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C.N.E.N. - Centro di Saluggia

L.F.C.E.C.

SURVEY OF QUALITY CONTROL METHODS USED AT
L.F.C.E.C. FOR THE DEVELOPMENT OF CIRENE
FUEL ELEMENT.

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1. INTRODUCTION

The Laboratorio Fabbricazione e Controllo Elementi di Combustibile (L.F.C.E.C.) has been charged to develop a fabrication process and provide a capability to manufacture high quality, fully characterized fuels for the Cirene reactor program.

The development program started in the 1968, during this period many fuel pins and bundles have been fabricated for irradiation and other test programs.

Following the reference process development, at the end of this year a process proof test will begin to demonstrate the ability to produce three fuel bundles a day for about one month.

This paper presents a summary of the procedures used at the L.F.C.E.C. for the characterization and quality control of the fuel elements for use in the Cirene program.

Some results obtained during the last process development phase are also presented.

The Cirene fuel element to which these methods and results are applied consists of eighteen fuel pins mounted as a fuel pin bundle of 500 mm length.

The fuel pin consists of a stack of UO_2 dished pellets 474.5 mm long., sheathed in a Zircaloy-2 cladding tube 19 mm in inside diameter, 0.53 mm thick.

The fuel pellet diameter is selected to ensure a pellet-to-cladding diametral gap in the range 0.14-0.23 mm; the length is 21 mm; the minimum density is 94% of theoretical.

2. INCOMING-MATERIAL INSPECTION

Regular inspection of incoming materials serves the purpose of assuring us that only materials in accordance with specifications are used in fuel elements manufacture.

It serves another very important purpose by providing a qualification of materials suppliers together with the complete data on quality of each lot of materials to be used.

Regular inspection of incoming materials is performed on tubing for fuel pin cladding, bar for end caps and plenum inserts, sheet for end plates and spacer pads, and on uranium dioxide.

2.1. CLADDING INSPECTION

In the first development phase a significant work has been the qualification of high quality Zircaloy tubing suppliers.

Owing to the unusual diameter and thickness ratio of the fuel cladding, the specification requirements for Cirene cladding tubes include a tight control of mechanical properties and chemical composition (e.g. carbon content), small dimensional tolerances (inside diameter $\pm 0,05$ mm with an average inside diameter range max. 0,04, wall thickness 0.53 mm $\pm 7.5\%$) and rigorous control of defects.

At this time more than 3.500 tubes have been bought from four suppliers and subjected to a rigorous incoming-material inspection including the following tests:

- 1° - visual inspection
- 2° - surface condition

- 3° - dimensions (inside diameter, wall thickness, total length, straightness)
- 4° - ultrasonic test
- 5° - metallography (grain size and hydride orientation)
- 6° - mechanical properties (tension and burst testing)
- 7° - chemical composition
- 8° - corrosion test.

Diameter of each fuel cladding tubes is measured by air gaging: a two-nozzle air plug is used for checking inside diameter and a four-nozzle plug is used to furnish a verage diameter readings.

Wall thickness is measured during the ultrasonic test with an ultrasonic thickness gage.

Each tube is ultrasonically tested for detection of defects. The test is performed as a pulse echo immersion test with water as the couplant by means of apparatus calibrated by a repeat runs of an artificially notched standard.

Present size of standard defects are: longitudinal notch 1 mm long, transverse notch 0.50 mm with a depth 10% of tube wall thickness -The artificial defects are made with a mechanical method.

All signals also less than those induced by standard notches are recorded to provide a comparative index of tube quality.

Over 3,000 tubes less than 1% was rejected during the ultrasonic test because of an ultrasonic signals greater than those induced by the standard defects.

Much more tubes were rejected for wall thickness non conformance. Typically, 5% per cent of an 500 tube lot were rejected.

Although an ultrasonic inspection for all tubes was required by the tube procurement specification, this relatively high number of non acceptable tubes indicate that rigorous incoming-material inspection is always an essential step in the fuel element fabrication process.

A 2 per cent sample is selected at random from each lot of tubes for mechanical testing.

Two specimens are cut from each tube: one for tension testing and the other for hydrostatic burst testing.

The hydrostatic burst testing is used for the determination of circumferential ductility - calculated as the increase in perimeter of a fractured burst test specimen and expressed as percentage of the original circumference.

The results are usually very scattered and therefore a good characterization of the lot of tubes is often difficult.

Typically for a lot of tubes it has been obtained on a sample of ten a mean circumferential elongation of 19% with a confidence interval for 95% confidence of $\pm 5\%$.

Higher degree of accuracy can probably be achieved through a review of the test procedure in order to get a more standardized test.

2.2. URANIUM DIOXIDE ANALYSES

Regular inspection of each lot of UO_2 powder supplied is performed.

For this analyses an homogeneous sample of powder representing the entire lot is obtained by blending aliquotes taken from all containers of the lot.

The chemical and isotopic composition, the physical properties of the UO_2 powder are confirmed by these analyses:

- 1° - total uranium
- 2° - uranium isotopic composition
- 3° - oxygen-to-uranium ratio
- 4° - impurity
- 5° - particle size distribution
- 6° - surface area.

Confirmation of chemical composition is important for several reasons. Accurate knowledge of the total uranium content and its isotopic composition is important for nuclear materials accountability and safeguards.

Surface area and particle size measurements helps to evaluate the quality of uranium oxides used in the fabrication of fuel pellets.

In fact these physical characteristics of the powders affect the sintering performance.

Potentiometric titration by the ferrous sulfate reduction in phosphoric acid-dichromate titration method is used for the determination of uranium.

The oxygen-to-uranium ratio is determined by a gravimetric method. Our oxidation techniques involves a preliminary oxidation at 470°C for a minimum of 4 hours followed by 4 hours at 900°C .

The surface area is determined by a volumetric methods using a glass apparatus. Equilibrium points are established for at least three different pressures and the adsorption data are handled according to the BET theory.

The method is time consuming but gives reliable results.

3. PROCESS INSPECTION

A thorough characterization of the fuel produced cannot be obtained without a quality control plan during the fabrication.

The production is divided into subgroups (process batches). The size of these subgroups is determined on the basis of the following criteria:

- the material shall come from the same lot of received material
- identical and simultaneous processing in a single piece of equipment.

3.1. PELLETS QUALITY CONTROL

Since the fuel composition particularly some impurities, has a significant effect upon the life time of the fuel under in-reactor conditions, the chemical composition and physical properties of the fuel must be determined.

This is done by using a statistically designed quality control plan according to the U.S.A. military standard "Sample procedures and tables for inspection by variables for per cent defective" MIL-STD-414.

The quality index in MIL-STD414 is the acceptable quality level (AQL). The choice of the AQL values depends on the risk we take of accepting a lot that contains a given number of pellets exceeding the specification limits.

For each pellets process batch the parameters to be tested for and the frequency of testing are:

1° - visual inspection	all pellets
2° - dimensions (diameter, length, dish)) as per MIL-STD-414 table A-2 inspection level IV AQL 0.4
3° - density	
4° - oxygen-to-uranium ratio	
5° - impurity analysis (moisture, fluoride, gas content, carbon)) 1 per batch
6° - microstructural analysis	

The density measurement is used to evaluate pellet porosity which is an important physical property because it governs the release of fission gas during irradiation.

The pellets density in per cent of theoretical is obtained by process personel using a geometric method to obtain the volumes by pellets dimensions.

All the values obtained from the dimensional and density measurements are processed first to establish if the lot pass the acceptance criterion and second to characterize the fuel pellets.

Applications of a microcomputer is help ful in this fuel evaluation.

An analysis of dimensional and density variance on 6 pre-production lots made as process-capability test showed a mean of 18.852 mm,

a mean length of 20.86 mm, a mean density of 95.27% of theoretical, with a 3 σ limit of 0.0096 mm for the diameter, 0.57 mm for the length and 0.60% T.D. for the density.

Impurities that can have an adverse effect on the life of fuel pins are water, absorbed gases, particularly hydrogen, and fluoride. These impurities cause severe corrosion of the zircaloy cladding and contribute toward pressure build up within pins.

The moisture content of the pellets has been determined till now by means of a commercial solid moisture analyzer based on the coulometric electrolytic principle.

The method is subject to problems when the amount of water available for measurement is small. For the Cirene pellets a maximum water content of 5 ppm is required. Therefore, the desire for greater precision and accuracy led to use a different instrumentation based on the same principle but employing a more sophisticated electronic counter.

The sample is heated at 300°C in a stream of dry nitrogen.

As regards the gas content many analyses have been made in CNEN's Laboratories using different methods.

The lack of standards for water and other gases evolved at higher temperature makes difficult to resolve differences between laboratories and the methods cannot be completely evaluated. Till now we have not decided which apparatus to be used at L.F.C.E.C. for routine analyses.

Microstructural analyses and void size distribution in pellets is obtained from photomicrographs of polished and etched sections of pellets.

The evaluation is made by comparison of observed void size distributions with standard photo micrographs.

3.2. FUEL PINS QUALITY CONTROL

In order to assure the integrity and an hermetic sealing of the fuel pin a close control of the end plug welds is necessary.

This is obtained by the following tests:

- 1° - helium leak test
- 2° - radiography
- 3° - metallography.

Each weld is checked by an helium mass spectrometer leak detector; in addition each weld is radiographically tested for lack of penetration and porosities, cracking, inclusions and other discontinuities.

Owing to the lack of a satisfactory non-destructive testing method adequate reliability of welding process is obtained only with an accurate set-up and qualification of the welding procedures. During this phase many specimens are sectioned for metallographic examination to determine the welding parameters.

Moreover, during the production phase two sample welds are made at the start of the welding of each batch and two sample welds are made at the end of the batch welding and metallographically examined. If any of these welds fail to meet the metallographic requirements the batch is rejected.

Inspection of completed pins for dimensional pin length, diameter and bow conformance is made using mechanical indicators and inductive displacement electronic gages.

4. CONCLUSIONS

Some quality control, methods used during development and manufacture of Cirene fuel elements have been described.

Operating experience and current limitations in the use of these methods have been also presented.

From our experience, there are no serious deficiencies in the status of quality control methods for nuclear fuels, though some methods must be improved for greater reliability and faster analyses.

The only lack is that of a sensitive and reliable non destructive test method for control of end plugs seal welds.

At the present time, our activity is devoted to the following objectives:

- to establish a plan to ensure the reliability of the methods used and to ensure that this reliability is maintained throughout routine fabrication;
- to establish a system for accumulation and processing of all fabrication and inspection data.

Data processing equipment will help in sorting and storing this large amounts of information generated about each batch of fuel in order to obtain a better characterized fuel.

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