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(M-3679, 19th Edition)

ELECTROKINETIC PROCESSES - NUCLEAR ASPECTS

QUARTERLY PROGRESS REPORT

February 1 - April 30, 1957

by

A. C. Werner, H. Katz

May 15, 1957

Contract No. AT(30-1)-1996, Vitro Job 2091

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ELECTROKINETIC PROCESSES - NUCLEAR ASPECTS

QUARTERLY PROGRESS REPORT

FEBRUARY 1 - APRIL 30, 1957

Research Supervisor: A. C. Werner
Staff: H. Katz

ABSTRACT

A number of coatings were investigated for protection of graphite against high-temperature oxidation and erosion. Well-sintered, well-bonded silicon carbide coatings were obtained by depositing elemental silicon on graphite, densifying, and sintering in CO at 1300°-1400°C. However, the coatings were not perfect since complete protection was not offered against oxidation. This work will be continued.

A 75-15-10 nickel-chromium-silicon alloy sintered at 1100°, 1200°, and 1300°C wet graphite very well, coatings with high metallic luster being obtained at the higher temperatures.

Fecral, alumina, and chromium-silicon-iron-aluminum alloys were deposited electrophoretically on graphite. Although excellent green coatings were obtained, sintered coatings were poor. The fecral coatings did not wet the surface; the alumina coatings were brittle and non-adherent indicating that a higher temperature was necessary.

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QUARTERLY PROGRESS REPORT

FEBRUARY 1 - APRIL 30, 1957

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INTRODUCTION

This is the fourteenth of a series of formal quarterly progress reports on Electrokinetic Processes - Nuclear Aspects for the Atomic Energy Commission. As of December 1, 1956, the original contract (Contract No. AT(30-1)-850) was extended as Contract No. AT(30-1)-1996. For purposes of internal contract control, this work is now identified as Vitro Job 2091 (an extension of Vitro Job 2018 which was an extension of Vitro Job 1099).

Previous Reports

KLX-1714, Quarterly Progress Report, November 1, 1953 - January 31, 1954, Job 1099, dated February 15, 1954.

KLX-1721, Quarterly Progress Report, February 1 - April 30, 1954, Job 1099, dated May 15, 1954.

KLX-1726, Quarterly Progress Report, May 1 - July 31, 1954, Job 1099, dated August 10, 1954.

KLX-1737, Quarterly Progress Report, August 1 - October 31, 1954, Job 1099, dated November 30, 1954.

KLX-1744, Quarterly Progress Report, November 1, 1954 - January 31, 1955, Job 2018, dated February 21, 1955.

KLX-10001, Quarterly Progress Report, February 1 - April 30, 1955, Job 2018, dated May 31, 1955.

KLX-10009, Quarterly Progress Report, May 1 - July 31, 1955, Job 2018, dated August 31, 1955.

KLX-10017, Quarterly Progress Report, August 1 - October 31, 1955, Job 2018, dated December 15, 1955.

KLX-10021, Quarterly Progress Report, November 1, 1955 - January 31, 1956, Job 2018, dated February 15, 1956.

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KLX-10029, Quarterly Progress Report, February 1 -
April 30, 1956, Job 2018, dated May 15, 1956.

KLX-10041, Quarterly Progress Report, May 1 - July 31,
1956, Job 2018, dated August 15, 1956.

KLX-10056, Quarterly Progress Report, August 1 -
October 31, 1956, Job 2018, dated November 15, 1956.

KLX-10065, Quarterly Progress Report, November 1, 1956 -
January 31, 1957, Job 2018 and 2091, dated February 15, 1957.

SUMMARY

A number of coatings were investigated for protecting graphite against high-temperature oxidation and erosion. The coatings were applied by electrophoretic techniques, from zein-activated dispersions.

Silicon carbide coatings on graphite were prepared by electrophoretically depositing silicon, and sintering in CO at 1300° and 1400°C. Well-sintered, well-bonded, crack-free coatings were obtained at the higher sintering temperature. Pressed coatings appeared to be better sintered than unpressed coatings. These coatings provided increased, although not complete, oxidation resistance. Knoop hardness tests indicated that the coatings were not as hard as commercial silicon carbide.

Nickel (75% by weight), chromium (15% by weight), silicon (10% by weight) coatings on graphite sintered well in argon at 1200° and 1300°C. The coatings were well-bonded with high metallic luster. At 1300°C, there was an apparent flow of molten metal to the lower area of the graphite samples. It was noted that this alloy has a strong wetting action upon graphite, and may be useful for joining graphite components. Photomicrographs of these sintered coatings indicated that both pressed and unpressed coatings had low porosity and excellent bonding to the graphite surface.

Attempts to prepare coatings of fecral, a 70.3% iron, 23.7% chromium, 6% aluminum alloy were successful. Although the coatings sintered well, bonding to the graphite was very poor.

Three different alumina formulations were electrophoretically deposited on graphite specimens. One dispersion contained 96% levigated alumina, 2% cupric oxide powder, and 2% titanium dioxide powder. A second dispersion composition consisted of 96% alumina, 2% titanium dioxide and 2% manganese oxide. The third dispersion contained 98% alumina, 0.5% bentonite and 1.5% Kentucky No. 4 ball clay. Excellent green coatings were obtained. After isostatic pressing, the coated specimens were fired in purified argon at 1400°C; however, the final coatings were not well sintered.

Graphite specimens were coated with a dispersion containing 56.8% chromium, 41.5% silicon, 0.4% aluminum, and 1.3% iron, by weight, of the individual metal powders. The specimens were pressed isostatically, and fired in argon at 1350°C. The coatings were poorly sintered, and poorly bonded to the graphite surface.

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EXPERIMENTAL

Silicon Carbide Coatings on Graphite

Silicon was electrophoretically deposited on graphite. Pressed and unpressed coated specimens were sintered in CO at 1300° and 1400°C. Well-sintered, well-bonded, crack-free coatings were obtained at the higher sintering temperature. The pressed coatings appeared to have a higher degree of sintering than the unpressed coatings. The surface color was not uniform, areas varying from light gray to black. Microscopic examination of polished cross-sections indicated that the pressed coatings sintered at 1400°C had a low porosity and good bonding. The coatings averaged approximately 4-mils thick. A Tukon Hardness Tester was used to determine the Knoop Hardness Number of the coating. The Knoop Hardness Numbers obtained, from 505 to 645, are much lower than the hardness of commercial silicon carbide. An oxidation test was performed at 1000°C in static air. A comparison of a coated sample with two uncoated graphite panels is shown in Figure 1.

Although the coating gives considerable protection, it does not appear to be as good as would be expected of a dense silicon carbide coating. This consideration, in addition to the low micro hardness numbers obtained, indicate that some free silicon may remain in the coating. Improved coatings may be obtained by a longer sintering time in CO, using a higher sintering temperature, applying thicker coatings, and by using a plating dispersion containing silicon carbide plus silicon instead of silicon alone.

Nickel-Chromium-Silicon Alloy on Graphite

A nickel (75% by weight), chromium (15% by weight), silicon (10% by weight) dispersion was used to coat graphite. Pressed and unpressed specimens were sintered in argon at 1100°, 1200° and 1300°C. Well-bonded coatings, with high metallic luster, were obtained at 1200°C and 1300°C. At 1300°C, there was an apparent flow of molten metal to the lower area of the graphite samples. It was noted that this alloy has a strong wetting action upon graphite, and may be useful for joining graphite components. Photomicrographs of cross-sections of these sintered coatings indicated that both pressed and unpressed coatings had low porosity and were strongly bonded to the graphite surface. Coatings averaged approximately 2-mils thick.

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FIGURE I OXIDATION OF GRAPHITE AT 1000°C IN STATIC AIR

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Fecral Coatings on Graphite

Fecral, an alloy containing iron (70.3% by weight), chromium (23.7% by weight), aluminum (6% by weight), has been reported in the literature to have high-temperature oxidation resistance superior to 80-20 Nichrome. A zein-activated dispersion containing iron, chromium and nickel powders in the ratio specified above was used to deposit coatings upon graphite. The coated specimens were placed in thin-wall rubber tubing, which was subsequently evacuated and sealed at both ends. The enveloped specimens were then subjected to an isostatic pressure of 20 tsi. After removing the envelopes, the specimens were fired in either a purified hydrogen or purified argon atmosphere at 1100°-1400°C. Coatings fired below 1400°C were not well sintered. Coatings fired at 1400°C in argon for 2 hours, were well-sintered and bonded. However, there was a tendency toward the formation of metallic beads upon the coated surface indicating poor wetting of the graphite surface by the alloy.

Alumina Coatings on Graphite

Three different alumina formulations were electrophoretically deposited on graphite. One dispersion contained 96% levigated alumina, 2.0% cupric oxide powder, and 2.0% titanium dioxide powder. The second dispersion contained 96% alumina, 2.0% titanium dioxide, and 2.0% manganese oxide. The third dispersion contained 98% alumina, 0.5% bentonite, and 1.5% Kentucky No. 4 ball clay. Excellent "green" coatings were obtained. After isostatic pressing, the coated specimens were fired in purified argon at 1400°C. The final coatings were not well-sintered. However, results indicated that this technique plus a slightly higher sintering temperature could produce dense, well-sintered alumina coatings on graphite.

Chromium-Silicon-Aluminum-Iron Alloy on Graphite

Graphite specimens were coated electrophoretically from a dispersion containing chromium powder (56.8% by weight), silicon (41.5% by weight), aluminum powder (0.4% by weight), and iron powder (1.3% by weight). The coated specimens were pressed isostatically, and fired in argon at 1350°C. The coatings were poorly-sintered, and poorly-bonded to the graphite surface.

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