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AEC RESEARCH AND DEVELOPMENT REPORT

LOS ALAMOS SCIENTIFIC LABORATORY
OF THE UNIVERSITY OF CALIFORNIA • LOS ALAMOS NEW MEXICO

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**QUARTERLY STATUS REPORT OF THE LASL
PLASMA THERMOCOUPLE DEVELOPMENT PROGRAM
FOR PERIOD ENDING JANUARY 20, 1963
(Title Unclassified)**

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IN-PILE EXPERIMENTS

Fluted Emitter Test

An in-pile test was made of a cell with a fluted UC-ZrC fuel pin to determine the effect of increasing the electron emitter surface area while keeping the radiation area the same as in previous tests. Output data showed little improvement over the standard cell under the same test conditions. This result indicates that the cells are being operated in the plasma-limited rather than in an emission-limited mode.

Metal Pin Tests

A test has been made of a UO_2 -molybdenum cermet pin. A 0.5-mil layer of tungsten was deposited from the vapor onto the cermet to constitute the electron emission surface. The test was a duplication of a test conducted previously, except for the inclusion of a small crystal of cesium fluoride in a pocket in the collector to provide a small partial pressure of this material in the plasma. Only a limited range of cesium pressure-emitter temperature parameter spectrum could be covered by these tests. Further tests with different thermal barriers will be required to cover the entire range of parameters. The bare tungsten results are in agreement with Langmuir's data for cesium-coated tungsten. The data from the test with cesium fluoride on the tungsten are as yet too limited to permit evaluation of the influence of the fluoride.

Thousand-Hour Irradiation Tests

The second five-pin assembly subjected to in-pile irradiation for 1000 hours has been disassembled. In this test, the pins were in an atmosphere of cesium vapor at about 1 torr. Three of the five pins were from one batch

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pressing (HSR-525), made from the powder which has resulted in other pin failures. These pins were swollen and crumbled on disassembly. The other two pins in the test (HSR-501-5 and 505-1) were made from different powder. One of these, although receiving as much as one and more radiation than those mentioned above, was much less damaged. The other pin was intact and not eroded although the or alloy section had increased about 6% in diameter. The results indicate that a useful life for full-density pins of the order of 1000 hours is possible.

Low Density Pin Tests

A low-density $(\text{UC})_{0.3}-(\text{ZrC})_{0.7}$ fuel pin, made by General Atomics and having about 80% theoretical density, was used as emitter in an in-pile test. Porosity measurement of a sample of the same material indicated a ratio of open to closed porosity of about 7 to 1. The cell gave low ion and electron currents and so the test was terminated. Upon disassembly, it was found that the cell contained about 0.5 atm of gas which would explain the low currents. It is suspected that the outgassing procedures employed for low-density pin assemblies are responsible, since there was no indication of leakage of the cell during or after operation.

In future test assemblies involving porous fuel pins, either a method must be found to rid the pin of absorbed gas during assembly - something probably not accomplished by baking out at 200°C for several days - or the present cell volume of approximately 1 cm^3 must be increased to several hundred cm^3 as in the 1000-hour test geometry.

Irradiation Tests of Fuel Pins

A series of irradiation tests is being made of various fuel pin configurations. In these tests the pins are mounted in an argon-filled enclosure and operated at a predetermined location in the Omega West Reactor, so that the pin temperature corresponds closely to that in an operating cell. Omission of cesium, insulators, and instrumentation leads to a simple reliable capsule for these tests. Periodic radiographic

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inspections and complete post-radiation inspection are made on the test assemblies. Under test at present are two cermet pins of 40 v/o UO_2 loaded in molybdenum. These pins are operating at 2100°K when the Omega West Reactor is at 5 MW. A second test consists of two $(\text{UC})_{0.3}-(\text{ZrC})_{0.7}$ pins of differing densities. Both tests will be run for 1000 hours.

A 4-hour test was made to check pins fabricated from recent UC-ZrC block pressings made at LASL. One swelled and cracked in the manner observed previously, but another was not damaged. However, samples from the block showed a second metallographic phase and another sample disintegrated when held in liquid cesium at 500°C for 24 hours. Further experiments are necessary to re-establish the cesium immersion and reactor test correlation.

EXPERIMENTAL CELLS

Isothermal Plasma Cell (2000°C Cell)

The isothermal plasma cell, described in the preceding quarterly report (LAMS-2796, p. 6), has been operated extensively. The purpose so far has been to establish temperature profiles throughout the cell and to adjust cooling rates in the sapphire window end-pieces. These end-pieces must be cooled to avoid melting their brazed joints, yet at the same time they must be maintained above the cesium condensation temperature which can be as high as 400°C . This problem has been solved by using a combination of water cooling, oil cooling, and auxiliary heaters. The preliminary runs are now complete and installation of spectroscopic apparatus is proceeding. Cesium will be added after the background light that arises during vacuum cell operation is measured.

Window Cell

A plane-parallel window cell, shown in Fig. 1, has been built and operated as a vacuum diode. The cell is a completely welded and brazed enclosure heated in air rather than silicone oil. Disassembly and reassembly

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Fig. 1. Window Cell

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will be accomplished by cutting and re-welding the sheet metal flanges. The emitter is heated by electron bombardment using a commercial electron gun. The design of the cell permits the emitter-collector spacing to be varied by means of a bellows and also allows electrically isolated radiation shields or grids to be inserted. At present the emitter is tungsten sprayed on tantalum.

The first measurement to be made with this cell will be an attempt to determine the effect of radiation shields. The efficiency of the cell will be determined first without and then with radiation shields.

Cesium Fluoride Variable Spacing Cell

The variable spacing cell and vacuum system have been disassembled, cleaned, and reassembled with a new, sprayed tungsten emitter incorporated in the cell. Before assembly, the emitter was outgassed to 2000°C and later an x-ray scan of the surface was taken. The x-ray data have not yet been reduced to give the surface crystal orientations. After assembly, the entire cell was baked out to temperatures above 360°C for approximately 8 hours. The CsF reservoir was left purposely from the final assembly.

A series of vacuum emission current measurements were taken for the emitter in the temperature range from 1300°C to 1800°C . The effective work function of tungsten as determined from these measurements agreed with those of other workers, indicating that the emitter was a reasonably clean tungsten surface.

The CsF reservoir is a stainless steel capsule which is fixed to the cell system by means of a Swagelok fitting. Both the capsule and the CsF loading were vacuum baked before introducing them into the system. The cell system was flooded with dried argon at a slight pressure, and the filled CsF reservoir was fixed to the system. The CsF reservoir is located so that the gaseous CsF will flow through the emitter-collector volume towards a colder portion of the cell.

After a suitable bakeout, an attempt was made to determine the work function of the CsF-W system for various pressures of CsF in the cell,

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corresponding to CsF temperatures from 60°C to 350°C. Although work function changes were expected to be quite noticeable, the experimental results indicated no appreciable change in the work function relative to that of the plain tungsten emitter. These results tend to confirm the earlier work in which CsF was added to the Cs-W system with no change in electron emission.

It appears from ion current measurements that CsF is arriving at the emitter surface, but that some, as yet unknown, mechanism is nullifying the expected effect of the F ions at the emitter. However, to be certain that the CsF is in the emitter-collector volume, the cell is being opened to dried argon and CsF will be introduced into the cell volume directly.

MATERIALS AND FABRICATION DEVELOPMENT

UC-ZrC Preparation and Pin Fabrication

A method is being developed for producing UC-ZrC solid solution by arc melting U, Zr, and graphite as a common charge and subsequently homogenizing the cast button by heat treatment. Stoichiometric amounts of suitably sized pieces of sheet U and Zr with 1.05 times the theoretical weight of graphite, to yield (UC)_{0.18}-(ZrC)_{0.82}, were charged into a melting cavity in a fixed copper electrode. The movable electrode was 2% thoriaated tungsten. Total melt time was 30 min with button reversal after each 6 min. The UC-ZrC was crushed and the powder separated into several fractions. Compacts were made by hot-pressing and subsequently sintered for 4 h at 2050°C. The sintered products had approximately 90% of theoretical density.

Pins which were vacuum treated at 2200°C for 6 h were contaminated by furnace materials. Analysis of a partial non-adherent coating on the pins has indicated < 10% Ta, > 10% Zr, 1-3% Mo, 0.1-1.0% U, and < 0.1% W. It appears that Ta vaporized from the furnace heater to react with UC of the pin forming TaC and U which volatilized and deposited on the surface

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walls. A new resistance heater of W will be made to avoid contamination of the pin during the sintering step. This step is necessary to facilitate physical or chemical changes beyond those taking place at the 1750°C hot pressing temperature. The stability of the pin is then better assured under the proposed operating conditions.

Three pins were milled electrochemically from a 0.75-in. diam by 1.00-in. thick slug of UC-ZrC. A hollow glass electrode, 50 v/o HNO_3 solution, and anode current density of 300 A/in^2 were used. Hole diameters were 0.345 in. with pin diameters of 0.235 in. These dimensions represent ~ 54% material loss per pin.

Bonding of Fuel Pins to Metal Components

The braze area at the junction of the fuel pin to the Nb collector has been examined metallographically. There is evidence that the Vd braze metal diffuses into the D-38 end of the pin when the assembly is held at elevated temperature. Cracks which appear to follow the boundary of the diffused region with the D-38 may be the result of differential thermal expansion of the two areas. If this is the case, then a serious problem may exist in material compatibility, particularly under thermal cycling.

Ceramic-to-Metal Seals

Two tungsten metallized Al-13 (Al_2O_3) insulators brazed to Nb collectors with Pd foil continue to survive thermal cycling from room temperature to 1450°C in vacuum (~ 80 cycles).

The Al_2O_3 insulators of new design, which separate adjacent Nb collectors, have shown high leak rates. These insulators have a thinner wall than the earlier type and, although the porosity present is closed, gas diffuses through the thinner wall section. No leak-tight braze joints between insulator and collector has been made thus far with the new design.

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Coating Development

The Al_2O_3 coatings placed on the exterior of the collectors to insulate them from the outer Nb sheath have contained porosity. The coating is being treated with several thicknesses of a ceramic overspray (CMA eutectic) to seal the pores. This overspray will be fired and the composite tested for leak-tightness.

The use of a spray coat or vapor deposition coat of Nb as the outer sheath is receiving consideration. Arc-sprayed Nb fired at 1850°C for 3.5 h increased in density from 88.2% (as sprayed) to 97.4% of theoretical. It is possible that a sprayed coat will provide better thermal contact with the Al_2O_3 than the presently considered swaged tube.

Satisfactory results were not obtained with Pd-arc sprayed on Al_2O_3 or WO_3 -metallized Al_2O_3 insulators. The coat showed poor adherence. Inert atmosphere spraying and different sized powders will be tried.

Magnetic Swaging

Samples have been prepared of two concentric Nb-1 w/o Zr tubes separated by a layer of Al_2O_3 , the outer tube being magnetically swaged onto the Al_2O_3 . The thermal conductivity of these specimens will be measured to determine how effective magnetic swaging is to provide full surface contact between the Nb and Al_2O_3 .

In these specimens, the inner Nb tube represents the collector. Unless heat generated at the collector can be conducted away, a hot spot could develop and lead to melting in that region. To prevent such an occurrence, good physical contact between the three elements is essential. In order for the magnetic swaging process to form intimate contact between the Nb sheath and the Al_2O_3 insulation a considerable force is required. The shock may be severe enough to cause fracture of the insulation which might then provide a path for electrical contact between the collectors and sheath. This situation is being considered by evaluating various means of magnetic swaging of the outer sheath.

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Cermet Fuel Pins

Preliminary attempts to hot-swage sintered Mo-UO₂ bars from 5/8 in. diam to 1/4 in. diam at 1100-1200°C resulted in fracture of the rods.

A problem which exists with cermet (Mo-UO₂, W-UO₂) fuel pins is the loss of U at elevated temperatures. To curtail this loss, various coatings, such as Ta, W, and Re, have been considered. A number of tests were made in which W was used in one or more coats. The results indicate that at the proposed operating temperatures of 2000-2500°C, three to five coats are required to prevent excessive loss. Studies reported in the literature indicate that the barrier coat becomes less effective with time because of diffusion into the metal matrix of the fuel material.

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