LAP-AC-6889 (Full Paper)

CONF-650509-~~20~~²

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APR 21 1965

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THE ROLE OF NONDESTRUCTIVE TESTING IN THE LOS ALAMOS
REACTOR PROGRAM*

By

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ABSTRACT

The Los Alamos Scientific Laboratory, operated by the University of California for the U. S. Atomic Energy Commission, has been active for more than 20 years in developing, designing, and building nuclear reactors of four general types: research, power, rocket propulsion, and critical assembly.

The Nondestructive Testing Group gives service to practically all the activities and projects of the Laboratory; this paper describes some of the unique nondestructive testing techniques and applications developed for and used in the reactor program.

LAPRE (Los Alamos Power Reactor Experiment) was based on the use of a uranium phosphate solution at high temperature. This solution is very corrosive, and all reactor parts in contact with it were clad with gold. Special radiographic techniques were used to inspect the gold during the process of rolling it from cast ingot into sheet. The welded seams were similarly inspected.

*Work supported by the United States Atomic Energy Commission

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An electrode-potential inspection method was developed for checking the gold surfaces for embedded impurities.

The fundamental concept of LAMPRE (Los Alamos Molten Plutonium Reactor Experiment) is the use of liquid--rather than solid--plutonium metal as fuel. Tantalum capsules contained the fuel. Novel nondestructive testing methods were used to check the soundness of base metal and welds during the production of the capsules, and to study the plutonium-loaded capsules before, during, and after melt-freeze tests. A molten plutonium pump experiment was observed by radiographic techniques, including a gamma-ray closed television circuit.

For UHTREX (Ultra High Temperature Reactor Experiment), now under construction, microradiographic and electron microscopic studies have been made on 350-micron-diameter pyrocarbon-coated uranium carbide beads, to evaluate uranium migration as a function of temperature. The amount and uniformity of the uranium loading in the UHTREX graphite elements are determined with specially designed scintillation counters.

About 90 percent of the work on this subject has not been previously published.

INTRODUCTION

The University of California's Los Alamos Scientific Laboratory was established on 1 January 1943 to develop the first nuclear weapons. Despite this specific mission, the reactor development program started only 1 year later, resulting in the development and construction of the world's first homogeneous reactor, the Water Boiler, and the world's first fast reactor Clementine. The reactor activities of the Laboratory expanded thereafter into four distinct fields:

1. Research reactors
2. Power reactors
3. Critical assemblies
4. Development of a reactor capable of propelling a rocket

The Nondestructive Testing Group gives service to practically all the activities and projects of the Laboratory. In this presentation are described some of the unique nondestructive testing techniques and applications developed for and used in the reactor program.

THE LOS ALAMOS POWER REACTOR EXPERIMENT (LAPRE)

In the mid-1950's, two reactors in the LAPRE series were constructed and operated at high temperatures with a uranium phosphate solution. This completely new concept, at that time, in nuclear power reactor design allowed a homogeneous reactor to operate at temperatures and pressures far greater than those previously considered. At the same time, it was realized that this liquid fuel was highly corrosive. All reactor parts which were in contact with the uranium phosphate had to be gold clad.

The reactor vessel was thoroughly radiographically inspected to guarantee the soundness of the walls in every respect (see Figure 1, bottom photograph). Because of the weight and the wall

thickness of the vessel, a specially designed scaffold had to be constructed to hold the vessel at the same height from the floor as the horizontal X-ray beam emanating from the doughnut of the 24 MEV Allis-Chalmers Betatron. By using a 5-ton crane, the vessel could be rotated about its axis, thereby making it possible to cover radiographically the entire circumference of the object under investigation.^[2]

Whenever permissible and possible, a cobalt-60 source was inserted along the axis of the vessel and radiographs taken of an entire 360° band of its cylindrical portion.^[3,4]

The gold to be used for cladding was rolled to size by the Metallurgy Department of the Laboratory. It was important to ascertain that no dust or debris infiltrated the gold during the rolling process. Two types of nondestructive inspection were therefore performed:

A. Radiography of the gold sheet after each rolling (see Figure 1, top photograph). The gold sheets were transported and kept in plastic containers. Lowest possible X-ray energies and fine-grain films were used to obtain highest radiographic contrast and quality.

B. Electrode potential testing.^[5] Since the gold sheets were rolled from a 1-inch-thick slab down to a thickness of 0.015 inch, many rollings had to be performed. Despite extreme care and cleanliness during the rolling operation, it was impossible to prevent the pick up of inclusions of a size sufficient to penetrate the finished product completely.

The Electrode Potential Tester (see Figure 2) was developed by members of the Nondestructive Testing Group. The principle of this tester is based on the knowledge that one could detect an electric current or field produced by the action of an electrolyte in the region of a surface inclusion or pinhole. It is presupposed that the inclusion

and matrix materials will pass in ionic form into the electrolyte and that the two reactions do not produce equal potential differences.

The first prototype tester, as illustrated in Figure 2, consisted of a standard vacuum tube voltmeter, the D.C. probe of which was inserted into a lucite holder. In it were embedded an electrolyte reservoir, and electrolyte feed channel, a gold wire electrode, and a $\frac{1}{4}$ -inch-diameter sponge used to confine the electrolyte (in this case $\text{HCl-H}_2\text{O-HAc}$, 2:2:1) on the test specimen. The tester was manually operated, as is seen in the lower photograph of Figure 2.

This electrode potential scan of the gold sheet was applied after each rolling reduction to determine if pickling was necessary. Pickling operations of sufficient duration to dissolve all possible inclusions would have taken a prohibitively long time; therefore, the tester was used after each pickling cycle to determine if inclusions had been removed. By applying this tester, the estimated processing time with the prescribed pickling after each reduction was shortened from three months to two weeks.

THE LOS ALAMOS MOLTEN PLUTONIUM EXPERIMENT (LAMPRE)

This experiment is based on the idea of using metallic plutonium as a fuel in the liquid rather than in the solid state. It was part of the concept of a fast breeder reactor to produce power and at the same time manufacture more new fissionable material than it consumes.

Capsules of tantalum metal were used to contain the fuel in the core. Heat was removed from the fissioning fuel by a circulating stream of molten sodium. The operating temperature was about 950°F with a design power level of 1,000 KW.

Some of the interesting nondestructive test methods developed for and applied to this reactor experiment are described in the following paragraphs.

A. Tantalum Bar Stock and Thimbles

The bar stock was ultrasonically inspected for internal cracks. A Curtiss-Wright Immerscope was used for this purpose. The axis of the ultrasonic beam emanating from a line-focused lithium sulfate transducer was directed at the center of the bar. A frequency of 5 megacycles was used in this case.

The thimbles were impact-extruded from this bar stock to a wall thickness of about 0.030 inch and a diameter of about 0.430 inch. These thimbles could not contain any scores, pits, galls, and cracks, because of the danger of rapid corrosion when in contact with molten sodium.

Three types of nondestructive inspection were therefore developed for this purpose.

1. Radiographic inspection. Three views were taken of each thimble (0° , 60° , and 120°) with its axis parallel to the film. 1000 KVP X-rays were utilized to penetrate both walls of the specimens. By using fine-grain films (double film technique) and 0.005-inch lead intensifying screens in vacuum cassettes, a radiographic resolution of 0.004 inch was obtained.

2. Fluorescent penetrant inspection. Experiments had to be performed in order to establish the appropriate emulsification time, thereby visualizing under the ultraviolet light only those surface defects which could be harmful. Figure 3 illustrates in a sketch and photograph the decreased lengths of longitudinal surface defects of increased emulsification time. The insert is a photomicrograph of a cross-section of such

a rejectable defect (200X). Based on these experiments the following technique was applied:

Penetrant:	ZL-22 for 20 minutes
Emulsifier:	ZE-2 for 20 minutes
Washing time using automatic rotary washer:	6 minutes at 95° to 100°F
Drying time:	10 minutes at 115° to 140°F
Developer:	ZP-4 for at least 10 minutes

Most of the surface defects uncovered by this fluorescent penetrant inspection could not be detected by visual or radiographic means.

3. Electromagnetic method to measure wall thicknesses of thimbles. The thimble wall was inserted between two coaxial coils mounted on the ends of a U-shaped yoke. The variations in the wall thickness caused a linear phase shift in the signal induced in the receiving coil. This shift was displayed on an oscilloscope which was calibrated against known tantalum thicknesses. By moving the coils axially along the thimble and rotating the thimble, the minimum and maximum wall thicknesses and their locations could be obtained. The frequency range for this investigation was between 200 and 300 kilocycles, depending upon the thickness range of the walls of the thimbles.

B. Highly Radioactive Tantalum Capsules Filled with Plutonium

Betatron radiography was used to ascertain the physical condition of these capsules after they were removed from the reactor (see Figure 4). Remotely controlled handling

devices were used for personnel safety reasons. A finite object-to-film distance utilizing the inverse-square law decreased the amount of undesirable radiation emanating from the specimen and exposing the film, thereby contributing to an increased radiographic contrast.

C. Molten Plutonium Pump Experiment^[6]

This experiment was a subcritical mock-up of a reactor core in which a plutonium-iron alloy was circulated by means of a sodium lift pump. This type of circulation would permit continuous flushing of fission gases generated, and would create the potential for fuel reprocessing during operation. Sodium for the lift pumping was circulated by an E.M. pump in an isothermal loop at 500°C. The purpose of this test was to study pump characteristics, instrumentation, sodium-fuel separation, and fuel transfer systems.

Special nondestructive inspection methods were developed in order to observe the operational characteristics of the core and the fuel transfer systems. Figure 5 illustrates the setup of this experiment and a radiograph of the core showing the catch pan and the liquid plutonium level.

Radiographs were taken in order to study the test assembly involving static and steady state conditions such as float location, fuel level, sodium in the lift pump annulus while pumping, etc. Iridium-192 and cobalt-60 were the two sources of gamma radiation which lent themselves very successfully for this investigation. Iridium, because of its lower energy, produced better radiographic definition and contrast. The iridium sources were contained in tungsten collimators and exposed by removing the front plug. The shielding and collimation were sufficient to allow personnel to remain in the area behind the source during exposure.

The entire experimental setup located behind the board was painted on the front, as is seen in the photograph.

This painted sketch made it possible to place the isotope in the desired position in order to radiograph the area of interest.

A gamma alarm was placed behind the test section; it activated a red light visible to the operators whenever the sources were exposed.

In addition to taking radiographs, a gamma-ray TV system was developed which made it possible to study on the screen the various events as they were taking place. The varying fuel flow, the starting of pump action, the fuel levels, and fuel transfer operations could be readily observed. Movies were also taken of the TV screen, thereby permanently recording the various phases of this experiment.

THE ULTRA HIGH TEMPERATURE REACTOR EXPERIMENT (UHTREX)

This experiment now under construction at Los Alamos will use unclad highly refractory fuel in a gas-cooled reactor. [7]

The fuel elements will be a mixture of "Triplex" pyrocarbon-coated UC_2 beads, with average diameters of 350 microns, and graphite. They will have cylindrical shapes with a diameter of about 1.2 inches, and will be extruded and graphitized to size.

Again, various nondestructive test methods were developed and applied in order to study the behavior of these beads when exposed to various temperatures. It is of importance to determine the degree of uranium migration through the pyrocarbon coatings as a function of temperature and time.

Figure 6 illustrates microradiographs of such beads as received from the manufacturer and after heat treatment for 4 hours at 2300°C. These radiographs were taken under darkroom conditions by placing the beads directly on the photographic emulsion of spectrographic plates. Soft X-rays with an energy of about 6 KVP were utilized in a helium atmosphere. The X-rays penetrated the pyrocarbon layer and visualized clearly the

migrated uranium after heat treatment.

Some of these beads were sectioned and metallographic photographs taken, as seen in Figure 7. Again, uranium migration could be observed as a result of heat treatment.

In order to study these phenomena in greater detail and beyond the range of optical microscopy, replicas were made of the sectioned surfaces of these beads. These replicas were then shadow-casted and transmission electron micrographs were taken. Figure 8 illustrates these 5500X electron micrographs. The boundaries between the UC_2 core and the surrounding pyrocarbon containing migrated uranium can be clearly seen.

The manufactured fuel elements have to be inspected for uranium loading and homogeneity of uranium distribution. Ring-type sodium-iodide thallium-activated scintillators are used for this purpose. Figure 9 shows the model of such a scintillation counter with one segment removed. The other half of this figure is the photo of the entire gamma-counting instrument with automatic read-out equipment. Data are recorded on punch tapes and fed into computers for final evaluation.

DISCUSSION

In this paper a brief review was presented of some of the various activities of the Nondestructive Testing Group of the Los Alamos Scientific Laboratory. In order to be of service to all the projects, many factors must be taken into consideration. Close communication and cooperation are the basis of successful service. The problems have to be crystallized and discussed. All possible and probable nondestructive test methods, and especially their limitations, have to be fully understood. Last, but not least, the data obtained have to be analyzed and evaluated in order to determine their usefulness with regard to establishing respective specifications.

ACKNOWLEDGEMENT

The content of this paper is the result of the work of many members of the Nondestructive Testing Group over many years. They all deserve the credit. In addition, the progressive atmosphere of the Los Alamos Scientific Laboratory created and stimulated these evolutionary and fruitful activities.

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TEXT FOR FIGURE CAPTIONS

Figure 1

- Top: Gold Sheet Radiography for Reactor Liner
- Bottom: Betatron and Cobalt-60 Radiography of LAPRE Reactor Vessel

Figure 2 - Electrode Potential Tester

- Top: Schematic Cross-Section of the Tester
- Bottom: Tester Applied to Gold Slab

Figure 3 - Fluorescent Penetrant Inspection of Tantalum Thimbles

- Top: Sketch of Visualized Longitudinal Surface Defects as Function of Emulsion Time
- Bottom: Photograph of Tantalum Thimbles Taken Under Ultra-violet Light
- Insert: Photomicrograph of Cross-Section Containing Typical Defect as Detected by this Surface Inspection Method

Figure 4

- Left: Radiographic Setup for Inspecting Highly Radioactive Tantalum Capsules Filled with Plutonium using the 24 MEV Betatron
- Right: Radiograph of Such a Capsule

Figure 5

- Top: Photograph of the Molten Plutonium Pump Experiment
- Bottom: Radiograph of the Core Showing the Liquid Plutonium Level

Figure 6 - Microradiographs of Pyrocarbon Coated UC_2 Beads Average Diameter - 350 Microns

- Top: Radiograph of Bead as Received
- Bottom: Radiograph of Bead after Heat Treatment for 4 Hours at 2300°C

Figure 7 - Metallographic Photographs of Sectioned Pyrocarbon

UC₂ Beads

Top: Bead as Received

Bottom: Bead after Heat Treatment for 4 Hours at 2300°C

Figure 8 - Electronmicrographs of Interface Between UC₂ Core and

Pyrocarbon Coating of Beads (5500X)

Top: Interface of Bead as Received

**Bottom: Interface of Bead after Heat Treatment for 4 Hours
 at 2300°C**

Figure 9 - Scintillation Counting Equipment to Determine Uranium

Loading of Fuel Elements

**Top: Model of Scintillation Counter with One Segment
 Removed**

**Bottom: Scintillation Counting Instrument with Automatic
 Read-out Equipment**

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Figure 1

Top: Gold Sheet Radiography for Reactor Liner

Bottom: Betatron and Cobalt-60 Radiography of LAPRE
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Figure 2 - Electrode Potential Tester

Top: Schematic Cross-Section of the Tester

Bottom: Tester Applied to Gold Slab

1 MIN.

10 MIN.

6 MIN.

12 MIN.

8 MIN.

20 MIN.

1 INCH

EFFECT OF
EMULSIFICATION TIME

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Figure 4

Left: Radiographic Setup for Inspecting Highly Radioactive
Tantalum Capsules Filled with Plutonium using the
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Right: Radiograph of Such a Capsule

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Figure 5

Top: Photograph of the Molten Plutonium Pump Experiment

Bottom: Radiograph of the Core Showing the Liquid Plutonium
Level

($\overline{\hspace{1cm}}$)
50 μ

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Average Diameter - 350 Microns

Top: Radiograph of Bead as Received

Bottom: Radiograph of Bead after Heat Treatment for 4 Hours
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()
50 μ

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Bottom: Bead after Heat Treatment for 4 Hours at 2300°C

μ

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Figure 9 - Scintillation Counting Equipment to Determine Uranium
Loading of Fuel Elements

Top: Model of Scintillation Counter with One Segment
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Bottom: Scintillation Counting Instrument with Automatic
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