CANADA

DEPARTMENT OF MINES AND TECHNICAL SURVEYS

OTTAWA

MINES BRANCH INVESTIGATION REPORT IR 63-91

EVALUATION OF "BERKATEKT" COATINGS FOR PROTECTION OF ZIRCONIUM AND URANIUM DURING HEAT TREATMENT

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W. G. HUTCHINGS & H. M. SKELLY

PHYSICAL METALLURGY DIVISION

COPY NO. 16

SEPTEMBER 12, 1963

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SUMMARY OF RESULTS

An evaluation was made of the effectiveness of a proprietary product, called "Berkatekt", as protection for zirconium and uranium against excessive oxidation during heat treatment. Berkatekt coating No. 301 protected small sheet specimens of a zirconium alloy from excessive oxidation, but the coating was not self-descaling unless the heat treatment was carried out at 900 °C and followed by water quenching. Berkatekt coatings No. 10 and No. 301 did not protect uranium from oxidation.

* Technician and ** Senior Scientific Officer, Nuclear Metallurgy Section, Physical Metallurgy Division, Mines Branch, Department of Mines and Technical Surveys, Ottawa, Canada.

1. INTRODUCTION

Some of the investigations carried out in the Nuclear Metallurgy Section involve the heat treatment of reactive metals such as zirconium and uranium alloys and, in order to avoid excessive oxidation, it is often necessary to take precautions such as sealing the specimens in quartz tubing or performing the heat treatment in molten salt or in vacuum. If these procedures could be obviated by some simple and economical method it would prove most useful. It was learned from articles in the technical journals [1,2] that F. W. Berk and Company Limited, were marketing a product called "Berkatekt" that had been developed by Rolls Royce Limited for protecting metals and alloys during heat treatment operations. The metal to be protected is coated with the appropriate grade of Berkatekt prior to heat treatment, several grades being available for protecting various metals, including zirconium and its alloys. Although the suppliers do not claim that Berkatekt will protect uranium or its alloys from oxidation, it was decided to investigate this application also.

F. W. Berk and Company Limited, forwarded samples of three grades of Berkatekt for experimental purposes, and this report describes the tests that were carried out with two of the grades.

2. EXPERIMENTAL MATERIAL

2.1 Coating Material

The following two types of Berkatekt coating are available: (a) a thin non-self-descaling non-fusing coating, and (b) a self-descaling coating, incorporating a fusible phase. Each of these types is available in several grades for various applications. Details of the two grades of Berkatekt that were investigated are as follows:

Berkatekt No.	Type of Coating	Metals Protected	Operating Temperature
10 (Red)	Non-self-descaling	Mild steel, stainless steel, nickel alloys, titanium.	600-1150 °C
301 (Green)	Self-descaling	Zirconium alloys.	700 °C (min)

Berkatekt is a combination of solution and suspension of various ingredients in a liquid medium or solvent. The solvent for Berkatekt No. 10 is toluene and that for Berkatekt No. 301 is thought to be trichlorethylene. Colouring matter has been added to facilitate distinguishing between the different grades - thus No. 10 is red and No. 301 is green.

Spectrographic analyses of the two grades were carried out by the Mineral Sciences Division and the results are given in the Appendix. The main elements detected in No. 10 were silicon, aluminum, iron, and magnesium, and in No. 301 were silicon, iron, zirconium, titanium and lead. The chief difference between these two grades appears to be in the higher zirconium, titanium and lead contents of No. 301 and the higher aluminum and magnesium contents of No. 10. These elements are most likely present as compounds - possibly oxides and silicates. Boron was detected in both products, but only in very small percentages.

2.2. Test Material

The zirconium specimens were prepared from some available 0.061 in. thick sheet of nominal composition 1.0% copper, 1.5% molybdenum, balance zirconium. It was rolled down to 0.015 in. thickness by the Metal Forming Section prior to the test specimens being cut out.

The uranium specimens were 3/16 in. thick discs machined from 3/4 in. diameter bar.

3. PROCEDURE AND RESULTS

3.1 Tests with Zirconium

All the tests on zirconium were carried out with Berkatekt No. 301. The test specimens were prepared by cutting the 0.015 in. thick sheet into 1-1/4 in. squares that were pickled for 30 sec in the following mixture:

nitric acid (conc.)	45 ml
hydrofluoric acid (48%)	10 ml
water	45 ml

The effectiveness of Berkatekt as protection against oxidation was assessed by heating the coated specimens to various temperatures for 15 or 60 min in air in a muffle furnace, and then carrying out a visual examination of the surface of the specimens. Some test specimens were cooled in air and others were quenched in water after heat treatment. Various thicknesses of coating were investigated to ascertain the optimum thickness under the conditions applying. When necessary, the Berkatekt was thinned down by adding the appropriate liquid vehicle. Uncoated specimens were also tested to provide a basis for comparison. The tests with zirconium were divided into three groups depending upon the procedure used to dry the Berkatekt coating before heat treatment. In every case the coating was applied by immersion of the specimen in the Berkatekt.

In the first group of tests each coating was allowed to dry unaided in air for 20 min before application of another coating and from one to five coatings were applied in this way. In all these tests, listed in Table 1, the Berkatekt protected the specimens from excessive oxidation. There was some darkening of the specimens but the oxidation was not nearly as severe as occurred with the uncoated specimens and the darkening could be readily removed by pickling. Berkatekt No. 301 is said to be a self-descaling coating but it was found that descaling did not occur until a temperature of 900 °C was reached and then only in the case of specimens with two or more coats that were quenched in water. The coating was adherent after heat treatment at temperatures up to 800 °C although specimens with four or five coats that were quenched in water from 800 °C could be descaled by bending the sheet specimen slightly. (Such specimens are indicated by the notation "Descaled mechanically" in Table 1 et seq.).

TABLE 1

Results of Tests with Berkatekt No. 301 on Zirconium Alloy

Specimens air dried for 20 min after each coating

Heat Treatment	No Coat	1 Coat	2 Coats	3 Coats	4 Coats	5 Coats
700 °C for 15 min Air cooled	Dark oxide, areas of white oxide	No descaling Adherent coating	No descaling Adherent coating	No descaling Adherent coating	No descaling Small cracks in coating	No descaling Large cracks in coating
700 °C for 15 min Water quenched	Dark oxide, areas of white oxide	No descaling Adherent coating	No descaling Adherent coating	No descaling Adherent coating	No descaling Small cracks in coating	No descaling Large cracks in coating
800 °C for 15 min Air cooled	Dark and white oxide	No descaling Adherent coating	No descaling Adherent coating	No descaling Adherent coating	No descaling Cracks in coating	No descaling Cracks in coating
800 °C for 15 min Water quenched	Dark and white oxide	No descaling Adherent coating	No descaling Adherent coating	No descaling Adherent coating	Descaled mechanically Dark oxide	Descaled mechanically Dark oxide
900 °C for 15 min Air cooled	White oxide	No descaling Adherent coating	Large area descaled mechanically Dark oxide	Descaled mechanically Dark oxide	No descaling Cracks in coating	No descaling Cracks in coating
900 °C for 15 min Water quenched	White oxide	No descaling Adherent coating	Large area self-descaled Dark oxide	Self-descaled Dark oxide	Self-descaled Dark oxide	Self-descaled Dark oxide
1000 °C for 15 min Air cooled	White oxide	No descaling Adherent coating	Descaled mechanically Dark oxide	Descaled mechanically Dark oxide	Descaled mechanically Dark oxide	Descaled mechanically Dark oxide
1000 °C for 15 min Water quenched	White oxide	Large area self-descaled Dark oxide	Self-descaled Dark oxide	Self-descaled Dark oxide	Self-descaled Dark oxide	Self-descaled Dark oxide

In general, the coatings fused to a smooth glassy surface except when heat treated at 700 °C, in which case the specimens with two or three coats appeared pebbly and those with four or five coats were rough due to lack of fusion of the coating.

Table 2 gives the results of the second group of tests in which the specimens were air dried for 20 min after each coating application and then oven dried at 100 °C for 30 min after all the coats, (one, two or three) had been applied. The results here are no different from those listed in Table 1. The total coating thicknesses are given in Table 2, and the individual coats were 0.003 in. or 0.0035 in. thick.

In the third group of tests, the specimens were air dried for 30 min and oven dried at 100 °C for 30 min after each coating application. The results are given in Table 3. Again, although the Berkatekt protected the specimens from excessive oxidation, there was no self-descaling even on cooling from 850 °C. Several of the heat treatments listed in Table 3 were carried out for one hour as compared to the tests listed in Tables 1 and 2, which were all of 15 min duration. The thickness of the individual coatings varied from 0.001 in. to 0.0025 in.

TABLE 2

Results of Tests with Berkatekt No. 301 on Zirconium Alloy

Specimens air dried for 20 min after each coating, followed by oven drying at 100 °C for 30 min after all coats applied

Heat Treatment	1 Coat 0.003 in.	2 Coats 0.0065 in.	3 Coats 0.0095 in.
700 °C for 15 min Water quenched	No descaling Adherent coating	No descaling Adherent coating	No descaling Adherent coating
800 °C for 15 min Water quenched	No descaling Adherent coating	No descaling Adherent coating	No descaling Adherent coating

TABLE 3

Results of Tests with Berkatekt No. 301 on Zirconium Alloy

Specimens air dried for 30 min then oven dried at 100 °C for 30 min after each coating

Heat Treatment	1 Coat 0.0025 in.	2 Coats 0.0045 in.	5 Coats 0.007 in.
700 °C for 15 min Water quenched	No descaling Adherent coating	No descaling Adherent coating	No descaling Cracks in coating
750 °C for 1 hr Air cooled	-	No descaling Adherent coating	-
750 °C for 1 hr Water quenched	-	No descaling Adherent coating	-
800 ° C for 15 min Water quenched	No descaling Adherent coating	No descaling Adherent coating	No descaling Cracks in coating
800 °C for 1 hr Air cooled	-	Small area descaled mechanically, dark oxide	-
800 °C for 1 hr Water quenched	-	Large area descaled mechanically, dark oxide	-
850 °C for 15 min Air cooled	-	Small area descaled mechanically, dark oxide	-
850 °C for 15 min Water quenched	-	Large area descaled mechanically, dark oxide	-
850 °C for 1 hr Air cooled	-	Descaled mechanically, dark oxide	-
850 °C for 1 hr Water quenched	-	Descaled mechanically, dark oxide	-

3.2 Tests with Uranium

The uranium specimens were prepared by electropolishing in a solution comprised of the following:

1 part of a solution of 118 g chromium trioxide in 100 ml water
4 parts of acetic acid.

Berkatekt coatings No. 10 and 301 were tried on the uranium specimens. The specimens were coated by immersion and then allowed to dry unaided before the next coating was applied. The specimens were tested by heating for 15 min in air at 700 °C but in every case they were severely attacked and no protection was provided by the Berkatekt.

4. DISCUSSION OF RESULTS

The results of the tests with Berkatekt No. 301 as reported in Tables 1, 2 and 3 show that the coating protects zirconium from excessive oxidation but is not self-descaling until a heat treating temperature of 900 °C is reached and the specimen is quenched into water. The suppliers claim that Berkatekt No. 301 is self-descaling at 700 °C, and the reason for the discrepancy is not apparent. A possible reason for the failure to descale might be the relatively small size of the test specimens used.

5. CONCLUSIONS

(a) Berkatekt No. 301 protects zirconium from excessive oxidation during heat treatment.

(b) Berkatekt No. 301 is not self-descaling on small sheet specimens until a heat treating temperature of 900 °C is reached, followed by water quenching.

(c) Berkatekt No. 10 and 301 do not protect uranium from oxidation during heat treatment.

6. REFERENCES

1. "Coatings for Heat Treatment" - Metal Treatment and Drop Forging, 29(200), 206-208 (May 1962).

2. "Surface Protection During Heat Treatment" - Metallurgia, 65(391), 232-234 (May 1962).

7. APPENDIX

Semi-Quantitative Spectrographic Analysis of Berkatekt No. 10 and No. 301. (Carried out by Mineral Sciences Division, Report No. SL-62-279)

%	Si	B	Al	Mn	Fe	Mg	Ca	Zr	Ti	Na	Cu	Pb	Sb	Be	Bi	Nb
# 10	6	0.001	2	0.005	0.7	0.7	0.05	0.009	0.03	ND	ND	Tr	Tr	Tr	ND	ND
#301	10	0.009	0.06	0.006	0.3	0.1	Tr	0.2	2	ND	0.006	PC	Tr	ND	Tr	Tr

ND = not detected Tr = trace PC = principal constituent

WGH:HMS:vb