BNA PELLET MATERIAL PROPERTIES TEST PROGRAM IN SUPPORT OF ACR-1000 FUEL DESIGN

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Abstract

The ACR-1000®[1] CANFLEX®[2]-ACR fuel bundle design is an evolution of the 43-element CANDU®[3] 6 CANFLEX Mk-4 fuel bundle. The centre ring of the CANFLEX-ACR fuel bundle consists of a single large-diameter element containing burnable neutron absorbers (dysprosia and gadolinia) in an yttria-stabilized zirconia matrix (with no fissile material), and is designed to control the coolant void reactivity during postulated accidents.

The material properties of zirconia have been studied extensively. However, the material properties of a composition containing zirconia, yttria, dysprosia and gadolinia have yet to be fully characterized. Accurate assessment of material properties is important for determining conditions and behaviour of the centre element. Therefore a test program must be developed to provide correlations and tables of empirical values for the properties of various BNA pellet compositions. While in-reactor tests are important for confirming behaviour of this new material, the material properties test program focuses on out-reactor testing to determine thermal and mechanical properties, as well as to investigate the phase stability, oxidation-resistance and leaching behaviour. This paper provides an overview of this ongoing program.

1. Introduction

The ACR-1000 CANFLEX-ACR fuel bundle design is an evolution of the 43-element CANDU 6 CANFLEX Mk-4 fuel bundle. The CANFLEX-ACR fuel bundle also has 43 elements with 2 different element sizes. The outer 3 rings of the fuel bundle consist of 42 elements, each with an 11.5-mm diameter and containing low enriched uranium (LEU) pellets. The centre element has a larger diameter of 20 mm and contains burnable neutron absorber (BNA) pellets made of dysprosia and gadolinia in a fully yttria-stabilized zirconia matrix. The larger diameter of the centre element, as well as the use of the BNA pellets (with no fissile material) in the centre element, are designed to control the coolant void reactivity during postulated accidents.

The ACR-1000 BNA pellet compositions are selected to provide stability to the zirconia matrix, as well as provide the required coolant void reactivity (CVR) control. All the component oxides in the BNA pellets are well characterized. The material properties of yttria-stabilized zirconia have been studied extensively and it is known to have good irradiation behaviour. In addition, dysprosia and gadolinia are commonly used to control CVR and have been well studied in connection with UO_2.

[1] ACR-1000® (Advanced CANDU Reactor®) is a registered trademark of Atomic Energy of Canada Limited (AECL).
[2] CANFLEX® is a registered trademark of AECL and the Korea Atomic Energy Research Institute (KAERI)
[3] CANDU® (CANada Deuterium Uranium) is a registered trademark of Atomic Energy of Canada Limited (AECL).

This knowledge of the component oxides gives confidence in proceeding with the use of BNA pellets. However the material properties of a composition containing zirconia, yttria, dysprosia and gadolinia have yet to be fully characterized. Accurate assessment of material properties is important for determining conditions and behaviour of the fuel element. Therefore, a test program must be developed to provide correlations and tables of empirical values for the properties of this new BNA pellet material. The data may then be used directly for the evaluation of the centre element performance or used to extend the capabilities of computer codes to model BNA pellet behaviour.

While in-reactor tests are important for confirming behaviour, the BNA pellet material properties test program focuses on out-reactor testing to determine thermal and mechanical properties, as well as to investigate the phase stability, oxidation resistance and leaching behaviour. Fluence is not expected to significantly affect the material properties of the material since during irradiation, transmutation of the elements will result in isotopes that have similar properties to the original. In addition, open literature indicates that radiation damage can be annealed out at high temperatures so the material properties may not be significantly affected (see Section 9). However, irradiation tests will also be performed to confirm this. This paper provides an overview of the ongoing BNA pellet material test program.

2. BNA pellet compositions

The amounts of dysprosia and gadolinia in the centre element have been optimized, based on physics analysis, to achieve the required CVR over the life of the reactor.

Pure zirconia undergoes phase transformations when temperature increases. One effect of this phase change is a significant resulting volume change. The volume change causes cracking of the material during cooling. To prevent phase change and the resultant cracking, zirconia can be "stabilized" by doping it with various oxides, including dysprosia, gadolinia and yttria. Zirconia can be fully-stabilized or partially-stabilized depending on the amount of stabilizer added. Dysprosia, gadolinia and yttria are all rare earth oxides. The fully-stabilized region of the phase diagram for a zirconia-rare earth oxide composition is the region in which the material has a cubic crystal structure. By adding stabilizers, the transformation temperature at which phases other than cubic appear in the material is lowered, which reduces the rate of phase change enough to make it negligible. An advantage of being in the cubic phase is that the cubic structure has been shown to be highly radiation-resistant [1]. The amount of yttria specified in the compositions of both intermediate and reference ACR fuel, in combination with the amount of dysprosia and gadolinia, place the compositions within the fully-stabilized region of a predicted phase diagram (see Section 6).

Yttria has been selected because there is irradiation experience with yttria-stabilized zirconia (Section 9) and yttria has a small neutron-capture cross-section, which has a negligible impact on the reactivity of the fuel bundle. This allows for future adjustments of BNA in the centre element – the amounts of BNA and yttria can be adjusted while keeping the total amount of stabilizer at the same mol% rare earth oxide.

There are seven compositions of interest:

- One composition representing the centre element material composition of fuel used during the transition to the reference equilibrium core (Composition 1)

- Five compositions to investigate the effects of varying the amounts of each BNA:
  - One composition of yttria and zirconia only to compare with known data for yttria-stabilized zirconia and to investigate the effects of no BNA (Composition 2)
  - One composition to investigate the effects of a lower amount of total BNA (Composition 3)
  - One composition to investigate the effects of a higher amount of total BNA. This particular composition bounds the reference fuel composition (Composition 7)
  - One composition containing more gadolinia than dysprosia (Composition 4)
  - One composition containing more dysprosia than gadolinia (Composition 5)
- Two compositions to investigate the effects of varying the total mol ratio of rare earth oxides:
  - One composition to represent the low end of the fully-stabilized region of the predicted phase diagram. This composition contains approximately the same amount of BNA as Composition 1, but contains no yttria (Composition 6)
  - One composition to represent the high end of the fully-stabilized region of the predicted phase diagram. This composition is the same as the composition listed above to investigate the effects of a higher amount of total BNA (Composition 7)

The above seven compositions may be represented as shown in Table 1. In order to optimize the test program, a smaller matrix of these material types against the different tests is being examined.

Specimen Batch	mol% BNA	Total mol% Rare Earth Oxide
Composition 1	Nominal	Nominal
Composition 2	None	Nominal
Composition 3	Low	Nominal
Composition 4	Nominal – High Gd	Nominal
Composition 5	Nominal – High Dy	Nominal
Composition 6	Nominal	Low
Composition 7	High	High

Table 1 Specimen Compositions

3. Density

Although density is not a thermal property, it is required in order to determine thermal conductivity.

During manufacturing, the density achieved is lower than the theoretical density; however the poison composition can be adjusted accordingly to attain the required neutron absorbance. Densities achieved during manufacture are recorded in the specific fabrication reports.

The Archimedes (immersion) method is being used to determine the density at room temperature. To obtain the density at other temperatures, the density at room temperature will be combined with the coefficient of thermal expansion (Section 4.3). This will provide the densities at all temperatures for which the coefficient of thermal expansion was measured.

The density measurements are being done by the Institute for Transuranium Elements (ITU) as part of the thermal property tests (Section 4).

4. Thermal properties

Thermal properties of the BNA pellet material are required to verify that the fuel meets the acceptance criteria on centre element thermal behaviour, including that pellet temperature will not exceed melting temperature and the element will not expand in length beyond the allowed clearance.

The thermal properties to be determined in this test program are:

- Thermal diffusivity and thermal conductivity
- Specific heat capacity
- Coefficient of thermal expansion (CTE)
- Solidus melting temperature
- Density (discussed in Section 3)

Each property will be measured from room temperature up to as high a temperature as possible. This will establish a relationship between the measured thermal property, temperature, and material composition. The diffusivity, specific heat capacity and density, which are used to calculate conductivity, will be measured under the same conditions. If interpolation or extrapolation is required, justification and documentation will be provided.

The thermal property tests are being done by ITU, which has the capability of performing high temperature thermal property measurements (greater than 1800 °C).

4.1 Thermal diffusivity and thermal conductivity

The thermal diffusivity was measured using the laser flash method. ITU's LAF.I laser flash apparatus provided thermal diffusivity measurements to approximately 1200 °C. For higher temperatures, ITU's CLASH laser flash apparatus is being used.

The thermal diffusivity will be used with specific heat capacity (Section 4.2) and density (Section 3) to calculate thermal conductivity.

4.2 Specific heat capacity

A differential scanning calorimeter (DSC) was used to measure specific heat capacity up to a temperature of approximately 1200 °C.

4.3 Coefficient of thermal expansion (CTE)

A commercial dilatometer was used to measure the coefficient of thermal expansion (CTE) up to a temperature of approximately 1200 °C.

4.4 Solidus melting temperature

The solidus melting temperatures are measured using the pulsed laser heating method. The melting temperature corresponds to the plateau (freezing point) on resulting thermograms.

5. Mechanical properties

Mechanical properties of BNA pellet material are required to verify that the fuel meets the acceptance criteria on bundle mechanical behaviour, including that centre element will withstand any load applied to it during operation (including during refuelling).

The mechanical properties to be determined in this test program are:

- Fracture strength
- Stress-strain curve
- Plastic modulus (Work hardening rate)
- Elastic modulus
- Poisson's ratio
- Fracture toughness
- Hardness measurement

Each property will be measured from room temperature up to as high a temperature as possible. This will establish a relationship between the measured mechanical property, temperature, and material composition.

The mechanical property tests are being done by Chalk River Laboratories (CRL).

5.1 Fracture strength (flexural), stress-strain curve, plastic modulus

Four-point bending tests determined the flexural fracture strength of the BNA pellet material at ambient and elevated temperatures. The fracture load of the samples was measured in a four-point-1/4 point-loading configuration similar to ASTM C1161-02c [2] at ambient temperature and ASTM C1211-02 [3] at elevated temperatures, but for smaller samples than those specified in the ASTM standards.

Data from the bending tests and the elastic modulus tests (Section 5.2) are used to obtain stress-strain curves to a maximum of 300 °C.

The bending tests and resulting stress-strain curves will also give the plastic modulus (work hardening rate) if the material exhibits plastic deformation. At low temperatures, such as those being tested, ceramics do not exhibit plastic deformation. If the transition temperature at which the material begins to exhibit plastic deformation is apparent from the stress-strain curves, the plastic modulus can be determined. If no plastic deformation occurs at the temperatures tested, then the plastic modulus is not determined.

5.2 Elastic modulus

Determination of the elastic modulus using direct deflection or strain measurements is not very accurate. The elastic modulus of the BNA pellet compositions as a function of temperature from ambient temperature to above 300 °C was determined using the automated piezoelectric ultrasonic composite oscillator technique (APUCOT) [4] [5], which is an acoustic method (also called the resonance frequency method) based on the principles of ASTM C1198-01 [6]. The APUCOT measures the resonant frequencies of a specimen, which can then be used with specimen geometry and mass to determine elastic modulus.

5.3 Poisson's ratio

Poisson's ratio is a calculated value, based on transverse strain and longitudinal strain. For small specimens, such as these ceramic specimens, these strains are difficult to measure. However, for an isotropic material, Poisson's ratio can be calculated based on the elastic modulus and the shear modulus:

$$\nu = \frac{E}{2G} - 1 \tag{1}$$

where, ν = Poisson's ratio

E = Elastic Modulus

G = Shear Modulus

The elastic modulus as a function of temperature has been determined in Section 5.2. The method by which the shear modulus is determined is dependent on the elastic modulus. If the elastic modulus is within 15% of the values published in open literature for zirconia-based ceramics, the shear modulus will be assumed to be equal to the open-literature values of shear modulus for zirconia-based ceramics. If the elastic modulus differs by more than 15% of the open-literature values for zirconia-based ceramics, the shear modulus will be determined experimentally using an acoustic test method (likely based on ASTM C1259-01 [7])

Once the shear modulus is obtained, Poisson's ratio will be calculated.

5.4 Fracture toughness

The fracture toughness at ambient temperature was estimated from the length of the cracks emanating from the corners of a Vickers indentation test based on Reference [8]. The crack length was then used with indentation size, hardness, and elastic modulus (Section 5.2) in an analytical expression [9] [10] [11].

5.5 Hardness

The Vickers indentation hardness (HV) at ambient temperature has been determined according to ASTM C1327-03 [12].

6. Phase stability

As mentioned in Section 2, to prevent phase change and the resultant cracking, an amount of yttria is specified in the centre element of ACR-1000 fuel that, in combination with the amount of dysprosia and gadolinia, place the composition within the fully-stabilized region of a predicted phase diagram. The lowest transformation temperature for the cubic phase in a zirconia-yttria mixture occurs in approximately the middle of the cubic (i.e. fully-stabilized) region of the phase diagram. This composition (i.e. mol% yttria) and temperature correspond to a eutectoid point. Since they are also rare earth oxides, the eutectoid points for mixtures of zirconia-dysprosia and zirconia-gadolinia fall at roughly the same composition (i.e. mol% rare earth oxide) [13]. Selecting a centre element material with the same mol% rare earth oxides should ensure that it will remain fully stable (i.e. in the cubic phase).

The phase stability tests involve three parts. The first part investigates the formation of the cubic phase for various BNA pellet compositions and temperatures using thermodynamic calculations with F*A*C*T (Facility for the Analysis of Chemical Thermodynamics) software and developed models for ZrO_2-Dy_2O_3-Gd_2O_3-Y_2O_3 systems. The second part investigates the possibility of decomposition of the cubic phase to other phases (such as the monoclinic phase) at operating temperatures in the ACR-1000 with long-term annealing tests. The third part confirms the in-reactor stability of the BNA pellet with irradiation tests as described in Section 9.

The first two parts of the work is being done by the Royal Military College (RMC), who have already produced phase diagrams. The irradiation tests will be done by CRL. More information regrading the phase stability testing program is available in Reference [14].

7. Oxidation behaviour

For UO_2 fuel, the pellet oxidizes if there is a sheath defect and coolant enters between the sheath and the pellet. If this occurs for the ACR-1000 centre element, it is unknown what will happen to the BNA pellet. Behaviour of the composition when exposed to coolant is important to understand because oxidation (or reduction) may affect the pellet's material properties and thus affect performance of the central element of the ACR-1000 fuel bundle. Knowing the oxidation behaviour of the BNA pellet material will allow a better understanding of the in-reactor performance of bundles containing defective central elements, which can then be used to verify that the fuel meets the acceptance criteria on centre element thermal behaviour and mechanical behaviour.

The amount by which a material is oxidized is measured in terms of the number of oxygen atoms to the amount of metal atoms (i.e., zirconium, gadolinium, dysprosium and yttrium), known as the oxygen-to-metal (O/M) ratio. A greater O/M ratio indicates greater oxidation. Therefore, the oxidation tests measure the O/M ratios as well as any other information that could be used to characterize oxidation behaviour.

The oxidation behaviour tests require that the O/M ratios are measured for an oxygen partial pressure corresponding to the steam condition (the steam condition is the worst-case scenario - oxidation and secondary hydriding will result in sheath failure and expose the material to the steam) and for a range of temperatures covering normal operating conditions and abnormal operating conditions. However, the reaction is expected to be very slow at low temperatures, and if the experiment takes too long to reach the equilibrium O/M ratio at the lower temperature limit, then the test can be stopped and a new test will be performed with the lower temperature limit increased to the next temperature.

If the test results show that significant oxidation occurs and other material properties (e.g. thermal, mechanical, leaching) need to be determined for oxidized BNA pellet material, tests will be defined at that stage.

The oxidation behaviour tests are being done by CRL.

8. Leaching behaviour

For UO_2 fuel, when there is a sheath defect and coolant enters between the sheath and the pellet, the coolant dissociates to combine with the Zircaloy sheath, which results in hydriding, causing further degradation of the sheath. The increase in sheath hole size allows the coolant to access the pellet surface, allowing leaching of small grains or particles from the fuel element. If the sheath failure occurs in the central element of the ACR-1000 fuel bundle, dysprosium, gadolinium, yttrium and zirconium could be released to the reactor primary heat transport system (PHTS) and deposited downstream. Since dysprosium and gadolinium are neutron absorbers, their release, if any, could affect other reactor components including the defect detection and location systems. Knowing the leaching rate and deposition behaviour of BNA pellet material will allow a better understanding of the in-reactor performance of bundles containing defective central elements, which can then be used to verify that the fuel meets the acceptance criteria on failed fuel location and chemistry requirements.

The solubility and leaching behaviour of UO_2 have been studied extensively. Also, the solubility and leaching of zirconia have been studied extensively, and the solubilities of dysprosia and gadolinia have been investigated. However, very little is known about the leaching behaviour of a composition containing zirconia, yttria, dysprosia and gadolinia. Therefore, leaching tests are required to understand the leaching behaviour of BNA pellets.

The leaching tests are carried out in de-aerated water with an apparent initial pH at the upper limit of primary coolant in ACR at a typical shutdown temperature in CANDU reactors and at a representative normal operating temperature. Because information on the solubility of yttria is inconclusive, leaching tests are being done on yttria powder as well as the BNA pellet compositions.

Although there are several specific compositions of interest, thermodynamic calculations of the solubility of various BNA pellet compositions performed by the Royal Military College indicate that the solubilities are below detection limits. Therefore, only two compositions are being tested in addition to yttria powder. However, if the test results show that the leach rates are affected by a process that is not driven by solubility, other compositions may also be tested.

At least one set of the high temperature tests (~325 °C) are being carried out in the presence of pressure tube material coupons. This will determine if the presence of pressure tube material has an effect on the leaching behaviour and if the leached elements are likely to be deposited on PHTS materials. If time and resources allow, tests using sheath material coupons will also be performed.

The leaching tests measure the concentrations of the dissolved metal elements (Zr, Dy, Gd, Y) in the solution to show if leaching is likely to occur. Deposition, if any, on the coupons is determined with surface analysis techniques such as Scanning Electron Microscopy (SEM) and/or X-ray Photoelectron Spectroscopy (XPS). In addition, XRD is performed, which indicates if a phase change takes place during the tests (see Section 6 for the phase stability test program). When the tests have been completed and the results have been analyzed, any additional tests required will be defined at that stage.

The leaching tests are being done by CRL.

9. Irradiation behaviour

Irradiation behaviour of zirconia has been studied; however, a composition containing zirconia, yttria, dysprosia and gadolinia is a new material for which no irradiation data exist. The irradiation behaviour of this material is required to determine how the material will react in reactor (i.e. volume swelling, phase changes, degree of irradiation damage). Three types of irradiation tests are possible in the NRU – NRU multi-capsule rod irradiations, NRU fast neutron (FN) rod irradiations, and NRU high-pressure loop (demountable bundles) irradiations. The different locations have different neutron spectra and operating temperatures.

The variables for the irradiation tests include pellet composition, diametral clearance (with the wall thickness varied to prevent longitudinal ridging), pellet Length-to-Diameter (L/D) ratio, neutron fluence and temperature (when possible).

After the tests, microstructure examination, sheath inner surface examination, sheath outer diameter measurement, changes in volume and changes in composition will allow characterization of the BNA pellet material irradiation behaviour, including phase changes and swelling.

The irradiation behaviour tests are being done by CRL.

At the time of preparation of this paper, these irradiation tests are in the final stages of planning and component preparation, therefore there are no results available at this time. However, one of the reasons that zirconia was chosen as the matrix material is that there is extensive experience with irradiating zirconium in CANDU reactors. Zirconium alloys are used in pressure tubes as well as fuel bundle sheaths, end caps, end plates and appendages. Zirconium oxidizes easily. Therefore, an as-manufactured pressure tube or fuel bundle has a very thin layer of oxide (zirconia) on it before it goes into the reactor. The bundles oxidize more and the zirconia layer becomes thicker as they reside in the reactor. However, numerous observations at CRL have shown no significant difference in properties between the layer of zirconia on fresh bundle sheaths and the layer on irradiated bundle sheaths. Although this is an indication that zirconia exhibits good irradiation behaviour in CANDU reactors, the zirconia film formed due to sheath oxidation is mostly monoclinic and does not contain the rare earth oxides that the BNA pellet has.

As part of the research for inert matrix fuels (IMF), there have been extensive irradiations of stabilized zirconia. Irradiations of fully yttria-stabilized zirconia have been performed in the past which indicate that fully-stabilized zirconia exhibits good radiation resistance [15] [16] [17]. In addition, a study on the radiation tolerance of the cubic-fluorite crystal structures confirms that stabilized zirconia, which has a cubic crystal structure, has good radiation resistance [1].

Although previous experience with zirconia and yttria-stabilized zirconia gives some indication of good in-reactor behaviour, the BNA pellet irradiation tests are still required to definitively determine how a combination of zirconia, dysprosia, gadolinia and yttria in the cubic phase will behave in-reactor.

10. Summary

A test program has been implemented to establish the material properties of the BNA pellet used in the centre element of ACR-1000 fuel. This program involves the determination of thermal properties, mechanical properties, phase stability, oxidation behaviour, leaching behaviour and irradiation behaviour of various compositions of BNA pellet material over a range of temperatures representing in-reactor temperatures. These properties are important to determine the behaviour of the fuel in-reactor and ensure that it meets the acceptance criteria.

Although the testing has not been completed, results at the time of preparation of this paper indicate that the BNA pellet is acceptable for use in the central element of the ACR-1000. There have been no unexpected results and measurements are showing that BNA pellet material behaves similarly to yttria-stabilized zirconia.

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