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PROGRESS ON THE DEVELOPMENT OF
URANIUM CARBIDE-TYPE FUELS

Phase I Report on AEC
Fuel-Cycle Program

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Phase I Report on AEC Fuel-Cycle Program

Edited by Frank A. Rough and Walston Chubb

This report covers the first 6-month period of a 2-1/2-year program for the development of uranium monocarbide on the AEC Fuel-Cycle Development Program. It has been confirmed that uranium monocarbide can be prepared by reacting uranium metal powder with methane at about 650 C, and that sintered carbides of greater than 90 per cent of theoretical density can be made. The cast density of carbides has been found to vary from 13.56 g per cm³ for UC to 11.7 g per cm³ for UC₂. The electrical resistivity of cast carbides has been found to vary from about 40 microhm-cm for UC to about 90 microhm-cm for UC₂. About 500 ppm of iron or silicon has been found to decrease the quality of arc-melted castings of uranium monocarbide. Oxygen reacts with molten carbide to produce metal and carbon monoxide. Nitrogen seems to react with molten carbide to displace carbon from the monocarbide and produce some dicarbide. Diffusion rates in the carbides have been found to be appreciable at 1600 C ($D = 3.6 \times 10^{-8}$ cm² per sec) and very rapid at 1980 C ($D = 2.2 \times 10^{-6}$ cm² per sec). The activation energy for diffusion in this system is very high (92,000 cal per mole).

Additional studies are in progress to determine suitable chemical methods of preparing carbides from available raw materials, to develop techniques for making high-density compacts of carbide powder, to cast large shapes of carbide by melting techniques, to measure the mechanical and physical properties of cast uranium carbide and its alloys, to determine the chemical compatibility of carbides with various metals and reactor coolants, and to obtain information on the mechanism by which carbide-type compounds are damaged by fission fragments during neutron irradiation.

INTRODUCTION

Uranium monocarbide is being developed as a ceramic-type nuclear-reactor fuel having many potential economic advantages over uranium dioxide. Uranium monocarbide is considerably more dense than uranium dioxide and appears to have similar resistance to irradiation damage. Uranium monocarbide may be cast from the molten state, and the product has a thermal conductivity far greater than that of uranium dioxide. The tendency of the carbide to oxidize at high temperatures and to react with water prohibits its use in reactors using water or oxidizing coolants, but this property does not seriously interfere with its manufacture or handling in massive form. The present program of research is intended to remove a major obstacle to the utilization of carbide as a reactor fuel: the lack of precise production and property information concerning it.

Until very recently studies of the uranium carbides have been more or less of an academic nature⁽¹⁻¹⁹⁾. However, within the last 3 years studies have demonstrated that uranium carbides can be melted by standard arc-melting techniques⁽²⁰⁻²²⁾ and cast with suitable equipment⁽²³⁾. The existence of massive castings of uranium carbide has stimulated a tremendous interest in its properties as a potential reactor fuel material and has provided a suitable material upon which these properties can be determined with accuracy. The present research program is a logical outgrowth of the foregoing studies, and consists of research and development efforts in five related areas of interest.

(1) References at end.

For purposes of administration and reporting, the research program has been divided into three periods: 6 months, 1 year, and 1 year in consecutive order. This report covers the research performed in the initial 6-month period, designated Phase I. Research in all five areas was initiated during Phase I, and research in all five areas will continue as planned through Phase II. The five areas of research are:

- (1) An investigation of techniques and reactions by which dense bodies of uranium carbides can be prepared other than by melting techniques. This part of the program is directed toward producing more economical uranium carbide powder than can be prepared by direct reaction of uranium metal and carbon; and it is directed toward simultaneous or separate production of dense carbide bodies. It is expected that sufficient information will have been accumulated on this part of the program to conclude the research effort near the end of Phase II.
- (2) Development of equipment and techniques for casting uranium carbides of variable size and shape and of reproducible quality and alloy content. This part of the program is intended to result in further development of uranium carbide casting equipment so as to make the process more versatile and reliable. In addition, this research effort is expected to evaluate raw materials and the effects of impurities and alloy additions upon the quality of castings.
- (3) Determination of the physical and mechanical properties of uranium carbides as influenced by composition and processing variables. The effect of impurities, carbon content, alloy additions, and heat treatment will be measured in terms of suitable properties. More detailed engineering properties such as thermal conductivity, strength, thermal expansion, specific heat, thermal-shock resistance, elastic modulus, and compatibility with metals, fluids, and gaseous media are to be determined on uranium monocarbide and on any alloys of uranium monocarbide that show special promise.
- (4) Studies of the rates of self-diffusion and interdiffusion of uranium and carbon in uranium monocarbide. These studies are intended to provide a basic understanding of sintering phenomena, mechanical properties at high temperatures, precipitation phenomena, and diffusion and release of fission products from irradiated specimens.
- (5) An investigation of the mechanism of irradiation damage to uranium monocarbide. This study will involve visual, X-ray, and other physical measurements of the properties and structure of uranium carbide in an attempt to define the nature of fission-product damage and the behavior of fission products in compounds of this type.

The progress that has been made in each of these areas during the first phase of this research program is described in the following pages.

DEVELOPMENT OF ALTERNATE FABRICATION TECHNIQUES

Methods of producing high-density UC compacts by powder-metallurgy techniques as well as various methods of producing UC powder are being investigated. Methods thus far investigated include the reactions of uranium metal and uranium oxides with carbon, the reaction of UC_2 with uranium, and the reaction of uranium with methane. Also, sintering studies have been conducted on UC powder containing 4.70 and 4.85 w/o carbon obtained by crushing arc-cast buttons. The feasibility and economics of mass production as well as the quality of the UC produced are being considered.

Several possible reactions leading to the formation of UC have been considered. Since thermal data are unreliable in many cases, these equations have been used only as a guide to research. A reaction which is successful for producing fine UC powder is $U + CH_4 \rightleftharpoons UC + 2H_2$. A ΔF value of -24,600 cal has been calculated at 1000 K. A modified Sieverts apparatus has been constructed to investigate this reaction and other uranium metal-alkane reactions leading to the formation of UC. UC powder of near stoichiometric composition has been prepared by the uranium metal-methane reaction by first hydriding the uranium to obtain fine uranium powder and then reacting with methane gas at a pressure of less than 1/2 atm at a temperature of 650 to 700 C. After several charges of methane had been added with evacuation between charges, it was found beneficial to drop the temperature and hydride the unreacted uranium metal in order to maintain a loose mass so that the reaction with methane would occur freely when the temperature was again raised. The completion of the reaction was indicated when no decrease in pressure occurred as the reaction tube was allowed to cool to room temperature. If a decrease in pressure was noted, hydriding, breaking down of the hydride, and reaction with methane were repeated. Additional studies will be conducted to fully evaluate this reaction and evaluate the powder produced. Also, alkanes such as acetylene and ethane are being considered for reaction with uranium for the possible formation of UC powder.

Other reactions which appear promising for obtaining a porous sintered mass of UC are $U_3O_8 + 11C \rightleftharpoons 3UC + 8CO$ and $UO_2 + 3C \rightleftharpoons UC + 2CO$. Since the U_3O_8 probably reverts to UO_2 , the UO_2 reaction probably governs the reaction. The calculated free energy of the dioxide reaction is +16,000 calories at 1800 C. Experimental results indicate that either the thermodynamic data are incorrect or the kinetics of the reaction are such that the reaction with carbon can take place. The stoichiometric composition of UC was prepared from powders of Mallinckrodt U_3O_8 and minus 600 mesh AGOT graphite. The powders were mixed 2 hr in a V-type mixer. The mixture was compacted at 30 tsi in a powder-compacting die, resulting in a green density of 2.87 g per cm^3 . The pellet was transferred to a vacuum-induction furnace which was modified to study these high-temperature reactions. The furnace is composed of a 3-1/2-in.-diameter Vycor vacuum chamber. A 2-in.-diameter graphite susceptor is used for heating. Norblack has been used for an insulating material around the graphite susceptor; however, severe degassing of this extremely fine powder occurred on each temperature increase. Zirconia powder used in place of the Norblack was found to react with the graphite susceptor at 3000 F, even though the materials were not in contact. A tantalum susceptor is being made to replace the graphite in an attempt to eliminate reaction and degassing problems. The U_3O_8 -graphite pellet was heated slowly to 1750 C in the unit while using the graphite susceptor and Norblack insulation. The vacuum was maintained at a minimum of 25 μ . Severe degassing became evident at 1400 C. When a temperature of 1750 C was reached, it was maintained until a vacuum of 7 μ was obtained, at which time the furnace was allowed to cool. The reaction was extensive as indicated by a density increase from

2.87 g per cm^3 to 8.11 g per cm^3 . Further verification of the extensiveness of the reaction is shown by the fact that of a total carbon content of 5.94 to 6.05 w/o, only 0.04 to 0.05 w/o was found to exist as free or uncombined carbon. The reaction appears promising from the standpoint of preparing a porous sinter mass which can be consolidated by additional powder-metallurgy treatments. Additional studies are being conducted with the oxide-carbon mixtures.

The reaction $\text{UC}_2 + 2\text{U} \rightleftharpoons 2\text{UC}$ has a calculated free-energy change of 0 and -10,000 cal at 1000 and 2073 K, respectively. The stoichiometric composition of UC was prepared from UC_2 powder containing 8.74 w/o carbon and minus 325-mesh uranium powder. The powders were mixed 2 hr in a V-type mixer. A pellet of this mixture was compacted at 40 tsi, resulting in a green density of 9.11 g per cm^3 . After vacuum heating for 2 hr at 1100 C the density was 8.37 g per cm^3 , and after an additional 4-hr heating at 1100 C the density was 8.34 g per cm^3 . No changes in dimensions were noted between the 2- and 4-hr sinters; however, a growth of 4.88 per cent was noted between the green volume and the volume after the 2-hr reaction time. The density data indicate very little reaction, and an evaluation of the structure could not be made by metallographic examination. Additional studies will be conducted using X-ray and chemical-analysis techniques.

In addition, a sample compacted at 40 tsi from a mixture of 5 w/o AGOT minus 600-mesh graphite and 95 w/o minus 325-mesh uranium powders resulted in a density of 8.15 g per cm^3 . After vacuum reaction for 2 and 6 hr at 1100 C, respective densities of 7.02 and 7.00 g per cm^3 were obtained. Photomicrographs of the sample after 6 hr at 1100 C indicated little or no reaction to form UC. The purity of the uranium metal and agglomeration of the graphite during mixing are felt to be factors preventing the reaction from occurring. Additional uranium-carbon samples containing 4 to 5 w/o carbon compacted to higher green densities are being prepared for reaction studies. Investigations will be conducted using various types of uranium and graphite powders and various mixing procedures.

Other reactions being considered for investigation for the possible formation of UC are listed in Table 1 with calculated free-energy values at 1000 and 2073 K.

TABLE 1. STANDARD FREE-ENERGY CHANGE OF POSSIBLE REACTIONS YIELDING URANIUM MONOCARBIDE AT 1000 AND 2073 K

Reactions	Standard Free-Energy Change, ΔF , cal	
	1000 K	2073 K
1. $2\text{UF}_4 + 3\text{Ca} + \text{CaC}_2 \rightleftharpoons 2\text{UC} + 4\text{CaF}_2$	-283,740	-274,900
2. $2\text{UF}_6 + 5\text{Ca} + \text{CaC}_2 \rightleftharpoons 2\text{UC} + 6\text{CaF}_2$ ^(a)	-655,740	-550,900
3. $\text{UF}_4 + \text{SiC} \rightleftharpoons \text{UC} + \text{SiF}_4$	+27,100	-9,290
4. $\text{US} + \text{C} \rightleftharpoons \text{UC} + 1/2\text{S}_2$	+13,800	-7,000
5. $8\text{US} + \text{C}_8\text{H}_{18} \rightleftharpoons 8\text{UC} + 4\text{S}_2 + 9\text{H}_2$ ^(b)	-32,390	-400,000

(a) Probably the $\text{UF}_6 \rightleftharpoons \text{UF}_4 + \text{F}_2$.

(b) $\text{H}_2\text{S(g)}$ is completely dissociated at 2073 K.

In addition, samples prepared from crushed arc-cast UC buttons have been vacuum sintered for 90 min at temperatures ranging from 1730 to 2000 C. UC samples having compositions of 4.70 and 4.85 w/o carbon were sintered at each temperature. All particle sizes were minus 325 mesh. The pellets were pressed at 40 tsi after evaporating off nearly all alcohol from a camphor-alcohol solution which was placed on the powder to prevent oxidation. Table 2 shows the results of sintering for 90 min at the temperatures indicated. In addition, a 4.85 w/o carbon UC pellet pressed at 40 tsi was vacuum sintered for 30 min at 2120 C, resulting in a density of 11.7 g per cm³.

TABLE 2. DENSITIES OBTAINED AFTER VACUUM SINTERING UC PELLETS OF 4.70 AND 4.80 w/o CARBON FOR 90 MIN

Specimen(a)	Approximate Green Density, g per cm ³	Sintering Temperature, C	Sintered Density, g per cm ³
A-1	9.35	1730	9.57
B-1	9.61	1730	9.00
A-2	9.43	1800	10.41
B-2	8.93	1800	9.97
A-3	9.40	1900	11.05
B-3	9.10	1900	10.05
A-4	8.89	2000	11.03
B-4	9.15	2000	11.07

(a) "A" specimens were prepared from powder containing 4.70 w/o carbon, while "B" specimens were prepared from powder containing 4.85 w/o carbon.

In addition to the work indicated by the preceding paragraphs, additional sintering studies will be conducted on various types of UC powder to determine the sinterability as well as the activation energy.

MELTING AND CASTING

This part of the research program is concerned with the evaluation of the effects of starting materials, alloy and impurity content, and melting and casting methods on the ease of casting, surface quality and soundness, cast structure, and homogeneity of the casting. The product is to be examined primarily by nondestructive techniques such as metallographic and radiographic examination, resistivity measurements, and ultrasonic testing.

Initial investigations explored the effects of elements which might be picked up during melting, such as iron, nitrogen, oxygen, silicon, and tungsten. It was found that iron and silicon in amounts of approximately 500 ppm or greater had a detrimental effect on the quality of small shapes. Iron produced internal cracking; silicon reduced melt fluidity and increased the tendency to pipe. The others had no effect up to a concentration of 800 ppm. No decrease in ease of casting was noted for material melted in an atmosphere contaminated with hydrogen.

A study of the effect of carbide-forming alloy additions is now in progress. Additions of 1, 10, and 50 mole per cent of Al_4C , Cr_3C_2 , Mn_3C , Mo_2C , NbC , TiC , TaC , VC , and ZrC are being made to UC, and the alloys are being cast into cylindrical specimens 3/8 in. in diameter and 1-3/4 in. long.

Melting and casting procedures for the preparation of as-cast shapes have been developed previously for production of castings in sizes up to 5/8 in. in diameter and 2-1/2 in. long. Carbon content can be controlled to 4.8 ± 0.1 w/o carbon. Preparations for melting and casting of uranium carbide by skull arc-melting techniques to larger sizes and to varied shapes have been initiated. It is planned to melt uranium carbide in a skull of the same material and cast into graphite molds.

MECHANICAL AND PHYSICAL PROPERTIES OF CARBIDE MATERIALS

A study of the properties of uranium monocarbide has been undertaken to describe the effects of composition and heat treatment on the alloy as an engineering material. The study is related to product quality from the point of view of controlling and improving the mechanical and chemical properties of the alloy. Mechanical properties, such as rupture strength, hardness, and modulus, and physical properties, such as thermal conductivity, electrical resistivity, density, and specific heat, and corrosion resistance and compatibility are being surveyed as functions of carbon content, alloy content, and heat treatment.

Hydrogen, iron, nitrogen, oxygen, silicon, and tungsten were selected as the contaminants most likely to be introduced into the alloy unintentionally from the atmosphere, molds, or electrode tips. Uranium monocarbide test specimens 3/8 in. in diameter and 1-3/4 in. long containing these impurities in amounts of 100 to 800 ppm were prepared by inert-electrode arc melting. Oxygen was added as UO_2 , nitrogen as N_2 gas and as UN, hydrogen as a gas mixed with the normal helium atmosphere, and the others as metals. Densities, electrical resistivities, transverse rupture strength in three-point loading, microstructure, and corrosion resistance in 60 C water were examined in an attempt to evaluate the effects of these impurities. The tests disclosed that cast UC specimens of the stoichiometric composition (4.8 w/o carbon) containing impurities were stronger (average transverse rupture strength of 15,000 psi) than specimens with the same impurities that contained 5 w/o carbon (average transverse rupture strength of 10,000 psi). Neither carbon nor impurity content had an appreciable effect on electrical resistivity. A specimen containing 4.8 w/o carbon and no impurities had a corrosion rate in 60 C water of $600 \text{ mg}/(\text{cm}^2)(\text{hr})$, while a similar specimen containing 5.0 w/o carbon had a corrosion rate of $1000 \text{ mg}/(\text{cm}^2)(\text{hr})$. The impurity additions had little effect on corrosion rates with the exception of hydrogen. A threefold decrease in corrosion rate was observed for a specimen melted under a hydrogen-contaminated atmosphere. The actual hydrogen content of this specimen is unknown.

Metallographic examination of these alloys showed that excess carbon (over 4.8 w/o) tends to promote microcracking of specimens, as does 800 ppm iron present as an impurity. A UO_2 addition to a melt charge containing 4.8 w/o carbon produces free uranium in the cast alloy, presumably by the reaction $2\text{UC} + \text{UO}_2 \rightarrow 3\text{U} + 2\text{CO}$. Uranium mononitride additions to uranium monocarbide did not alter the appearance of the alloy; the phase formed is believed to be $\text{U}(\text{C}, \text{N})$ solid solution.

Specimens of uranium-carbon alloys containing additions of up to 3 w/o hydrogen, nitrogen, and oxygen have been prepared. Metallographic observations of these alloys have confirmed the above-mentioned effects of nitrogen and oxygen additions. Specimens containing 1 to 3 w/o nitrogen, having microstructures showing a U(C, N) matrix containing UC_2 , exhibited resistivities of about 60 microhm-cm. Oxygen and hydrogen additions had no noticeable effect on resistivity or density.

The effect of carbon content on the density and electrical resistivity of a series of uranium-carbon alloys is shown in Figures 1 and 2. Three-point loading tests on single specimens of these uranium-carbon alloys produced transverse rupture strengths ranging from 15,000 psi for specimens containing 4.5 to 7.0 w/o carbon to 8,000 psi for a specimen containing 8.75 w/o carbon.

Attempts to alloy UC with other carbides are now in progress. Alloys of UC containing 1, 10, and 50 a/o Al_4C , Cr_3C , Mn_3C , Mo_2C , NbC , TaC , TiC , UC, and ZrC are being melted and cast into cylindrical specimens.

Compatibility tests on uranium-4.8 w/o carbon and uranium-5.0 w/o carbon alloys are in progress. Carbide samples are being sealed in tubes of various metals. These will be hot swaged and then heated to various temperatures for testing. Aluminum and magnesium capsules will be tested at 600 C, copper at 1000 C, and Inconel, mild steel, stainless steel, and zirconium at both 1000 and 1200 C. Thermal-conductivity measurements, specific-heat measurements, and modulus and compressive-strength tests will be made as soon as large-size specimens are available. Specimens are being prepared for corrosion tests in NaK, terphenyl, other organics, and water.

DIFFUSION STUDIES OF URANIUM MONOCARBIDE

Diffusion-rate measurements are being made on uranium monocarbide in an effort to provide a basic understanding of phenomena occurring in this material at high temperatures. Sintering, relaxation of stresses, precipitation phenomena, environmental reactions, and diffusion of fission products are some of the processes which can be understood better in the presence of diffusion-rate data. It was intended that data would be obtained on the rate of interdiffusion of uranium and carbon in uranium carbides, the rate of self-diffusion of uranium in uranium monocarbide, and the rate of self-diffusion of carbon in uranium monocarbide. However, it appears that the technical problems involved in sampling uranium monocarbide for carbon in milligram quantities may prevent the determination of the rates of self-diffusion of carbon.

Measurements of the rate of interdiffusion of uranium and carbon in the system uranium monocarbide-uranium dicarbide are in progress, and most of the results are available now. These measurements are based on the rate of growth of the carbide layers formed between graphite and liquid uranium saturated with carbon. Strictly speaking, the method is precise only if a single carbide is formed between the graphite and the liquid or if the rate of diffusion is the same in all the layers formed. The initial tests showed that two layers (corresponding to the monocarbide and the dicarbide) were formed at all temperatures from 1800 to above 2000 C. In addition, the data showed that diffusion rates above 2000 C were too rapid in this system for practical determination of rates at such temperatures.

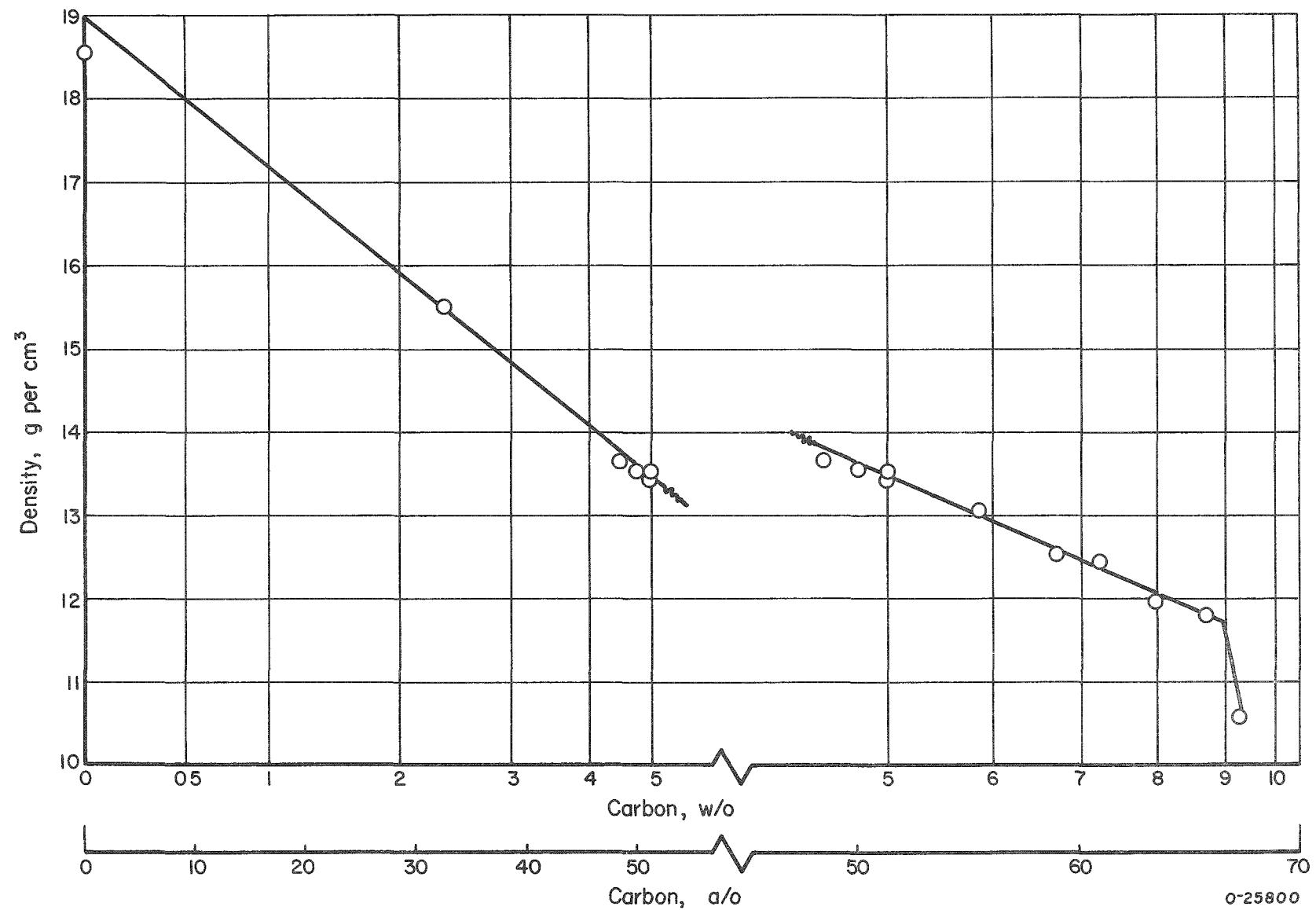
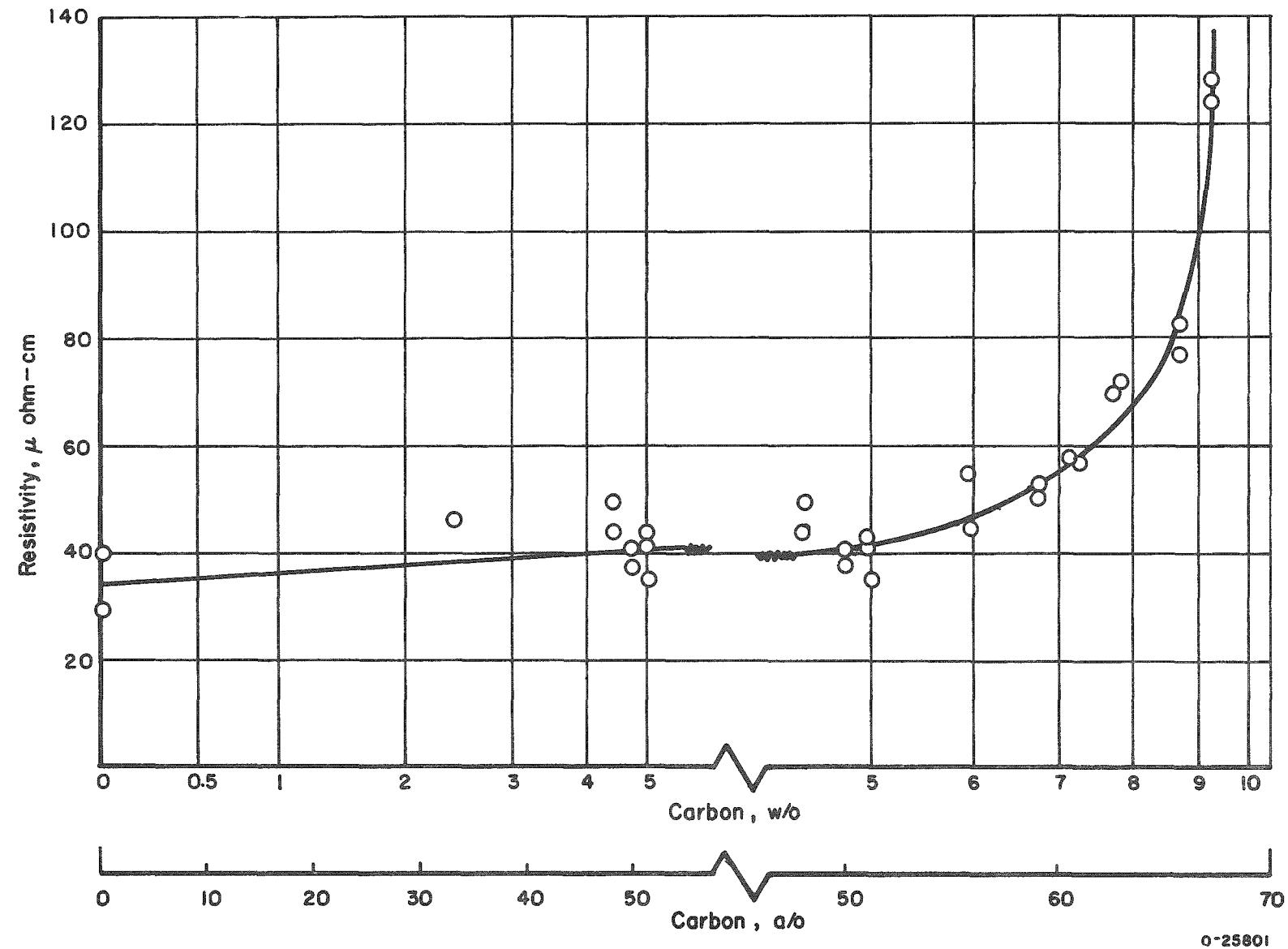


FIGURE 1. DENSITY OF AS-CAST URANIUM-CARBON ALLOYS



The results obtained on a number of such graphite-liquid uranium couples indicate that the layers formed by diffusion grow in thickness approximately as a function of the square root of time, as required by theory. However, it is clear also that diffusion is somewhat slower in uranium dicarbide at 2000 C than in uranium monocarbide. For example, in 101 min at 1980 C a diffusion zone 0.057 in. thick was formed consisting of a 0.017-in.-thick layer of dicarbide and a 0.040-in.-thick layer of monocarbide. In the absence of a suitable method for analyzing these data as separate parts, it must be assumed that diffusion rates in the two compounds are the same. It follows that the diffusion coefficients shown in the following tabulation and in Figure 3 represent some sort of an average of a somewhat higher diffusion rate in uranium monocarbide and a slightly lower diffusion rate in uranium dicarbide:

Temperature, C	Interdiffusion Coefficient, D, cm ² per sec
1600	3.6×10^{-8}
1800	2.3×10^{-7}
1800	3.3×10^{-7}
1900	1.6×10^{-6}
1980	2.2×10^{-6}

As shown in Figure 3, these results appear to fall very nearly on a straight line when the logarithm of D is plotted versus the reciprocal of temperature. The equation for this line is:

$$D = 1770 e^{-92,000/RT},$$

where 92,000 cal per mole is the activation energy for interdiffusion in this duplex, UC-plus-UC₂, system. This value appears to be reasonable for a system such as this one involving solvents having very high melting points.

Experimental techniques for determining rates of self-diffusion of uranium in uranium monocarbide are being developed concurrently with the above studies. It is expected that a thin foil of enriched uranium will be bonded between two pieces of depleted uranium carbide, diffusion annealed, sectioned, and analyzed by an activation technique. It has been found possible to prepare short cylinders of carbide from long castings using either a diamond cutoff wheel or an Alundum cutoff wheel and a kerosene coolant. It has also been found possible to bond two such short cylinders together with a uranium metal foil between them.

Future work will include a few additional measurements of sections from the interdiffusion melts, further development and actual application of techniques for measuring rates of self-diffusion of uranium in uranium monocarbide, and a minor effort to find a suitable technique for measuring the rate of self-diffusion of carbon in uranium monocarbide.

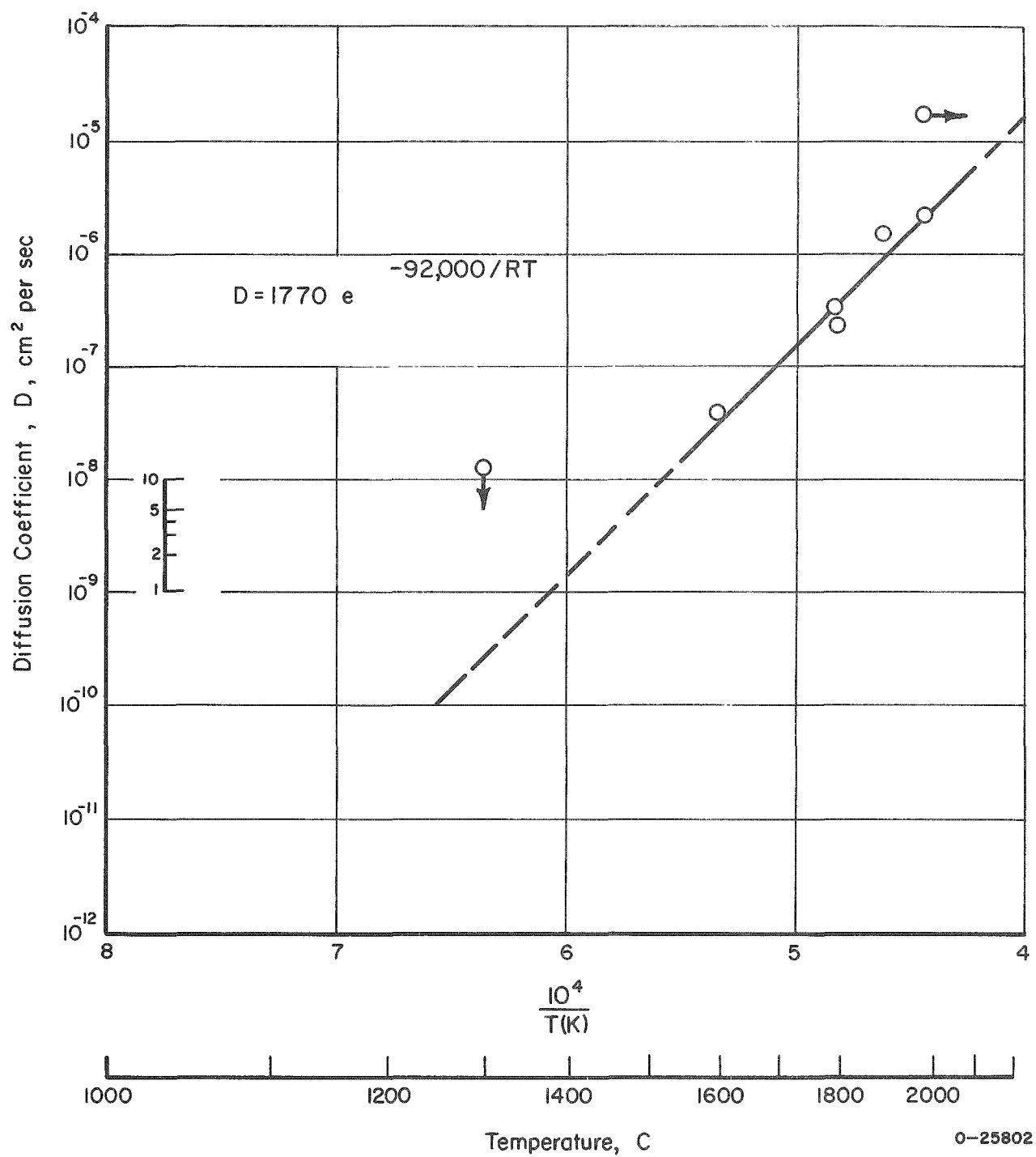


FIGURE 3. INTERDIFFUSION RATES IN THE UC-UC₂ SYSTEM

MECHANISM OF IRRADIATION DAMAGE

Uranium dicarbide, uranium sesquicarbide, and uranium monocarbide are different in their atomic bonding and physical properties compared with metals and oxides. Therefore, a basic knowledge of the effects of neutron radiation and of fission is needed to guide the fabrication and use of carbides. Information to be sought includes the extent of crystal strain, incidence of fragmentation, diffusion of fission products causing bubble formation and accelerated swelling, and loss of fission gases.

Uranium monocarbide has a strong covalent type of bonding between uranium and carbon atoms with relatively weak carbon-carbon and uranium-uranium bonds. However, there are free electrons available to give metallike electrical and thermal conductivities. Therefore, the carbides partake of some of the properties of both metals and refractory compounds.

The dependence of irradiation effects upon temperature may be similar to those of other refractory compounds. Thus, at low temperature the diffusion of fission products and interstitials may be quite slow, with crystal lattice strain increasing and fracturing occurring. However, at high temperatures there will be appreciable diffusion and possible relief of strain. Therefore, some accumulation of fission gases into bubbles leading to swelling may be expected.

The type of measurements which will yield the desired basic information on structural and physical properties have been investigated, and some conclusions can be made as follows: Normal hot-cell measurements of physical dimensions and density are needed for over-all correlation. Crystal-lattice expansion, strain, and fragmentation require analysis of powder X-ray diffraction data. The incidence of cracking and bubble formation can be followed by light and electron microscopy. The nucleation of clusters or gas bubbles may possibly be detected by low-angle X-ray scattering.

X-ray diffraction measurements on irradiated fuel materials require extensive care in specimen preparation and in elimination of the effects of specimen radiation. Such measurements can be obtained with diffractometer equipment by use of local specimen shielding, plus a crystal monochromator path for proportional counting with electronic discrimination circuits. It has been estimated that specimens having activities of 0.2 to 0.5 curie can be handled. This places a severe limitation on specimen size and burnup. The grain size of arc-cast carbide is too large for powder X-ray diffractometry. Therefore, it is necessary to prepare powder by crushing. Powder crushed to less than 325 mesh is adequate for giving X-ray diffraction lines suitable for measuring line contours for crystal strain, expansion, and size. The limitation on specimen activity requires an upper limit of about 25 mg of powder having a burnup of 0.5 a/o of uranium. Methods for mounting such specimens have been worked out. Special specimen holders have been designed for this purpose.

The observation of cracks and bubbles in irradiated material may be done by direct microscopy and by electron microscopical examination of replicas. The reactivity of uranium carbide with water requires additional care in metallographic preparation and replication. Suitable techniques have been developed with unirradiated materials. For these a dry atmosphere is necessary, and the application of the technique to irradiated specimens in the hot cell will be tried.

The accumulation of gaseous fission products into nuclei and the growth of bubbles can be determined in theory by means of low-angle X-ray scattering. The size and concentration of such scattering centers can be determined from the angular distribution of scattered monochromatic X-rays. Because of the high absorption of X-rays by uranium, specimens with sufficient transmission need to be quite thin, about 8μ for UC, and, therefore, are quite fragile and difficult to make. However, the thickness may be increased several fold by diluting the uranium concentration. Both UC and ZrC have the same crystal structure and they form a continuous solid solution. For a mixed carbide containing 80 w/o ZrC and 20 w/o UC, the thickness of specimen could be 50μ . Such a specimen appears feasible to prepare by thin-section lapping techniques. To test this procedure, alloys of nominal 90 w/o ZrC-10 w/o UC, 80 w/o ZrC-20 w/o UC, and 60 w/o ZrC-40 w/o UC were arc melted. The alloy of 80 w/o ZrC-20 w/o UC did not arc melt to give a usable button. The other alloys gave sufficiently large buttons. These were two-phase alloys, containing face-centered-cubic (Zr, U)C and body-centered-cubic (Zr, U) metal. The alloys were very hard and, therefore, difficult to cut and lap, requiring Alundum or diamond abrasives. However, the results appear sufficiently encouraging that further attempts will be made to produce a single-phase solid-solution carbide.

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