in alternate coating steps. Third, vacuum-dried, high-surface-area nickel oxide was used to produce the coatings.

Results of the investigations demonstrated the following:

1. The coatings applied by the use of air-dried nickel oxide showed serious surface cracking, although the coatings could be made thick.
2. The coatings applied by the use of high-surface-area nickel powder were thin (< 0.3 mil) due to the limited adhesive properties of the powder. These coatings did not show serious surface cracking.
3. Coatings using combinations of air-dried nickel oxide and nickel metal powder were unsuccessful due to excessive cracking.
4. Vacuum-dried, high-surface-area nickel oxide showed good adhesion toward the uranium dioxide spheres. The oxide coatings did not crack when reduced and sintered.
5. Oxides with lower bulk density gave better coatings than oxides with higher bulk densities.
6. The alpha activity of the uranium dioxide was reduced to approximately 100 counts per minute (originally 30,000 cpm) by the addition, in four steps, of a coating consisting of 13 volumes of nickel oxide (where original UO_2 volume is 1.0).
7. Smooth, dense, continuous coatings, ranging in thickness from 0.75 to 2.0 mils, can easily be applied to uranium dioxide spheres by coating them with vacuum-dried, high-surface-area nickel oxide followed by hydrogen reduction and sintering of the spheres in the same furnace.

STUDIES OF NICKEL COATINGS ON CERAMIC SPHEROIDS

PREPARATION OF MATERIALS

Nickel Oxide

The first step in the coating process was to prepare quantities of ultra-fine nickel oxide powder which could be used to coat the spheres directly or be reduced to metallic nickel before coating. The oxide powder used in this process was prepared in the following manner:

1. Purified nickelous nitrate (500 grams) was dissolved in two liters of distilled water contained in a four-liter Pyrex beaker.
2. The beaker of nickelous nitrate was heated to 85° C while being stirred with a laboratory-size stirrer.
3. While the nickelous nitrate was being stirred, equal volumes of 30% hydrogen peroxide and 58% ammonium hydroxide were added to the solution dropwise at the base of the vortex formed by the stirring. The addition was continued until the pH of the nickelous nitrate reached 6.8. (Initially, the pH was 5.5.)
4. The resulting nickelous hydroxide (green) precipitate was retained on a Whatman 40 filter paper.
5. Excess water was removed from the hydroxide and it was converted to the oxide in a forced-air oven operating at 290° C. The hydroxide was usually dried and converted within 24 hours.

Table 1 shows the characteristics of this powder. The material was highly agglomerated and did not deagglomerate on hydrogen reduction at 300° C, so it was broken

Table 1
CHARACTERISTICS OF FINE, AIR-DRIED NICKEL OXIDE
(Batch 151)

Helium Density (gms/cc)	5.0
Bulk Density (gm/cc)	0.64
Nickel Content (%)	70.13
Surface Area (m ² /gm)	26.45
Surface Area (m ² /cc)	132.25
Mean Particle Diameter (micron)	0.045
Major Contaminants (ppm)	
C	274
Cu	6
Pb	20
Si	60

up to pass through a -325 mesh screen. The nickel content compared approximately to nickel (III) oxide.

Other quantities of nickel oxide were prepared by approximately the same procedure except that the green hydroxide was vacuum dried for 24 hours at a temperature of 190° C before air drying for 24 hours at 290° C. The vacuum-drying technique produced fluffy oxide which was not highly agglomerated. The characteristics of four batches of oxide produced by this method are reported in Table 2. The nickelous nitrate used in these batches was reagent grade.

Table 2
CHARACTERISTICS OF FINE, VACUUM-DRIED NICKEL OXIDE

	Batch			
	1-185	2-185	1-215	2-215
Nickel Content (%)	75.61	73.50	75.48	73.53
Surface Area (m ² /gm)	12.90	15.45	14.26	16.61
Mean Particle Diameter (micron)	0.093	0.077	0.084	0.073
Bulk Density (gm/cc)	0.385	0.47	0.55	0.34
Major Contaminants (ppm)				
Al	800			
C	48			
Co	500			
Mg	50			
Pb	50			
Si	250			

The tables show that air drying and screening produced a higher-surface-area oxide than vacuum drying. The vacuum drying, however, produced an oxide which was fluffy, as indicated by the lower bulk density. The reagent-grade nickelous nitrate produced a more impure oxide than that produced by the purified grade.

Nickel Metal

A quantity of the agglomerated -325 mesh oxide reported in Table 1 was hydrogen reduced in the facilities shown in Figure 1. Nickel boats approximately 1/2-inch deep were filled with the material and inserted in the tube. The tube was then purged with a helium flow before starting the heat and hydrogen flow. The temperature was brought up to 300° C and held for three hours with a hydrogen flow of approximately 5 liters/minute. The hydrogen was purified by the use of the platinum catalyst and drying tower shown in Figure 1. The nickel metal powder produced in this manner was pyrophoric on contact with air. Consequently, it was necessary to use an inert-atmosphere glove box to remove the product. The average surface area of the metal powder produced was ~5.0 meters per gram, while the oxygen content was about 1.0% after slowly exposing to air to prevent complete oxidation. The oxide represented in Table 2 was not reduced to metal.

Figure 1. FACILITIES FOR REDUCING FINE NICKEL OXIDE AND SINTERING THE NICKEL METAL POWDER.

Coatings Using Nickel Metal Powder

Figure 2 shows a typical flow diagram to coat uranium dioxide spheres with high-surface-area nickel metal powder. The powder was produced by the hydrogen reduction of the agglomerated high-surface-area oxide described earlier (Table 1).

The dioxide spheres were initially mixed with the pyrophoric nickel powder without air exposure. No coating was accomplished. A small amount of distilled water was then added and the mixture rolled in a glass jar for approximately 15 minutes. Some coating of the spheroids was observed, but an appreciable excess of the nickel powder was separated when the mixture was screened on a 100-mesh screen. The coated material was then reduced at 300° C and sintered at 800° C. The resulting coating is shown in Figure 3(a). As can be seen, a very thin, discontinuous nickel coating resulted.

These coated spheres were then divided into two parts to test the role of the water addition to the mixture. In one, the spheres and air-exposed nickel powder were mixed before the water addition as before. In the other section, the spheres were wet with water before the air-exposed nickel powder was added. No difference in coating ability was observed in the two methods. Both were poor, as is shown in Figure 3(b) and (c). Next, the sections were recombined, mixed with air-exposed nickel powder, screened, and sintered. This coating stuck to the spheres better and made a thicker coating but still was uneven, as shown in Figure 3(d). A further coating cycle gave approximately the same results. The alpha count of the uranium dioxide was reduced from 20,000 counts per minute to approximately 750.

Although the alpha counts were successively reduced by the addition of the metal powder coatings, a smooth, continuous coating was not achieved. In most cases, half of the nickel powder was removed by screening. Air-exposed powder seemed to coat the spheres better than that achieved by moisture addition.

Figure 2. FLOW PATTERN FOR NICKEL COATINGS USING NICKEL METAL POWDER ONLY.

Coatings Using Air-Dried, High-Surface-Area Nickel Oxide Screened to -325 Mesh

Since the air-exposed nickel metal (high oxygen content) seemed to coat the spheres better than that achieved by moisture addition, a series of coatings was tried using only the air-dried, high-surface-area oxide of Table 1. These coatings were followed by a hydrogen reduction at 300° C and sintering at various temperatures without removing the spheres from the furnace.

The steps used to apply this coating are shown in Figure 4. An equal volume of the oxide was mixed with the dioxide spheres followed by rolling in a glass jar for 15

18252

(a) Specimen 1-159

400X

18341

(b) Specimen 1-165

130X

18342

(c) Specimen 2-165

130X

18463

(d) Specimen 1-169

125X

Figure 3. PHOTOMICROGRAPHS OF BEADS WITH NICKEL COATINGS
USING NICKEL METAL POWDER ONLY.

Figure 4. FLOW PATTERN FOR NICKEL COATINGS USING AIR-DRIED, HIGH-SURFACE-AREA NICKEL OXIDE.

minutes. The oxide coated the spheres better than the air-exposed nickel metal powder since only very small amounts were screened out by the 100-mesh screening step. The results of reducing and sintering the nickel oxide in place on the spheres are shown by the photographs of Figure 5(a). It can be seen that the thickness of the nickel coating increased by recoating to approximately 0.5 mil; however, the coating was not continuous. Extensive cracking of the thicker coating, shown in Figure 5(b), can be seen in the 33X view. The alpha count was decreased from 20,000 to 2100 cpm by this two-step process. Attempts to achieve a thick coating by one large addition of the nickel oxide powder proved unsuccessful due to large-scale cracking of the coating.

18110

33X 18110
(a) Specimen 1b-154

440X

18241

33X 18241
(b) Specimen 2-157

440X

Figure 5. PHOTOMICROGRAPHS OF BEADS WITH NICKEL COATINGS USING AIR-DRIED, HIGH-SURFACE-AREA NICKEL OXIDE.

Coatings Using Nickel Metal Powder and Air-Dried, High-Surface-Area Nickel Oxide Screened to -325 Mesh

Since the nickel oxide powder gave thicker coatings but with more surface cracking than the nickel metal powder, a series of coating steps was attempted using oxide followed by fill-in coats of metal powder. The flow diagram of this coating sequence is given in Figure 6 with photographs of the results in Figure 7.

The uranium dioxide spheres were mixed with the air-dried, high-surface-area nickel oxide which had been screened to -325 mesh (Table 1). The mixture was rolled in a glass jar where good particle coating was accomplished. Reduction of this material at 300° C followed by sintering at 800° C produced the coated spheres shown in Figure 7(a). As can be seen, large-scale surface cracking occurred. This cracking can again be attributed to the large nickel oxide-to-uranium dioxide volume ratio used. Large areas of the dioxide spheres can be observed which are not covered by the nickel coat. These coated spheres were then mixed and rolled with air-exposed metal powder which had been produced by the reduction of the oxide which was used for the first coat. The rolling was followed by a sintering cycle at 800° C. The photographs of Figure 7(b) show the results of this coating. The metal powder filled the cracks to a large degree, producing a much smoother coat; however, it was still discontinuous in some places.

A further coating, in the same manner, produced the spheres shown in Figure 7(c). Further reduction in the coat cracking is apparent with the total thickness of the coat reaching approximately one mil. A good tight coating was not achieved, as shown in the larger magnification where it can be seen that parts of coatings were not well established. A further coating attempt using the same method produced spheres where a large number were uncoated, and the thicker coatings cracked to a point where they became detached from the dioxide sphere. The alpha count of the uranium dropped after the oxide coat, remained at about the same level after the first metal powder coat, decreased with the second metal powder coat, then increased after the third metal powder coat.

Coating steps using nickel metal powder as the base coat followed by oxide coats were unsuccessful due to the lack of attaining a good continuous coating using the metal powder.

Coatings Using Vacuum-Dried, High-Surface-Area Nickel Oxide

The most desirable characteristic of the screened nickel oxide was its tendency to stick to the uranium dioxide spheres, but at the same time it gave large cracks after reducing and sintering. In an effort to increase the adherence and reduce the cracking problem, a quantity of nickel oxide was prepared using the procedure given earlier except that the hydroxide drying was carried out in vacuum at 190° C. Vacuum drying was followed by air drying at 290° C. A very fine, fluffy oxide which

Figure 6. FLOW PATTERN FOR NICKEL COATINGS USING -325 MESH NICKEL OXIDE AND NICKEL METAL POWDER.

displayed a high degree of attraction for the dioxide spheres was produced by the change in the drying procedure. This nickel oxide did not produce cracked coatings after reduction and sintering when applied in moderate amounts. The characteristics of the vacuum-dried powder are listed in Table 2.

18601

32X

18714

32X

18715

32X

18601

130X

(a) Specimen 1-177

18714

130X

(b) Specimen 1-181

18715

130X

(c) Specimen 1-183

Figure 7. PHOTOMICROGRAPHS OF BEADS WITH NICKEL COATINGS USING -325 MESH NICKEL OXIDE AND NICKEL METAL POWDERS.

The first coating sequence using the vacuum-dried oxide is shown in Figure 8 while the photographs of Figure 9 show the results of these coating steps. The oxide was applied in the same manner as in other coating steps. Approximately five volumes of nickel oxide (Batch 1-185) to one volume of uranium dioxide spheres produced the coating shown in Figure 9(a). The coating, shown by the 32X view, appears to be smooth, although not continuous in some cases, is uniform, and approximately 0.5 to 0.75 mil in thickness. This coating, sintered at 600° C, is shown to be porous. A coating was then applied using equal volumes of the nickel oxide powder and dioxide spheres. Further coats consisting of 5:1 and 3:1 volumes of nickel oxide powder and spheres were separately applied. Following the 3:1-volume coat, the coated spheres were sintered at 900° C for four hours. This operation produced the spheres shown in Figure 9(b). The 900° C sintering step sintered the coated spheres together into a friable mass, but the sintering and subsequent breaking up of the mass produced small pull outs on the nickel-coated surface which are visible in Figure 9(b). As noted in Figure 8, this action caused an increase in the alpha count, but the count was lowered by the small volume of nickel oxide added for the next coat. After the next small coating was applied, the coated spheres were sintered at 850 and 900° C, followed by a sinter at 1000° C. As seen in Figure 9(c), no appreciable coating deterioration was observed in breaking up the sintered masses of spheres at 900 and 1000° C.

The doublets (2 spheres coated together) are a result of incomplete breaking up of the mass of spheres following a sintering cycle before recoating with powder.

The 850° C sintering step, shown in Figure 9(c), produced relatively well-sintered coatings which varied from 0.25 to 1.5 mils.

A coating sequence, where weighed amounts of nickel oxide were used, is shown in Figure 10, and photographs of the resulting coatings are shown in Figures 11, 12, and 13. The first oxide coating (4.72 volumes) on the uranium dioxide spheres before reduction and sintering is illustrated in Figure 11(a). The smooth surface of the unbroken coats and the thickness of the broken coat are noteworthy. Figure 11(b) shows the coated spheres after the first sintering cycle. Some uncoated spheres and very limited cracking on other spheres can be seen. The coated spheres were removed from the uncoated after each sintering step by a magnetic separation step using a permanent magnet. The limited cracking shown in Figure 11(b) is the only case where this problem was apparent in the coating sequence. The photographs in Figure 11(c) show the spheres after the second coating step; those in Figure 11(d) show the coatings after the third addition of powder. A total of 10.34 volumes (original dioxide spheres equal 1 volume) of nickel oxide had been added to this point. The photographs in Figure 12(a) show the coating result from the addition of a total of approximately 13 volumes of nickel oxide powder. Figure 12(b) and (c) shows the spheres sintered to 800 and 850° C, respectively, without further powder addition. The sintering action on the porosity of the nickel coating is noteworthy. The photographs in Figure 13(a) and (b) show the result of sintering at 925 and 975° C, re-

Figure 8. FLOW PATTERN FOR NICKEL COATINGS USING VACUUM-DRIED, HIGH-SURFACE-AREA NICKEL OXIDE.

18759

32X

18791

32X

18859

32X

18759

130X

(a) Specimen 1-191

18791

130X

(b) Specimen 1-201

18859

130X

(c) Specimen 1-209

Figure 9. PHOTOMICROGRAPHS OF BEADS WITH NICKEL COATINGS USING VACUUM-DRIED, HIGH-SURFACE-AREA NICKEL OXIDE.

Figure 10. FLOW PATTERN FOR NICKEL COATINGS USING WEIGHED AMOUNTS OF VACUUM-DRIED NICKEL OXIDE.

18897

32X

(a) Specimen 1-213

18940

32X

19036

32X

18899

32X

(b) Specimen 1-213, Sintered

18940

130X

(c) Specimen 1-219

19036

130X

(d) Specimen 1-221

Figure 11. PHOTOMICROGRAPHS OF BEADS WITH NICKEL COATINGS USING WEIGHED AMOUNTS OF VACUUM-DRIFTED NICKEL OXIDE. (Beads after the First, Second, and Third Additions of Powder)

19037

32X

19038

32X

19039

32X

19037

130X

(a) Specimen 2-221

19038

130X

(b) Specimen 1-225

19039

130X

(c) Specimen 2-225

Figure 12. PHOTOMICROGRAPHS OF BEADS WITH NICKEL COATINGS USING WEIGHED AMOUNTS OF VACUUM-DRIED NICKEL OXIDE. (Beads after the Fourth Addition of Powder and Further Sintering)

32X

 19040 130X
 (a) Specimen 3-225

19040

32X

 19041 130X
 (b) Specimen 1-227

19041

Figure 13. PHOTOMICROGRAPHS OF BEADS WITH NICKEL COATINGS USING WEIGHED AMOUNTS OF VACUUM-DRYED NICKEL OXIDE. (Beads after Additional Sintering at 925 and 975°C)

spectively. The small uncoated areas are noteworthy in these photographs, thought to be caused by the action of being sintered together and then broken apart. Note that the alpha count increased slightly (approximately 100 cpm) due to this action. Attention is directed toward the almost complete absence of voids in the nickel coat of Figure 13(b) (at 130X). This fully sintered coating appears to vary in thickness from 0.75 to 2.0 mils.

It appears that if the dense coating produced by the higher temperature is required, a sintering medium would be required to prevent the spheres from contacting.

Although no photographs are shown of nickel oxide coatings using vacuum-dried powder other than Batch 1-185, coatings were attempted with the other batches of Table 2. The lower bulk-density batches of nickel oxide showed higher adhesion to the dioxide spheres and better sintering characteristics such as the elimination of cracks. This oxide was used to successfully coat spheres of various materials such as polyethylene. The 100-mesh screening used after the coating step was eliminated by the use of the vacuum-dried oxide due to the fact that all the oxide stuck to the spheres.

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