PILOT PLANT PROCESSING OF URANIUM-ZIRCONIUM ALLOY NUCLEAR FUELS BY A MOLTEN-SALT FLUORIDE-VOLATILITY METHOD

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Introduction

Nuclear reactor fue's are fissionable isotopes (uranium-235, uranium-233, or plutonium-239) that split to form other elements (fission products). These fission products are highly radioactive, and many of them capture neutrons that would otherwise be available to sustain the nuclear chain reaction. These two properties dictate the basic requirements for chemical processing of spent nuclear fuels. That is to say, the recovery of uranium from the fuel elements must be done behind walls thick enough to shield the workers from radiation, and the recovered fuel must be sufficiently free of fission products which capture neutrons (neutron poisons) that the fission process can be repeated in the new fuel element. Further, the new fuel element should, in the interest of simplicity, be suitable for direct handling.

A nuclear reactor is normally designed to operate with a range of concentrations of these neutron poisons. However, this range would usually be exceeded before all fissionable material is burned; hence, in order to be economical, the "unburned" fissionable material must be recovered for reuse in new fuel elements.

In recovering the fuel, or fissionable material (e.g., uranium), it must not only be separated from the major neutron poisons, but should if possible be separated from the other radioactive fission products, even though they do not rank very high as neutron absorbers. It is thus feasible to reuse fuel which has had some of the neutron poisons removed but which is still highly radioactive, although this requires expensive remote handling when the new fuel elements are made.

It is the purpose of this brief account to present an introduction to nuclear fuel processing and to point out the important aspects of a recent and useful method of processing. This method involves reactions with hydrogen fluoride and with fluorine, volatilization of uranium hexafluoride, and purification of the hexafluoride by selective sorption and desorption.

There are many processes for separating and recovering fissionable materials from fission products. Some have been abandoned; some are still used; and some are being developed. As with all technologies, there was a beginning. First was the bismuth phosphate precipitation process, used to recover plutonium for the first plutonium bombs. However, the uranium was not recovered and remained in solution with the fission products. Then, shortly after World War II, solvent extraction replaced that process. Even now, almost all commercial-scale units for fuel recovery are based on one of the many solvent extraction processes.

Although differing in detail in order to achieve specific goals, basically, these processes utilize an aqueous and an organic stream flowing counter-currently. The fuel is dissolved in an aqueous solution, such as nitric acid. For each chemical element in the fuel (fission products, uranium, etc.), the relative solubilities in the two streams (distribution coefficients) control its concentration in one stream and its depletion from the other. By proper selection of conditions, highly purified uranium can be obtained.

However, several nonaqueous processes are now in various stages of development. The major ones are the volatility and the pyrometallurgical. The pyrometallurgical methods, using procedures adapted from the metallurgical industry, are aimed primarily at removing enough of the neutron poisons to permit reuse of the fuel. This partial decontamination leaves enough radioactivity behind that remote fabrication is required. However, volatility processes produce more nearly pure uranium by a variety of methods, all relying on the relatively high vapor pressures of uranium and plutonium hexafluoride. For example, uranium hexafluoride can be volatilized from a fluoride melt or from a fluidized bed in which the fuel material is supported by powdered alumina. Additional procedures used in various volatility processes include distillation, sorption-desorption, and condensing in a cold trap. The volatility process being developed at Oak Ridge National Laboratory (ORNL) is distinguished from others by the fact that it involves dissolution in and volatilization from molten fluorides.

The ORNL molten-salt fluoride-volatility process was originally aimed at the recovery of uranium from the fuel for the Aircraft Reactor Experiment. The fuel consisted of molten fluorides of uranium, sodium, and zirconium, appropriately proportioned to melt at about 525°C. A pilot plant was built, and two complete reactor charges were successfully processed [1], [2] by passing fluorine through the melt, forming the volatile hexafluoride of uranium, plus volatile fluorides of some of the fission products. These fluorides were absorbed on beds of sodium fluoride pellets and then separated by selective desorption, a method also used in our present process.

Laboratory studies indicated that the process was particularly well adapted to the separation of uranium from spent uranium-zirconium alloy reactor fuels [2], and this was confirmed by further laboratory studies [3]. Basically, the present process starts with dissolution of uranium-zirconium alloy fuel elements in a molten fluoride solvent with anhydrous hydrogen fluoride. Fluorine is then brought into contact with

the melt, volatilizing uranium hexafluoride plus some volatile fission product fluorides. The uranium hexafluoride is further purified in a sorption-desorption cycle by use of a bed of sodium fluoride pellets and another bed of magnesium fluoride pellets. The desorbed uranium hexafluoride is then collected in a cold trap prior to transfer to shipping containers.

What follows is presented in the following sequence: First, the chemical reactions will be taken up in the order in which they take place in essential components in the pilot plant, beginning with the dissolver. Then the pilot plant flowsheet and the components themselves are discussed in the same order. Equipment design details are not included, but a few of the techniques used are of general interest; hence the flowsheet description is followed by a brief description of some of these interesting design features. The results of recent pilot plant studies are presented, and finally the main advantages and future prospects of the process are summarized.

PROCESS CHEMISTRY

The basic chemistry of the molten-salt fluoridevolatility process is fairly simple, since there are very few chemical reactions involved in the "main stream" of the process. (Only the chemistry involved in the process is discussed in this section; discussion of flow patterns and equipment is deferred to the next section, in which the pilot plant flowsheet is described.)

Dissolution of the Fuel

Dissolution of the alloy in the molten fluoride solvent utilizes the reactions:

$$Zr + 4HF \rightarrow ZrF_1 + 2H_2 \tag{1}$$

and

$$U + 4HF \rightarrow UF_4 + 2H_2. \tag{2}$$

Both tetrafluorides are fairly soluble in the molten LiF-NaF-ZrF₄, sometimes called the barren salt. The phase diagram for this particular fluoride salt system [6] is shown in Fig. 1. The phase diagram does not include uranium tetrafluoride (UF₄), but experience

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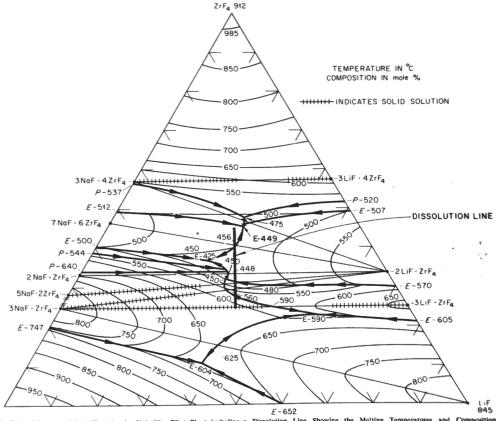


Fig. 1. Phase Diagram of Salt Used in the Volatility Pilot Plant including a Dissolution Line Showing the Melting Temperatures and Composition Changes (from 37.5-37.5-25 to 27.5-27.5-45 mole % LiF-NaF-ZrF4) as Zirconium is Converted to Zirconium Tetrafluoride and Dissolved.

indicates that the addition of UF4 under normal operating conditions results only in a slight depression

of the melting point of the mixed salt. If dissolution were to be initiated in LiF-NaF, omitting the ZrF4 from the barren salt, inconveniently high temperatures would be required. Hence, the barren salt charged for dissolution is 37.5-37.5-25 mole % LiF-NaF-ZrF4. Dissolution results in a final concentration (exclusive of the UF4 content) of 27.5-27.5-45 mole %. This change in composition is shown as a dissolution line on the phase diagram. As dissolution proceeds, the melting point of the salt rapidly drops from slightly above 600°C to about 450°C, the result of adding more zirconium tetrafluoride to the melt as the zirconium in the alloy is converted to the tetrafluoride by the HF that is bubbled through the salt. [See reaction (1).] This reduction in melting point permits a reduction in operating temperature during dissolution and results in a salt whose melting point is close to the minimum melting point for all subsequent operations.

Volatilization of Uranium Hexafluoride

Having dissolved the uranium-zirconium alloy in the melt, uranium is removed by converting it to the volatile hexafluoride, using the reaction $UF_4 + F_2 \rightarrow UF_6$. The fluorine is merely bubbled through the melt. The uranium hexafluoride (UF₆), with its vapor pressure of 1 atmosphere at about 55°C, is volatilized, leaving more than 99% of the fission product activity behind. However, some of the fluorides have an even higher vapor pressure than UF, and thus are volatilized along with it. Other contaminants are carried in the gas stream in only trace quantities, but even traces are unacceptable. For example, the vapor pressure of ZrF4 at the fluorination temperature (500° C) is less than 1 mm of Hg, but even the small quantity of fission product zirconium which is evolved during fluorination would prohibit direct handling of the UF6. Hence, the UF6 stream leaving the fluorinator must be purified, as described next.

Purification of the Uranium Hexafluoride

The purification of the uranium hexafluoride is done by sorption and desorption, as follows: The impure UF6 stream from the fluorination step is passed first through a bed of sodium fluoride pellets held at 400°C. At 400°C, UF₆ is not sorbed, but the fluorides of some of the corrosion products (such as chromium) and some of the fission products (such as niobium and ruthenium) are sorbed to varying degrees by the NaF.

The partially purified UF6 stream next enters a bed of NaF pellets held at 100°C. At this temperature, the UF, is sorbed by the NaF [4]. Some of the remaining contaminants, such as zirconium and molybdenum fluorides, are sorbed along with the UF₆, while others, such as tellurium, pass through as a part of the waste stream. Thus, the UF, is collected and is fairly pure, but still not pure enough.

The sodium fluoride bed containing the UF6 is then heated to 150°C and held for two hours while fluorine is passed through the bed. Most of the molybdenum

hexafluoride is desorbed from the bed (and sent to hexafluoride is described an anount of waste), while only a negligible amount of uranium waste), while this accompanies it and is lost, though it can be recovered accompanies it and very little UF₆ leaves at 150°C because it is in the form of a fairly heat-stable complex of NaF and UF₆, thus permitting it to remain behind while the molybdenum is being removed.

The sodium-uranium fluoride complex is completely dissociated at 1 atmosphere at 363°C [4]. Hence, the next step involves heating the bed to 400°C while "sweeping" the bed with fluorine to prevent reduction of the UF₆ to UF₅. This treatment removed UF₆ leaving behind contaminants such as ZrF₄. The UF₆ product finally passes through a bed of magnesium fluoride pellets at 125°C for preferential sorption of technetium and neptunium, while the UF₆ flows to a cold trap for final collection of the purified product

DESCRIPTION OF THE VOLATILITY PILOT PLANT

As mentioned earlier, a pilot plant was built to recover the Aircraft Reactor Experiment fuel [1]. This pilot plant was subsequently modified to permit processing high-burnup, short-cooled U-Zr alloy fuel elements [5]. ("Cooling" the fuel is just another way of saying, "Let the short-living radioisotopes decay and thereby make the fuel element less radioactive.") The details of the reactions that take place in the components of the system (dissolver, sodium fluoride beds. etc.) were discussed earlier in "Process Chemistry." In the pilot plant, specialized vessels were designed for these reactions. Briefly, the major vessels through which the uranium passes from the fuel element carriercharger to the product receiver (which is also the shipping cylinder) are, in order: dissolver, fluorinator. movable-bed absorber (NaF pellets), impurity trap (MgF₂ pellets), and product receiver (or cold traps). The pilot plant is described in more detail below.

Flowsheet

The pilot plant flowsheet is shown in Fig. 2. Although construction materials (primarily nickel and nickel alloys) and design details are not included, this schematic diagram does show the shapes and relative elevations of all major vessels.

Dissolution System—Fuel elements are lowered into the small (5-in. diameter) bottom section of the dissolver. Molten barren salt is added until the fuel elements are completely submerged. Anhydrous HF is then circulated through the system for 16 to 24 hours, converting the zirconium and uranium to the tetrafluorides. Dissolver off-gas, containing hydrogen, unreacted HF, and inert gas used for purges, entrains 300 to 500 grams of submicron-size particles per run. These particles are about evenly divided between salt and unfluorinated metal. They are highly radioactive and if allowed to remain in the gas stream would quickly plug the first valve they reach. Several methods of removal, such as filtration, were tried. The problem was reduced but not eliminated until the scrubbing system shown in the flowsheet was installed. The offgas from the dissolver first enters the flash cooler, into

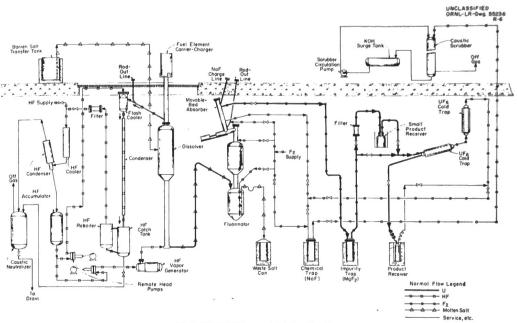


Fig. 2. Schematic Diagram of Volatility Pilot Plant

which liquid HF is being pumped. The gas stream is cooled to 20°C by vaporization of part of the liquid HF. The remaining liquid supplies a washing action. Additional scrubbing is achieved as HF is liquefied in the condenser. The particles settle in the bottom of the catch tank, and HF is vaporized in the reboiler for recirculation to the dissolver. Any water resulting from hydrofluorination of oxides is also retained in the catch tank. At the end of the run, the impurities are flushed out by draining the HF from the catch tank to the caustic neutralizer.

The off-gas leaving the HF reboiler passes through a filter used principally to determine the effectiveness of the scrubbing system. The HF is condensed and recirculated as a liquid to the flash cooler and to the HF vapor generator (which supplies HF to the dissolver). Noncondensibles in the off-gas stream are bubbled through potassium hydroxide solution to remove traces of HF before entering the main off-gas line.

Plant Off-Gas System-The main off-gas line collects all off-gas from the plant, including the noncondensibles discussed above, off-gas from the fluorination system, off-gas from the purification system, and cellventilation air. The main off-gas system includes an auxiliary scrubber (not shown) to protect the filters at the off-gas stack from accidental releases of fluorine or hydrogen fluoride.

Fluorination System-After dissolution of the fuel alloy is complete, the salt is transferred to the fluorinator. Bubbling fluorine through the salt for two hours results in uranium being removed as the volatile compound, UFa. The waste salt remaining is subsequently transferred to a waste can where it is allowed to freeze. The can is then buried in concrete. Thus the highactivity waste from the process is permanently disposed of in a relatively small volume, a prime virtue of the volatility process.

47

UF6 Purification System-The UF6 stream leaving the fluorinator enters the movable-bed absorber, a vessel that combines the functions of three sodium fluoride beds used during early pilot plant work. Pellets in the lower portion of the absorber are held at 400°C. During fluorination and molybdenum stripping, the waste-gas stream leaves the absorber through the outlet near the middle of the vessel. During these two stages of operation, the pellets just below the outlet are held at 100 and 150°C, respectively.

For desorption, the entire bed is heated to 400°C. and the product stream is carried out through the line near the top of the absorber. After desorption is complete, the NaF pellets at the bottom of the bed are discharged into the waste salt in the fluorinator, and fresh pellets are charged to the top of the bed.

The UF₆ stream leaving the absorber goes through the impurity trap, which contains magnesium fluoride pellets. Removal of technetium and neptunium fluorides from a stream of uranium hexafluoride by magnesium fluoride was pioneered at the Paducah Gaseous Diffusion Plant, and the design of the impurity trap was based on development work at Paducah.

The product stream is filtered to remove fines resulting from the powdering of the pellets. The filter consists of nickel mesh followed by a porous, sintered nickel plate.

Product Recovery System-For the relatively small quantities of uranium (less than a kilogram) in the current fuel charges, a small product receiver was installed in the system. This small metal bottle, immersed in dry ice-trichlorethylene, acts as a cold trap and also permits removal of the product for analysis after each run. At the end of a series of runs, product collected in these small receivers can be transferred to the larger UF6 cold traps and then to a shipping cylinder (shown as the product receiver at the bo'tom of the flowsheet).

Chemical Trap System-All off-gas from the fluorination system, the UF6 purification system, and the product recovery system goes through a chemical trap, which is a bed of NaF pellets at room temperature. This trap removes any trace quantities of UF₆ before the off-gas is discharged to the caustic scrubber.

Interesting Design Features

Some of the design features of the Volatility Pilot Plant are of interest in that they represent techniques not in general use but holding a potential for widespread application. The features discussed below are: heating of salt-transfer lines, "valves" for salt-transfer lines, and control of leakage through the flanges.

Autoresistance Heating-Salt-transfer lines are made of metal that has a relatively low electrical conductivity, such as Inconel. The transfer line is then made a part of a low-potential, high-current circuit (e.g., 7v, 135 amp). Thus the pipe line also becomes the heating element and does not require cumbersome external heaters

Freeze Valves-Conventional valves for molten salt service which are both cheap and reliable are nonexistent. This problem was solved in the Volatility Pilot Plant by deliberately including a nondraining low point in the salt-transfer lines. After a transfer is complete, the small quantity of salt remaining at the low point is allowed to freeze, effectively plugging the line. To open the "valve", the line is heated until the salt melts.

Flange Buffer System-Ring-joint flanges are used in process gas lines. Ring-joint flanges are joined by a ring (usually a soft metal such as copper) which fits into a groove in each flange face. The ring keeps the flange faces separated, so that tightening the bolts seals the joint by compressing the ring between the flanges. The ring is thicker than the width of the groove, so that it does not reach the bottom of either groove. The two lines of ring contact at the sides of each groove form the seal. Inside each groove, between these contact lines, is a "dead" space, sealed from the process fluids and the atmosphere. A connection is made from this dead space to a manifold system, and pressurized inert gas is maintained in the groove. By drilling a hole through the ring to connect

both grooves of a flange pair, a gas buffer is maintained on the entire joint by a single connection. In the on the entire joint of the event that a leak develops, the leakage is inert leavent that a leak develops, the leakage is inert leavent that a leak develops, the leakage is inert leavent that a leak develops, the leakage is inert leavent that a leak develops, the leakage is inert leavent that a leak develops, the leakage is inert leavent that a leak develops, the leakage is inert leavent that a leak develops, the leakage is inert leavent that a leak develops, the leakage is inert leavent that a leak develops is the leakage is inert leavent that a leak develops is the leakage is inert leavent that a leak develops is the leakage is inert leavent that a leak develops is the leakage is inert leavent that a leak develops is the leakage is inert leavent l event that a reas gas escaping or moist air leaking rather than process gas escaping or moist air leaking rather than process grant proper manifolding and measure in. In addition, with proper manifolding and measure. ment, the flange pair that is leaking can be identified and the rate of leakage can be measured

RESULTS OF PILOT PLANT OPERATIONS

A pilot-plant-scale uranium-zirconium alloy program was successfully completed. The two primary objectives, demonstration of continued operability and achievement of adequate decontamination from fission products, were reached. Reactor fuel cooled as short a time as six months was processed; some of the decontamination factors achieved are the highest that have ever been reported for a single-cycle radiochemical process.

In radiochemical processing, the degree of purification is often expressed as a decontamination factor, or DF which is defined as the ratio of any particular activity in the feed to the corresponding activity in the product Thus, a strontium-90 DF of 100 would mean that 1% of the original Sr⁹⁰ accompanied the product, and a cesium-137 DF of 5 x 104 would mean that purification from Cs137 was 99.998% complete. In each of the last four runs in the Volatility Pilot Plant, decontamination factors for some of the less volatile fission products were greater than 1010.

ADVANTAGES AND FUTURE PROSPECTS OF THE PROCESS

Volatility Pilot Plant operations have confirmed that two of the advantages of the ORNL volatility process, compared with conventional aqueous processes, are: Significantly lower volumes of high-activity wastes are produced, and extremely good decontamination is achieved.

Laboratory studies showed that this molten-salt fluoride-volatility process is applicable to other enriched-uranium fuels such as oxide fuels and aluminum alloy fuels. Hence, the next pilot plant program will be to study the processing of such fuels. Meanwhile, the feasibility of recovering uranium from highly enriched uranium-zirconium alloy fuels by this method has been demonstrated, with excellent results.

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NEWS OF TENNESSEE SCIENCE

(Continued from Page 43)

following a week's journey from Longview, Tex., where it was fabricated. It was unloaded onto a lowboy trailer and transported to the ORINS Medical Division.

In addition to ensuring that the steel, concrete, and surrounding olivine ore have low radioactivity levels, all construction materials within six feet of the counting chamber have been tested for background content. Those materials showing a background too high for this use have been rejected and other less active materials have been substituted.

When the completed facility is in operation, patients will lie in a horizontal sling and be transported into the counting chamber through a door in the front wall. A heavy steel door will be closed, and the counting procedure will begin. Gamma rays emitted by radioactive materials in the patient's body will be detected by eight large scintillation crystals arranged above and below the supporting sling.

Conceptual design of the whole-body counter was begun by Dr. Marshall Brucer, former chairman of the Institute's Medical Division, and actual design of the facility was carried out by Dr. Douglas A. Ross, a former member of the Division now at Oak Ridge National Laboratory, and A. C. Morris, Jr., associate scientist with the Medical Division. The design was done by the Medical Division in cooperation with the Engineering Dvison of the AEC Oak Ridge Operations over the past five years.

The fifteenth annual Fisk University Infrared Spectroscopy and Gas Chromatography Institute will be held August 11-15, 1964, at Fisk University, Nashville, Tennessee.

The sessions on basic gas chromatography and the fundamentals in infrared spectroscopy will be conducted during the same five-day period. The lectures for the two programs will be so arranged that persons enrolled in either program may, if they so desire, audit the general lectures of the other program without extra charge. A second infrared session is scheduled for August 17-21.

Announcement of the sessions and other information · are given on the inside back cover of this magazine.