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Overview of past and current activities on fuels for fast reactors at the Institute for Transuranium Elements

A. Fernandez [a,b,*], J. McGinley [b], J. Somers [b], M. Walter [b]

[a] European Commission, Joint Research Centre, Institute for Transuranium Elements, P.O. Box 2340, Karlsruhe, Germany
[b] European Commission, Joint Research Centre, Institute for Prospective Technological Studies, c/ Inca Garcilaso, 3. E-41092, Seville, Spain

ABSTRACT

Nuclear energy has the potential to provide a secure and sustainable electricity supply at a competitive price and to make a significant contribution to the reduction of greenhouse gas emissions. The renewal of interest in fast neutron spectra reactors to meet more ambitious sustainable development criteria (i.e., resource maximisation and waste minimisation), opens a favourable framework for R&D activities in this area. The Institute for Transuranium Elements has extensive experience in the fabrication, characterization and irradiation testing (Phénix, Dounreay, Rapsodie) of fast reactor fuels, in oxide, nitride and carbide forms. An overview of these past and current activities on fast reactor fuels is presented.

1. Introduction

The main Generation IV International Forum (GIF) criteria for future nuclear systems are sustainability, security, and reliability, economy, and non proliferation [1]. Furthermore, the fuel composition and specifications for the Generation IV gas, lead and sodium fast reactors (GFR, LFR and SFR) place severe requirements on the fabrication processes. The high-level goals of GEN-IV provide for group recycling of the actinides to ensure proliferation resistance in the fuel cycle. This implies that the actinides should be separated from the fission products, by hydro- or pyro-metallurgy processes, and then be converted into a solid compound for the fabrication of the fuels or targets. Together with materials technology, fuel is central to the successful deployment of all GEN-IV systems. Almost all such systems require the development of fuel technologies that are significantly different from those used in current power reactors, and for fast reactor systems the fuels must contain significant quantities of minor actinides, to satisfy proliferation requirements, and also to minimise long term radiotoxicity of the spent fuel. The incorporation of minor actinides in the fuel requires not only shielding but also automation of the entire fabrication process from liquid to solid conversion at the reprocessing plant, to fuel and assembly fabrication, to transportation, and ultimately to storage and loading in the reactor.

To date, the overwhelming majority of the world's nuclear fuel experience has been based on ceramic fuel pellets composed of uranium dioxide or a solid solution of mixed (U, Pu) oxides (MOX). Advanced fuel forms for GEN-IV fast reactor systems may be nitrides or carbides, due to their higher density and thermal conductivity, or composite fuels in which a fissile component is combined with a higher-conductivity inert matrix. Some experimental data is already available for such fuels, but far more information will be required in order to fully evaluate the suitability of these materials. Fuel microstructure can play a major role in determining the fuel in pile performance, and even when the fuel composition is selected, changes in microstructure (e.g., grain size, porosity distribution, etc.) can result in markedly different behaviour [2]. Conventional fuel fabrication technology is based on powder metallurgy methods. The introduction of minor actinides, however, will require dust free fabrication processes, which necessitates a renewal of interest in wet synthesis routes [3]. At the Institute for Transuranium Elements (ITU) a hybrid process consisting of a combination of sol–gel [4] and porous bead infiltration techniques [5] has been developed. The effects of fabrication processes on fuel microstructure are not yet fully established even for relatively well-known fuel materials, so carefully designed experimental programmes to explore this aspect are being planned. Finally, fuel chemistry evolution during irradiation will have a greater significance as operating temperatures and burnups will be higher than before. The R&D activities at ITU include development of fuel, pin fabrication and fuel irradiation testing. At present, the fuels considered are oxide and nitride although metal and carbide fuel fabrication and irradiation were also performed in the past. Current R&D activities also include irradiation experiments with new advanced cladding materials and concomitant welding development, in order to reach the ambitious goal of operating the innovative reactor systems at higher temperatures. Alternative cladding materials such as oxide dispersion-strengthened (ODS) steel and T91 steel are being considered.

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* Corresponding author. Address: European Commission, Joint Research Centre, Institute for Prospective Technological Studies, c/ Inca Garcilaso, 3. E-41092, Seville, Spain. Tel.: +34 954 48 8286; fax: +34 954 48 8300.
E-mail address: asuncion.fernandez-carretero@ec.europa.eu (A. Fernandez).

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2. Fabrication process development for fast reactor fuels

2.1. Oxide fuels

2.1.1. Sol–gel method

For Gen-IV fuels, consideration must be given to the quality of the powder, in terms of its particle size, generated in the liquid to solid conversion step. Conventional precipitation methods result in very fine powders which contaminate the internal surfaces in the glovebox, and will pose severe if not insurmountable problems. New solutions must be invoked. A flowsheet of the sol–gel method is shown in Fig. 1. The method requires the dissolution of the starting materials in nitric acid, and adjustment of the metal concentration and viscosity of the solution obtained. For industrial applications, the nitric acid feed streams from the reprocessing plant can be used directly. This homogeneous feed solution is then dispersed into droplets, which are collected in an ammonia bath, where gelation provides a droplet to particle conversion. The solid beads obtained are washed, dried using an azeotropic distillation procedure and then calcined. The size of the beads is largely determined by the viscosity of the feed solution and the characteristics of the device used to disperse the liquid into droplets. Using a rotating cup disperser, the calcined beads have diameters between 30 and 150 µm, i.e., they are essentially dust free, and their aerodynamic diameter is so large that transportation is not possible. This polydisperse size distribution of the beads facilitates their compaction (typically at pressures between 300 and 600 MPa) to give pellets, which, when sintered in a suitable atmosphere, reach densities greater than 90% of the theoretical value (TD). This process was tested at the JRC-ITU in the 1980s for the production of the fuels for the SUPERFACT irradiation experiment [6] where MOX fuels incorporating Am and Np were produced. Extension of this process to Cm is possible, but very rigorous cost evaluation and technical processes to deal with and recycle such highly active neutron and alpha emitting waste need development. Furthermore, the need to recycle Cm needs further evaluation.

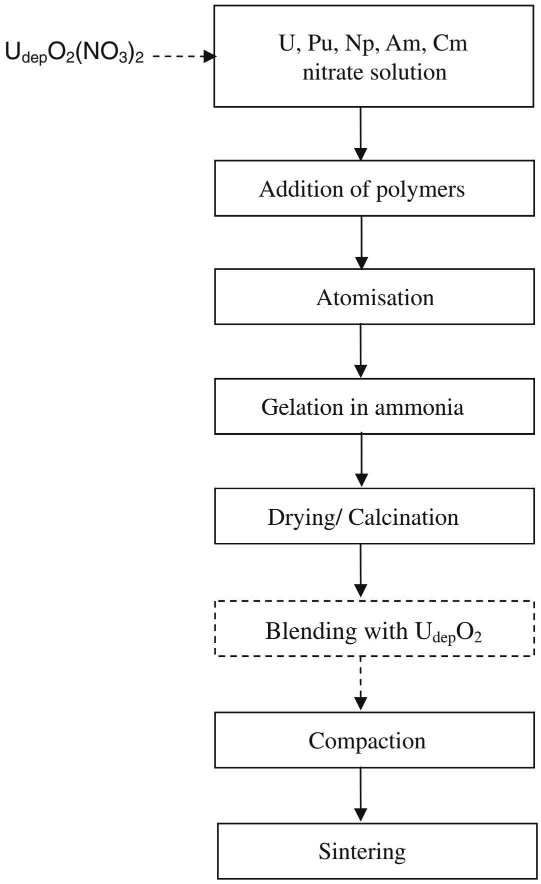

Fig. 1. Schematic flow sheet of the sol–gel fabrication process.

2.1.2. The infiltration method

At the ITU a hybrid process consisting of a combination of sol–gel and porous bead infiltration techniques has been developed [5]. It meets three critical criteria: (1) it is dust free, (2) there are no liquid wastes, and (3) it minimises the steps in which highly radioactive materials are handled. This process not only meets the criteria listed above but also is flexible and easily adapted to the requirements and specifications of new fuel compositions. Indeed, it has been implemented in the fabrication not just of oxide fuels but it is also the reference process for nitride fuel fabrication, whereby additional carbothermal reduction steps are required.

2.1.3. Composite fuels

The fabrication of composite pellets, in which the actinide oxides are dispersed in a ceramic (cercer) or metallic (cermet) uranium free matrix, is considerably more difficult than for solid solution oxides pellets, but offers considerable flexibility in the fuel design. Additional specifications arise due to the particle size and homogeneous distribution of the dispersed actinide phase, which is in fact the major problem to be solved, and has now been tested extensively and mastered for the fabrication of both cercer and cermet pellets. Particles containing the actinide phase are prepared by a combination of the sol–gel, and infiltration methods. Thereafter they are mixed with the matrix powder by conventional blending methods. Following compaction into pellets by cold pressing, they are sintered at high temperature. The effect of important parameters related to composite pellet fabrication, such as the volume fraction of ceramic (actinide phase) and the size of the ceramic particles, have been systematically investigated and reported in detail elsewhere [7,8]. Several irradiation tests have been and are being performed using both oxide and metal matrix fuels [9–11].

2.2. Mixed metal nitride and carbide (MX) fuels

2.2.1. Conventional process

Carbides, carbonitrides and nitrides can be produced by the general reaction given in Eq. (1).

$$\begin{aligned}&(\mathrm{U,Pu,MA})\mathrm{O}_2 + (3-x)\mathrm{C} + 0.5x\mathrm{N}_2\\&\quad\rightarrow (\mathrm{U,Pu,MA})\mathrm{C}_{1-x}\mathrm{N}_x + 2\mathrm{CO}\end{aligned}\tag{1}$$

where MA stand for minor actinides.

For nitrides this reaction is performed under flowing nitrogen and requires high temperatures, in excess of 1500 °C. In the case of the pure carbide, $x = 0$, the powders are heated directly in Ar, or preferably under vacuum. The progress of the reaction can be monitored by the CO content in the process gas stream emanating from the furnace. It should be noted that preparation in the carbon rich domain is difficult as it requires strict control of the N_2 partial pressure and the remaining reaction conditions. This reaction has been widely used for the production of U/Pu mixed metal carbides, nitrides and carbonitrides.

Unfortunately, the reaction to produce the carbides or nitrides is not as clean as indicated in Eq. (1) and shown in the flow sheet in Fig. 2. Superstoichiometric oxygen in UO_{2+x} reacts with carbon at relatively low temperatures (<1000 °C) to form CO_2. Therefore the full reaction to manufacture a carbide is given by Eq. (2).

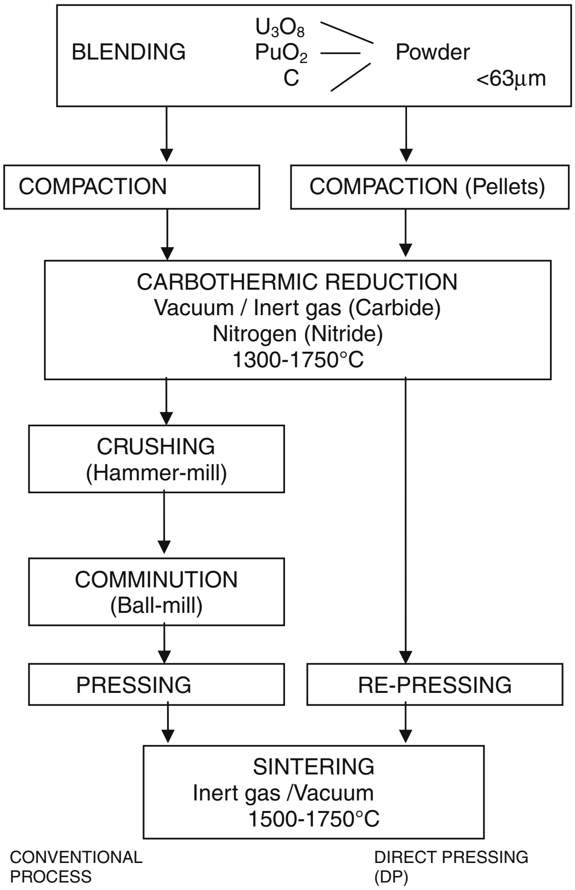

Fig. 2. Comparison of conventional and direct pressing method for MX production.

$$(1-z)UO_{2+x} + zPuO_2 + \{3+n+0.5x(1-z)\}C \rightarrow (1-2n)(U_{1-z}Pu_z)C + n(U_{1-z}Pu_z)_2C_3 + 2CO + \{0.5x(1-z)\}CO_2 \quad (2)$$

The production is complicated in the stoichiometry by the O/U ratio of the starting material and the possibility of forming the sesquicarbide M_2C_3. Considerable adjustment of experimental parameters is needed [12,13]. It was ITU experience that manufacturing carbides under vacuum (1 Pa) gave better results than when the reaction was performed under Ar. The reaction temperature was 100 °C lower, and less N impurity was obtained [14] (mainly due to the 10 ppm N_2 impurity in the Ar stream). During the manufacture of the carbides, Pu losses as high as 10% were observed [12,15]. This is unacceptable, not only for economic efficiency, but also because of a criticality risk, should their accumulation occur locally in an industrial plant. The Pu is not lost, but rather is plated out on the cold surfaces of the furnaces. Richter et al. [12] showed that increasing the CO partial pressure in the furnace during the carbothermal reduction step from 0.8 to 7 Pa could reduce the Pu losses from 7.2% to 0.2%.

The rate or efficiency of the carbothermal reduction depends intrinsically on the homogeneity and intimacy of the blend of the U and Pu oxides and carbon powders. Milling times of up to 36 h have been mentioned [16]. Poor intimacy in the mixture results in local impurities. If the carbon is deficient or in excess, either oxide or carbon impurity persist in the final product. The carbothermal reaction is favoured by high surface area powders (>10–15 $m^2 g^{-1}$) and small agglomerate sizes (<15 μm). Much can be gained in the preparation of the starting powders themselves and specifically for this purpose Louwrier et al. [17] developed a highly active PuO_2 powder derived from an oxalate precipitation, whose particle size was 2–3 μm, and surface area was 20 $m^2 g^{-1}$.

The manufacture of pellets from carbide powders prepared by carbothermal reduction is a difficult step. The carbothermal reduction step is often made on compacts pressed at relatively low pressure. This sustains the intimacy of the powder blend, but the compact products have to be crushed in a hammer mill and then ball milled to produce a powder reactive enough to be pressed again to yield a high density pellet. Sintering of the pellets is conducted at temperatures of 1650–1800 °C, either under Ar or vacuum. The quantity of oxygen and secondary sesquicarbide phases present in the product pellet is an interplay between the initial powder to carbon ratio and the product density itself. Increasing the carbon to oxide ratio certainly decreases the oxide impurity in the product, but leads to the presence of sesquicarbides in the products. In addition, it appears that higher density pellets have more oxygen, carbon and sesquicarbides present. The carbothermal reduction, if not completed, or if oxygen is taken up during the ball milling, can continue during the pellet sintering step. The elimination of porosity and densification of the pellet can play a role in this process. If there is no open porosity, the CO cannot be released, and equilibrium can be reached causing the formation of more sesquicarbides and leaving oxygen. Such an effect is always more likely in the bulk or central region of the pellet. At low pellet densities, inhomogeneities in the sesquicarbide distribution are undoubtedly due to the starting material preparation. This may be limited by the production scale, which is small in laboratories compared to industry [18]. Incorporation of MA in such production campaigns has not been done on any significant scale.

2.2.2. Direct pressing method

In the conventional carbothermic reduction-press-sinter procedure, the reaction product is comminuted and transformed into a reactive powder by ball milling. For glove box processing the comminution step involves powder handling and dust formation which can lead to a considerable radiation exposure hazard for the operator, and safety problems due to risk of self-ignition of the pyrophoric MX powders, in case of an accidental air ingress.

In the early 80s an advanced process, the so-called direct pressing (DP) process, was developed at ITU [19–21]. This fabrication process is also based on the carbothermal reduction, but without comminution. The fabrication flowsheet of this process is compared with the conventional carbothermal reduction method in Fig. 2. In this case, an oxide–carbon mixture is pressed into a geometrical shape which is adapted to the desired final geometry of the fuel pellet. Compacts of this mixture are thermally treated at 1400–1700 °C in an appropriate atmosphere, to achieve the carbothermal reduction. During this step, the desired chemical composition is obtained in a pre-determined shape with a bulk density of 40–50% TD. These compacts are then compacted again without undergoing an intermediate comminution step. Their density then increases to 65–75% TD. These directly pressed pellets are given a final thermal treatment at 1600–1800 °C to achieve final specifications of density and dimensions.

The main advantages of the direct pressing route are

No handling of MX powders with a high affinity for oxygen, and obviously lower oxygen content in the product.
Considerable reduction of the risk of self-ignition of pyrophoric MX powders, in case of an accidental air ingress in the facility.
Considerable reduction in dust produced.
Reduction in the incorporation of metallic impurities from ball milling steps.
Ease of material flow – pellets rather than non-flowable powders.
Fissile material inventory monitoring facilitated.

Process requires less time (and equipment), i.e., is more economic. The process has been successfully used and demonstrated for a variety of fuel irradiation experiments, such as NILOC (see below).

2.2.3. Carbon integration method

Recently, in an attempt to simplify the multistage powder carbothermic reduction pellet route for nitrides, a modified fabrication route, also based on the carbothermic reduction, has been developed and tested. As already mentioned, the main problem in fabricating nitrides by the conventional route is the comminution step which produces dust, and should be avoided, especially when handling minor actinides. Thus, the possibility of fabricating high density nitride pellets without the comminution step has been further investigated. In this case, beads of the required composition are fabricated by the sol–gel method but the feed solution is prepared by adding the carbon powder to the aqueous solution. This suspension is then atomised in the standard way. The beads are washed and dried as described above. Finally they are calcined at 800 °C under Ar/H_2 to convert the hydroxides to oxides without pyrolysing the carbon. Once calcined the beads are free-flowing and due to their small size and integrity they do not produce dust during the remaining fabrication steps. This method has the advantage that the contact between the metal oxide and the carbon is intimate as it is dispersed throughout the beads thus there is no need to compact the material into discs. In this case the carbothermal reduction is performed directly on the beads. Unlike the conventional method, these beads are directly pressed without crushing and milling steps, and the resulting pellets are sintered to obtain the final product.

This process has the advantage of total elimination of fine powder handling. Also, mixing the carbon in the starting solution guarantees a homogeneous dispersion of carbon, which is an essential condition for carrying out a solid state reaction effectively. This process avoids dust-producing comminution and granulation stages. The shorter process minimises contamination and also exposure of the operators to ionising radiation.

This method has been used to fabricate $(U_{0.81}Pu_{0.17}Am_{0.02})N$ pellets. In this particular case, $(U, Pu)O_2$ microspheres containing carbon were fabricated by the sol–gel method in an inert atmosphere to prevent oxidation of the added carbon. Then they were infiltrated with an americium nitrate solution and treated thermally to give $(UPu)O_2$–AmO_2 microspheres.

Due to the small particle size of the americium infiltrant (<2–3 μm) and the high sinter activity of the sol–gel material, a solid solution is readily attained at temperatures less than 1200 °C. The carbothermal reduction is then applied, and the resulting nitride powder pressed and sintered to produce pellets.

3. Fabrication facilities for MA-containing fuels

When dealing with transuranic elements, the use of gloveboxes is required to prevent risk of incorporation of highly toxic radioactive materials into the body. In addition, MA will require further biological shielding. Lead provides shielding from γ-emitters and a combination of water, lead and cadmium (or boron) provides shielding from neutrons.

The glovebox operating atmosphere should be inert. The level of inertness depends on the fuel composition. For oxide fuels, the O/M ratio is an important fuel design parameter and sintered product materials should be maintained in relatively inert atmospheres with less than 1% O_2 and preferably with less than 100 ppm H_2O. In contrast, the preparation of nitrides and carbides poses more severe atmospheric requirements. Both react readily with O_2 and H_2O, which is detrimental to the quality of the product in terms of oxygen impurity, which increases steadily during processing. A much more severe problem is the pyrophoricity of these materials, being particularly important for carbides in all geometries, but most important for finely divided powders with a high surface area. Thus, the handling of nitrides and carbides requires utmost attention and provision of highly chemically inert atmospheres in the gloveboxes. Typically, the O_2 and H_2O contents should be maintained below 10 ppm. Tests performed at the ITU have indicated that either Ar or N_2 can be used as inert gas with similar success. Given the economic factors, the choice is purified N_2 which provides an adequate and entirely acceptable solution.

Incorporation of the minor actinides in the fuel necessitates shielding, and concomitant automation of the entire process all the way through to assembly and loading in the reactor. In addition, the reprocessing and fuel fabrication for Gen-IV fuels should be integrated in a single installation at the reactor site. While today's MOX plants for pellet production are highly developed with extensive (actually complete) automation, they are so designed that operator intervention can be made via the gloveboxes. (pin and assembly fabrication are not as extensively automated). Minor actinide presence in the fuel can necessitate use of hot cells or hybrid laboratories, combining glovebox standards with remote operation by telemanipulators [22]. Transposing MOX fuel fabrication philosophy will probably not be satisfactory for another reason, namely dust. Both Melox in France and SMP in the UK base their fabrication on the use of powder metallurgy methods. While dust is an issue in that it must be cleaned up, it poses no severe or insurmountable problem. Introduction of minor actinides leaves dust not just as an undesirable nuisance, rather an unacceptable problem. For minor actinide fuels, processes devoid of dust production, such as the infiltration method, are essential. An advanced process has been adapted and qualified in the Minor Actinide Laboratory (MA-Lab) at ITU. It consists of a chain of seven conventional glove boxes, shielded by 50 cm water and 5 cm lead, for which the limiting masses are 150 g of ^{241}Am or 5 g of ^{244}Cm. Here the fabrication process is restricted to infiltration, pellet pressing, sintering, metrology, visual inspection, and pin filling, welding and control. Operation is achieved by telemanipulators and remote control. An extension of the facility to permit sol–gel processing is also planned.

4. Fuel irradiation testing programmes

The fuel performance in pile is a key issue; thus different irradiation programmes were launched in various fast reactors (Dounreay, Rapsodie, and most recently Phénix). An overview of some of these programmes including the aim for the experiment, fuel fabrication and PIE results when available is given below. Some experiments are complete including post-irradiation examination while others are still ongoing.

4.1. SUPERFACT

The SUPERFACT experiment was carried out in the Phénix reactor for 383 EFPDs from 1986 to 1988 and provides data on the behaviour of mixed oxide fuel in a homogeneous fuel matrix with significant Am and Np content. It consisted of four UO_2 based pins with MA compositions containing up to 45 wt% Np and 20 wt% Am irradiated to a burn-up of 4.5 at.%. The experiment also contained two fuel pins each of composition $(U_{0.74}Pu_{0.24}Am_{0.02})O_2$ and $(U_{0.74}Pu_{0.24}Np_{0.02})O_2$, that are representative of those envisioned for future transmutation fuels. All fuels were fabricated by the sol–gel method [23]. Photographs depicting the macro and microstructure of a SUPERFACT fuel are shown in Fig. 3.

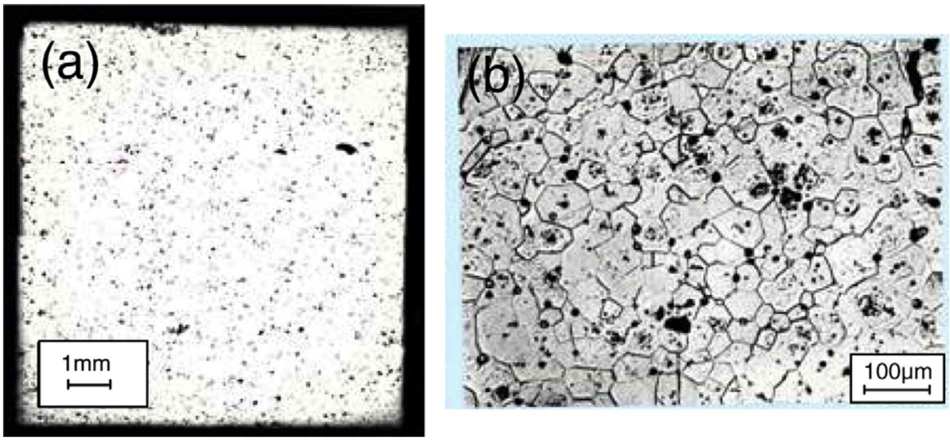

Fig. 3. Micrographs of a SUPERFACT fuel fabricated by the sol–gel method indicating: (a) macrostructure and (b) microstructure.

Three of the four fuel compositions performed well with only the fuel with a very high concentration of Am (20 wt%) exhibiting the onset of fuel clad mechanical interaction and some clad constituent migration into the fuel region. All of the test compositions exhibited high fission gas release as expected in fast reactor oxide fuel due to high centerline temperatures and high mobility of helium and other inert gases.

4.2. NIMPHE

The NIMPHE (NItrure Mixte dans Phénix à joint HElium) irradiation experiment in the Phénix reactor set out to compare the influence of three different fabrication routes (conventional carbothermal reduction and direct pressing of pellets and granules) on the irradiation performance of nitride (and carbide) fuels. The fuels of this experiment had densities between 80% and 85% TD. The NIMPHE 1 experiment consisted of a capsule containing eight ($U_{0.8}$, $Pu_{0.2}$)N pins and 11 Phénix standard ($U_{0.8}$, $Pu_{0.2}$)O_2 pins irradiated at medium linear power (45 kW/m). The nitride pin geometry was the Phénix standard, i.e., pellet and pin diameters of 5.45 and 6.55 mm, respectively. NIMPHE 2 consisted of a capsule containing five ($U_{0.8}$, $Pu_{0.2}$)N and two ($U_{0.8}$, $Pu_{0.2}$)C pins irradiated at a higher linear power of 73 kW/m. The pin geometry was the Superphenix standard with pellet and pin diameter of 7.11 and 8.5 mm, respectively.

The NIMPHE fuel was extensively studied in its as-fabricated stage so that some indicators were drawn about the influence of the fabrication route on the fuel stability. The direct pressing of pellets is a better means to generate pellets with improved thermal stability; however, the starting materials, the reaction temperature and the pressing conditions all influence the thermal stability. The direct pressing of granules decreases the densification under irradiation.

Non-destructive examination of 11 pins of NIMPHE 1 was carried out in 1993. Destructive examination is available on one pin of NIMPHE 1. At the moment PIE of NIMPHE 2, (U, Pu)C and (U, Pu)N fabricated by direct pressing and irradiated at a linear power of 73 kW/m with a burn-up of 5.8 at. %, is being carried out.

4.3. NILOC

The NILOC (NItride Low in Oxygen and Carbon) irradiation experiment set out to investigate the structural evolution of (U, Pu)N pellets with oxygen and carbon content below 500 ppm at the begin of life and to compare the influence of the fabrication process on the in pile behaviour. In all, four capsules containing 11 pins were irradiated in the HFR-Petten with a targeted burn-up of 0.5 at.%.

Two capsules (NILOC-1 and NILOC-2) containing three pins each were fabricated with Phénix and Superphénix pellet diameter. The fuel pellets were fabricated by the conventional carbothermal reduction with powder communition, by direct pressing method and a modification of the latter, the so-called granulation method, in which ‘clinkers’ of the nitride fuel are produced in the form of granules and these are directly pressed for sintering. Macrographs of the pellets fabricated by the different methods are shown in Fig. 4. The PIE showed a clear effect of the fabrication process on the irradiation behaviour, in terms of fuel densification, fission

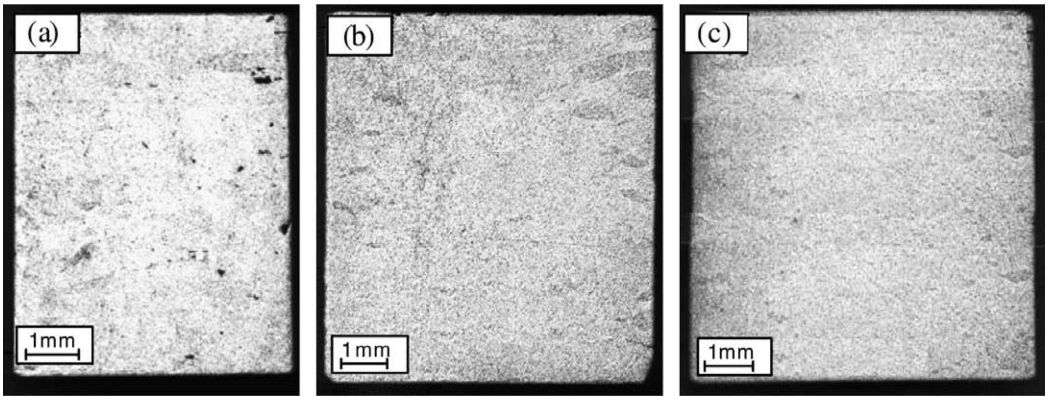

Fig. 4. Macrographs of axial sections of UPuN pellets fabricated by (a) conventional carbothermal reduction (b) direct pressing and (c) granule pressing methods.

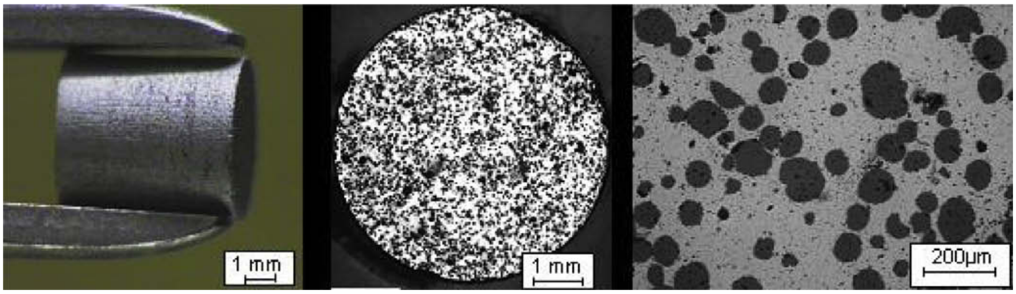

Fig. 5. Visual aspect and ceramographs of a FUTURIX sample $(Pu_{0.8}Am_{0.2})O_2$ + Mo.

gas release, and themochemical stability [2]. As an example, the fuel fabricated by the conventional method shows the largest grain growth and it is less stable than the direct pressing fuel.

4.4. FUTURIX

The purpose of the FUTURIX-FTA international irradiation programme is to demonstrate the feasibility of burning minor actinides in dedicated reactors including critical electricity-generating fast reactor transmuters or hybrid systems (e.g., Accelerator Driven Systems). Eight pins including metallic, nitride, CERMET and CERCER forms prepared by INL, LANL, ITU and CEA, respectively, are being irradiated in the PHENIX reactor starting in 2007 for 240 EFPD. This experiment will provide essential data concerning behaviour under irradiation in a fast spectrum and will allow qualification and validation of models developed to predict fuel performance. At ITU, two molybdenum based cermet fuels have been fabricated by the combination of the infiltration of sol–gel beads with conventional powder mixing methods. A $(Pu_{0.8}Am_{0.2}O_2$ + Mo) pellet is shown in Fig. 5. The ceramographs show an even distribution of the actinide ceramic phase in the Mo matrix.

5. Conclusions

ITU has a great deal of experience in fabrication, characterization, and irradiation and in post-irradiation examination of fast reactors fuels based on uranium and plutonium. In the past years, advanced processes to fabricate fuels containing minor actinides have been developed and tested. At present, an irradiation programme with $PuAmO_2$–Mo cermet fuel has been launched in the Phénix reactor. Favourable results have been obtained on the fabrication of (U, Pu, Am)N by the advanced process based on the combination of the carbon integration and infiltration methods and are promising for future development. Ongoing programmes now consider both homogeneous and heterogeneous recycling of MA in fast reactor systems, as well as alternative fuel forms (SPHEREPAC) which could alleviate some of the difficulties encountered in fabrication.

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