



ASGARD

Advanced Fuels for Gen IV Reactors: Reprocessing and Dissolution

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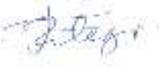
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DM	WP		
DM 1 <input type="checkbox"/>	WP 1.1 <input type="checkbox"/>	WP 1.2 <input type="checkbox"/>	WP 1.3 <input checked="" type="checkbox"/>
DM 2 <input type="checkbox"/>	WP 2.1 <input type="checkbox"/>	WP 2.2 <input type="checkbox"/>	WP 2.3 <input type="checkbox"/>
DM 3 <input type="checkbox"/>	WP 3.1 <input type="checkbox"/>	WP 3.2 <input type="checkbox"/>	WP 3.3 <input type="checkbox"/>
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CONTENT

- INTRODUCTION 4
- 1 PROJECT SUMMARY FOR THE SECOND YEAR 8
- 2 DOMAIN SUMMARIES FOR THE SECOND YEAR..... 11
 - 2.1 Domain 1 - Management, education and training, dissemination 11
 - 2.2 Domain 2 – Oxide fuels 12
 - 2.3 Domain 3 – Nitride fuels 15
 - 2.4 Domain 4 – Carbide fuels 16
- CONCLUSIONS 19

INTRODUCTION

The ASGARD project was constructed to bridge the scientific societies working in the area of recycling of nuclear waste. Traditionally these areas have acted rather separately in European framework funded projects even if some projects e.g. ACSEPT originally focussed on separation issues did also have work performed in conversion issues. The main parts of the circular nuclear fuel cycle are shown in Figure 1 together with some of the major EU funded projects in relation to the ASGARD scope.



Figure 1 The ASGARD project in relation to the steps in a circular nuclear fuel cycle and in relation to other important projects in the domain.

Essentially the circular nuclear fuel cycle consist of: the spent fuel being dissolved in a suitable matrix (usually nitric acid). Then the elements to be further used in production of new nuclear fuels (the actinides) are separated from the rest of the waste by either hydrochemical or pyrochemical techniques. In ASGARD we mainly aim at the hydrochemical techniques, but a smaller part on specialised fuels are also discussed with respect to pyrochemical methods. After the separation, the product has to be converted to suitable precursors for fabrication of the new fuel whereupon the fabrication process starts. It is in this area where the major part of the work of ASGARD is focussed. In future nuclear systems it is possible to use several different types of fuels whereas today oxide fuels dominate completely. It is expected that regardless of fuel type the processes outlined in Figure 1 will be necessary and thus in ASGARD we have decided to divide the work in domains given by the fuel types and tasks relating to the specific questions arising for that particular fuel type, see Figure 2.

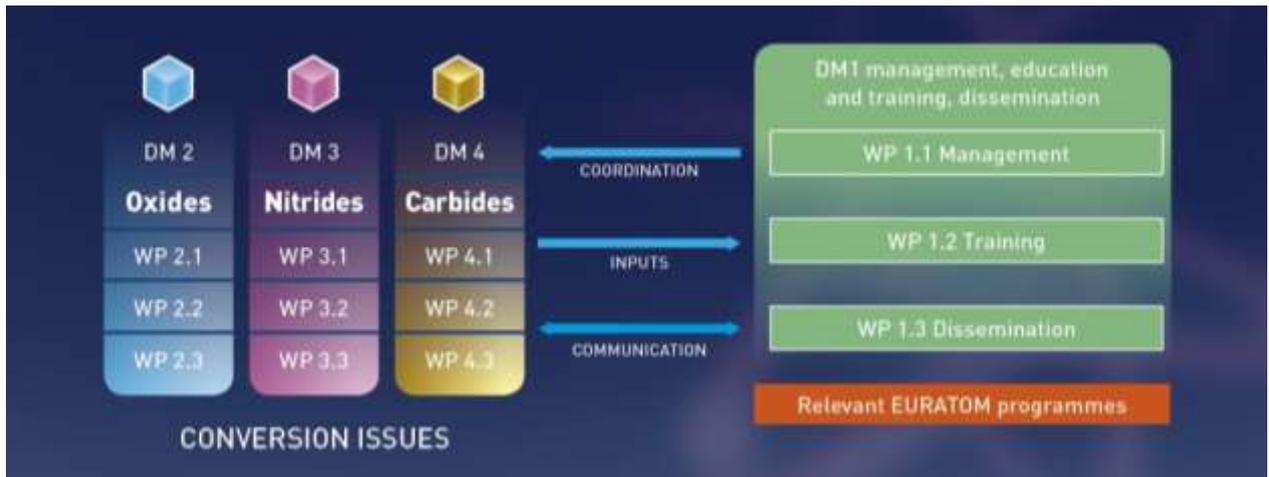


Figure 2 General structure of the ASGARD project.

As seen in Figure 2 there is a clear cross cutting activity when handling the conversion issue. There are also other specific issues that will cut cross the domains in more specific areas.

The work package division for each domain is given in Figures 3-5 below.

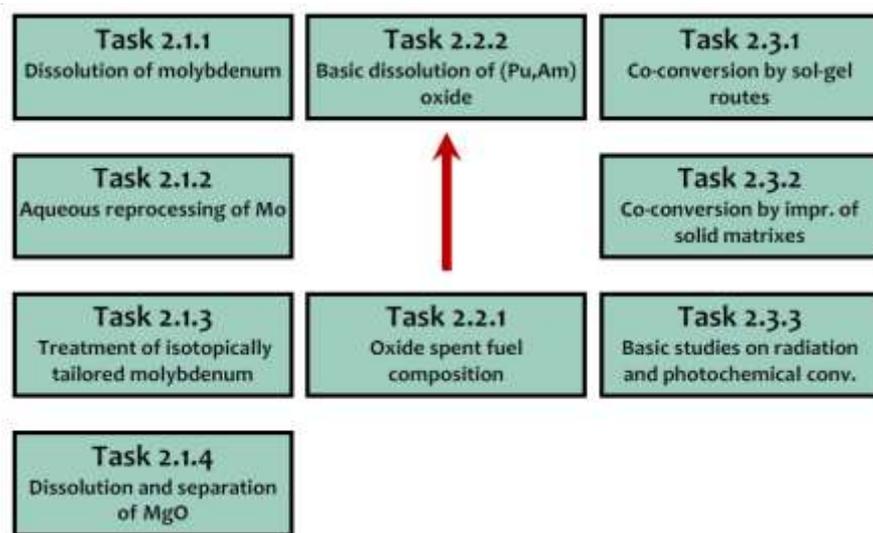


Figure 3 The organisation of the oxide domain.

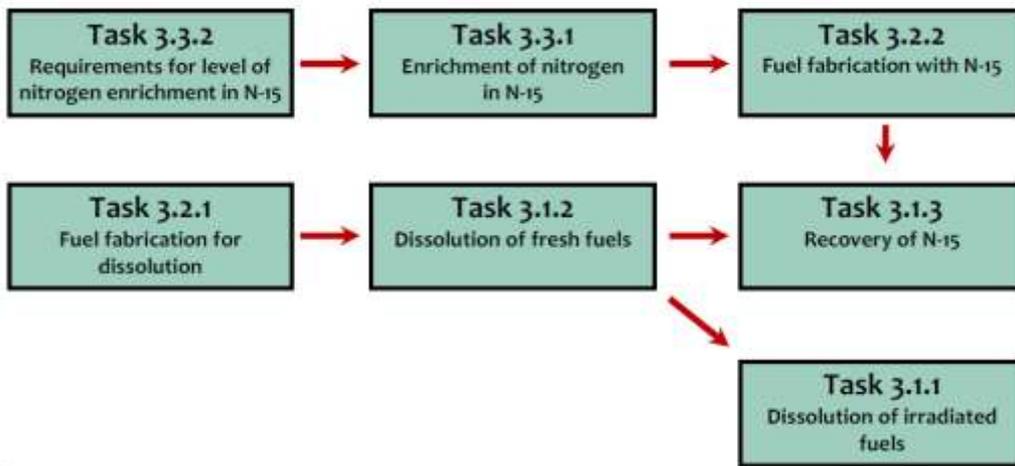


Figure 4 The organisation of the nitride domain.

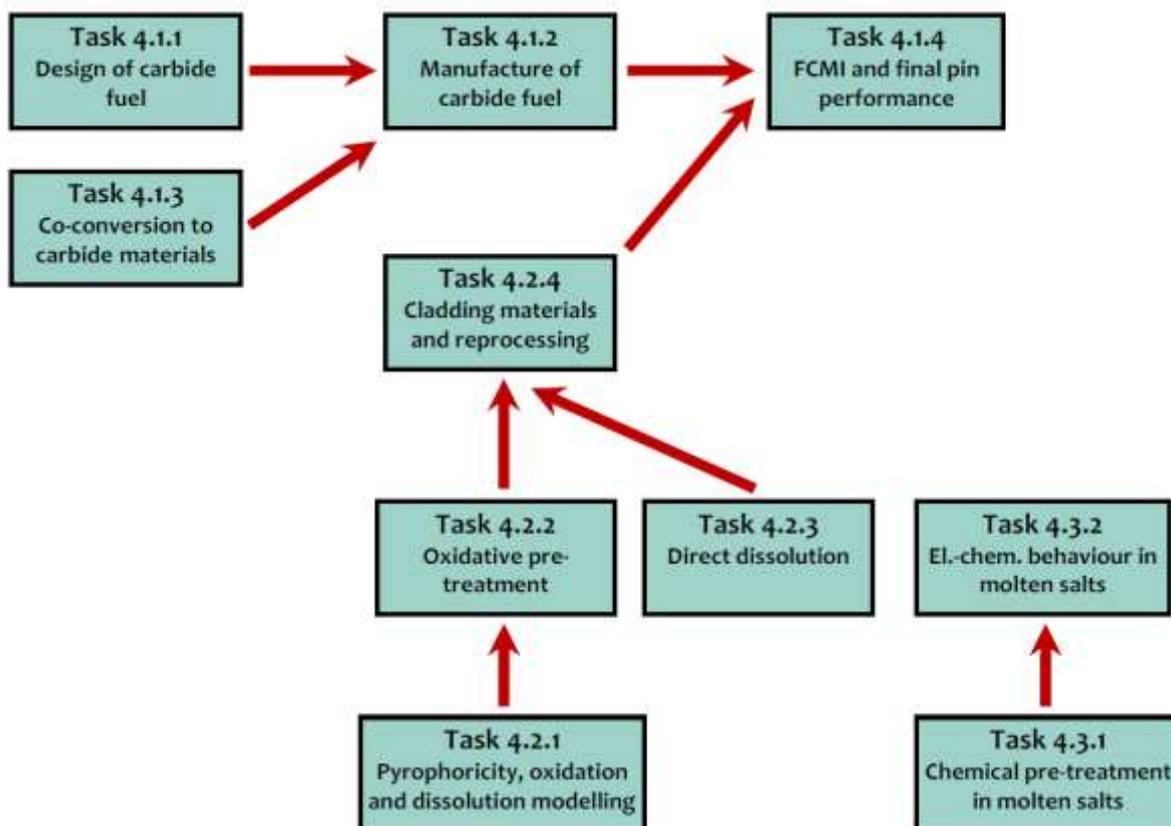


Figure 5 The organisation of the carbide domain.

Within the ASGAR project, 17 partners from 9 countries work together well distributed within the domains described above. The participation of the main nuclear actors in Europe will ascertain that the project will deliver scientific results of the highest standard in a structured and useful way. The last part is being addressed by a selected

Scientific Advisory Board and End Users Group making sure that the results of the ASGARD project are in line with what the future industry can accept at the same time as it is on a high scientific level.

Last but not least the ASGARD project has an extensive scheme for training and education of younger researchers. This program comprise not only dedicated lectures and summer(winter)schools, but also funding possibilities for shorter or longer visits to perform work in other laboratories than their own. In addition, it is also possible to receive funding to present ASGARD work at international meetings and conferences thus promoting not only the aspect of broadening the experiences of the younger researchers but also serves as an excellent tool for disseminating the results of the ASGARD project.

1 PROJECT SUMMARY FOR THE SECOND YEAR

The ASGARD project has now finished its second year of operation. Being the first of its kind joining the communities of fuel fabrications and separation/dissolutions science there was a rather long start-up period during the first year. This time is especially needed since a considerable amount of the work to be performed deal with significant amount of highly radioactive matter and thus rigorous safety cases have to be performed before any work can commence. This, however, was planned and thus the first year went according to the plans set from the beginning. As a result, now during the second year we start to see result not only in DM1 but also in the scientific domain. An example of the robust planning of the project and the dedication of our partners is that although the NRG had to shut down all work for several months this will not affect the overall time plan of ASGARD. Some work could be shifted around to accommodate for this unexpected delay and no real delay in deliverables will follow.

For the DM1 we have had one winterschool together with the other FP7 projects FAIRFUELS and CINCH given by one of our industrial partners, Westinghouse, with an emphasis on safety aspects related to dissolution, conversion, reprocessing and fuel fabrication under normal and accident conditions. Another Winter School has been held in Stockholm in January 2014 having as subjects fuel characterization and isotope separation. In addition, a continuous feed-back and eventual improvements with regard to safety and handling of materials will be established and implemented throughout the project. The key learning points will be collected, documented and presented at the end of the project. Further collaboration with other projects such as SACESS will be undertaken in the future showing a clear added value for all partners included and at the same time create a good atmosphere and contacts especially in the younger scientist community across Europe.

Successful travel and mobility support have been developed and implemented in ASGARD under DM1 named Travel Fund, aimed at allowing young scientists, students and trainers to disseminate and network into the nuclear community, as well as allowing access to relevant facilities. So far, 16 grants have been approved, of which 4 were for mobility to other laboratories, 1 for a trainer mobility and 11 for summer/winter school and conference participation.

Outreach of ASGARD project is measured by DM1 in numerous publications in peer-review journals, as well as conferences and public media. In addition to this more than 20 scientific publications have produced so far.

In the scientific domains a considerable amount of work has been performed in all the directions of ASGARD and a more detailed account of these are given in the later sections of this report. However, some short, selected results are given here.

For domain 2 the fuel fabrication and dissolution of both CERCER and CERMET fuel have been performed. The understanding of the effect of molybdenum during dissolution (in Mo containing CERMET fuel) has been increased by detailed chemical experiments on molybdenum chemistry as well as practical enhancements in the dissolution by adding e.g. iron to the dissolution liquor. Similar experiments were performed for magnesium containing CERCER fuel where the dissolution of fabricated pellets have shown details on the kinetics and how the pellets is affected by different leaching environments. The dissolution experiment of the irradiated $(\text{Pu}_{0.8}\text{Am}_{0.2})\text{O}_2$ in Mo matrix is finished. Sol gel fabrication methods research focused mainly on increasing of amount of neodymium up to 40% as surrogates of americium. Increasing of neodymium causes increasing of nitrates in gel, so new method -Double Extraction Process - was introduced.

For nitride fuels, i.e. DM3 the progress has mainly been in 2 areas. Already during the first year pellets were produced using both a dry and a wet routes showing two complementary production procedures that give acceptable qualities of the final pellets. The fabrication of isotopically pure N-15 is progressing and has shown good results and in parallel a “loss-less” fabrication process for nitride fuels has been developed. These two in combination show good progress towards the fabrication of cheaper, isotopically pure nitride fuel. By doing this the production of C-14 during the reactor cycle will be minimised. However, since the Gen IV fuels are supposed to be recycled the dissolution step is also crucial. If normal HNO_3 dissolution is used the enriched N-15 will be lost and exchanged in the acid. Thus a pre-treatment has been developed where the N-15 can be recovered before the actual fuel dissolution commences. These promising results solve many of the issues relating to industrial use of nitride fuels.

In DM4 where the carbide fuel chemistry is investigated both front and back end related issues have been addressed. It is clear that carbide powder is pyrophoric and thus fabrication needs to be done under controlled atmospheres. A significant effort has been put into understanding this pyrophoricity and to make experiments investigating its behaviour. A detailed understanding of this process has been obtained through 2D and 3D computer modelling.

In the case of reprocessability two main routes have been investigated, direct dissolution and oxidative pre-treatment. For direct dissolution of uranium carbide experiments have been performed and previously unknown organic materials that are generated in solution have been identified. A clear effect on the subsequent separation process has been observed. The kinetics of carbide dissolution has been explored for actual unirradiated UC fuel pellets. For the oxidative pre-treatment route issues relating to volatility of several fission products have been observed on irradiated carbide fuel. Thermodynamic calculations have also shown that using CO_2 to oxidise $(\text{U,Pu})\text{C}$ can limit the amount of insoluble PuO_2 residue left when the pre-oxidised material is dissolved.

Molten salt treatment of carbide fuels has also been assessed, especially with respect to the cladding material. A literature review has been performed and it was clear that the cladding material can be oxidised in molten salts

Christian Ekberg

ASGARD Coordinator

2 DOMAIN SUMMARIES FOR THE SECOND YEAR

2.1 DOMAIN 1 - MANAGEMENT, EDUCATION AND TRAINING, DISSEMINATION

The management of the project, sustainable education and training and dissemination of the knowledge achieved within ASGARD project are addressed in Domain 1 in three work packages (WPs).

The objective of WP1.1 is to assure efficient co-ordination of technical activities of ASGARD project, quality assurance of the project's deliverables and the processes leading to them, effective overall legal, contractual, ethical, financial and administrative management of the project and management of the project's decision-making processes to respect relevant standards. Establishment of internal communication within the partners is also one of the tasks.

The main objective of WP1.2 lead by CTU is to stimulate exchange of knowledge and practical experience among the community and future researchers. Students (BSc, MSc and PhDs) represent the primary target group. Teachers and other members of the community will benefit from ASGARD activities and measures in area of education/training and mobility.

Within the ASGARD project (www.asgardproject.eu), two summer/winter schools are planned to take place. The first Winter School 2013 was held on 29 – 30 January 2013 at NRG, Petten, the Netherlands. This course dealt with 'Fabrication methods & Irradiation performance' and was organized in cooperation with the 7TH FP projects FAIRFUELS (<http://www.fp7-FAIRFUELS.eu>) and CINCH (<http://cinch-project.eu>).

Students and young professionals working in the nuclear sector were welcomed to participate to this Winter School.



Figure 6 Winter School 2013 lecturers, held 29 – 30 January 2013 at NRG, Petten, the Netherlands

Another even which has been organized by ASGARD project during this period of time is the first Training Session dedicated to ASGARD participants.

The Training Session has been successfully held in January 2014 at ASGARD progress meeting in Stockholm, having KTH as host venue. The Training session has been delivered by KTH (Stockholm, Sweden) and INCDT (Cluj-Napoca, Romania) experts in fuel preparation and isotope separation. The training session has welcomed an

ASGARD

11 of 19

impressive number of project members.

The Travel fund is operating very well and in this reporting period, a number of 10 applicants were requesting and were granted funds for a) course participation, b) trainer travelling arrangements, c) mobility (four young researchers working on other laboratories), d) conferences.

The main objectives of WP 1.3 lead by NNL are to guarantee dissemination of knowledge of the project and results from the project into the nuclear community. The projects webpage (public) is the first achievement: <http://asgardproject.eu/>. Also, the ASGARD corporate identity, project logo, brochure and leaflets have been produced.

A series of at least 14 press releases and 25 conference contributions and publications have been the result of the latest months.

2.2 DOMAIN 2 – OXIDE FUELS

As Inert Matrix Fuels contains significant amount of inert matrix (e.g. MgO, Mo) special head end treatments after dissolution are necessary. On the other hand, it is necessary to remove the inert material to simplify the separation of the actinides and to improve the final conditioning of the remaining fission products before vitrification.

The Mo(/CeO₂) pellets are made and characterized. Mo pellet dissolution was performed at varied temperature and varied HNO₃ and Fe(NO₃)₃ concentrations. Dissolution progress was followed by ICP-MS analysis of the solution. Addition of Fe(NO₃)₃ was shown to accelerate dissolution. Increasing temperature resulted in faster dissolution but also in the formation of a precipitate.

The mixed Mo-Fe species in solution could explain the enhanced dissolution kinetics of Mo-metal in the presence of Fe(III). Furthermore, redox reactions between molybdenum and iron seem to play a role. Eu(III) that was added to the Mo solutions as analogue for Am(III) also forms mixed species with Mo in nitric acid solutions. Comparable Mo-Am solution species could have implications on the reprocessing of the Mo matrices.

Isotopically pure ⁹⁸MoO₃ was dissolved in HNO₃ in the presence of ⁹⁰Zr(IV) (as analogue for Pu(IV)). The solution was analysed by means of ESI-MS, showing the formation of mixed Mo-Zr complexes. In cooperation with FZJ-IEK6 ⁹⁸Mo metal was dissolved in HNO₃. Besides Mo(VI), also Mo(V) was identified in the dissolution solutions.

First steps towards the development of a measuring protocol to analyze solutions of molybdenum with natural isotopic composition with a commercially available ESI/MS/MS instrument (QTrap) were undertaken. The stability of the detected species, the influence of the solvent acetonitrile (AcN) on the relative abundance of polymolybdates, and the influence of the variation of the dilution factor for two solvents (water and 25% can) were considered. The mechanism of the dissolution of MgO has

been investigated. The evolution of the MgO pellet surface during dissolution has been studied. Mixed MgO/CeO₂ pellets have been characterized and their dissolution behavior has been investigated as a function of acid concentration and temperature.

The dissolution experiment of the irradiated (Pu_{0.8}Am_{0.2})O₂ in Mo matrix is finished. Different dissolution conditions (temperature, time, chemicals (HNO₃, and/or HF and/or Ag (II) etc.) are tested to try to dissolve the material completely. Four attempts were realized, none of them led to a complete dissolution of the fuel. The use of Ag(II) instead of HF in the nitric acid solution was explored, the reaction is at first quite vigorous. As no regeneration of the silver (II) has been installed, reaction is slowing down with the time and the black residue present in the reaction vessel remains. This residue will be investigated by SEM/WDS. Samples have been taken all along the reaction. The treatment of those sample for the recovery of the dissolved Pu will be done to evaluate the kinetics of the dissolution. That will be done by using a thermal ionization mass spectrometry (TIMS).

Several materials were screened for separation of Cs from Mo solutions. KNiFC and KCuFC appear to be most suitable. From these materials, composite absorbers on a PAN matrix were prepared and tested. Caesium sorption studies in dynamic conditions were finalized from the molybdenum solution with pH 9.1 and total molybdenum concentration 100 g/L. Practically usable dynamic sorption capacities were found for the KNiFC-PAN composite absorber with potassium-nickel hexacyanoferrate active component. The Tc hot experiments have been started by implementation and validation of the analytical method for ⁹⁹Tc. The assumption about the possible increase of molybdenum solubility in concentrated nitric acid due to the formation of molybdenyl ion was not confirmed in dissolution studies.

The fabrication and characterization of the UO₂/Mo pellets is finished. The pellets can be transported to partners for further dissolution studies.

MgO pellets were dissolved in HNO₃ (varied concentration, temperature, and stirring speed). Pellets having varied MgO/CeO₂ ratios were pressed, sintered, and characterised. These pellets were dissolved at varied HNO₃ concentration and temperature. Dissolution solutions were analysed. While MgO easily dissolves in HNO₃ ≥ 4 mol/L, nearly no CeO₂ dissolves.

For the fabrication of (Am, Pu)O₂ pellets, AmO₂ powder is necessary. KIT made AmO₂ powder available for this project. Transport of this powder from KIT to NRG was prepared and took place in M18.

In the co-conversion by sol-gel routes task, internal gelation method concentrated on characterization of reduced powders & comparison to the particles by XRD, on detailed SEM investigations of the particles (before and after thermal treatment) and thermal treatment of the dried particles in air atmosphere and SEM studies. Internal gelation

studies finished the XRD measurements of the reduced powders and comparison to particles prepared by internal gelation was finalized.

Complex Sol-Gel Processes focused on optimization and characterization of the preparation of sols solutions with molar ratio $ASC/UO_3=1$ and doped by Nd (MR $U/Nd=0,1$) as surrogates of MA. Sol gel fabrication methods research focused mainly on increasing of amount of neodymium up to 40% as surrogates of americium. Increasing of neodymium causes increasing of nitrates in gel, so applying of Double Extraction Process was needed. However to prepare perfect spherical particles of gel by modification of Double Extraction Process was needed. The gelation step focused on optimization and characterization of this step. Preliminary experiments of thermal treatment (calcination and reduction) of ascorbate-uranyl gel kernels obtained by internal gelation have been carried out. An optimization of reduction to uranium dioxide has been carried out. They focused on decreasing of time and temperature of reduction step.

The ion exchange resins Amberlite IRC-86 and Lewatit TP-207 are loaded with UO_2^{2+} and Nd^{3+} . Various parameters have been investigated to maximize the adsorption. The adsorption kinetics of UO_2^{2+} , Nd^{3+} and a mixture of both ions have been studied. In addition, the temperature influence and the effect of the pH on the adsorption of UO_2^{2+} and Nd^{3+} have been investigated. The kinetic studies show a significant faster adsorption of Nd^{3+} compared to UO_2^{2+} . A pH lower than 3 causes a decrease of the adsorption. Acid-deficient uranyl nitrate (ADUN) solutions can be used to maximize the pH of the uranyl nitrate solution without the introduction of foreign cations.

The study of photo- induced formation of uranium and/or thorium oxides focused on setup optimal conditions. The second objective was increasing the yield of both uranium and mixed uranium-thorium oxides, that could be achieved by i) increasing concentration of uranium and/or thorium in solution, ii) addition of hydrogen peroxide or iii) using other sources of radiation Concentration of U and/or Th nitrate in solution was increased to 0.01 mol/dm^3 . Solutions of U and/or Th nitrate were irradiated via accelerated electrons. Prepared solid phase was calcined under air or $Ar+H_2$ atmospheres and nanocrystalline oxides were formed and subsequently thoroughly characterized. Oxides prepared under various conditions were prepared in amount sufficient for pressing the pellets. The method of uranium and/or thorium oxides preparation was optimized. Europium and mixed europium-uranium oxide were prepared under UV radiation from solutions containing europium nitrate and ammonium formate.

Within the ASGARD consortium, cooperation between the institutes is running successfully. Mainly young scientists perform a part of their (PhD) research at institutes of Asgard partners using the specific laboratory equipment and studying different fabrication methods. Several new publications related to ASGARD project were presented.

2.3 DOMAIN 3 – NITRIDE FUELS

PUBLISHABLE SUMMARY FOR DOMAIN 3 – DAR 3

In domain 3, NRG has carried out dissolution tests of irradiated CONFIRM (Pu,Zr)N fuel samples. The tests confirm that (Pu,Zr)N does dissolve in nitric acid under the adopted conditions, albeit slowly. The composition of insoluble black residues is to be investigated.

The dissolution of unirradiated (Pu,Zr)N samples is ongoing at PSI.

At INCD-TIM, a high performance catalyst for reduction of sulphuric acid to sulphur dioxide has been identified. Tests have shown that α -Fe₂O₃ may provide higher conversion rates than more expensive alternatives. N-15 enrichment tests under varying pressure are proceeding according to plans.

The development of a method for recovery of N-15 using steam processing of UN is proceeding ahead of schedule at KTH.

At Chalmers, ZrN pellets have been manufactured using carbothermic nitriding of zirconia microspheres with carbon nanotubes in solution.

Fabrication of UN at KTH and (Pu,Zr)N at Chalmers is behind schedule due to problems with equipment. These problems have been resolved and sample fabrication is expected to place in mid-2014.

Two papers treating impurity control in fabrication of ZrN and (Pu,Zr)N samples have been authored by KTH, and are now published in Journal of Nuclear Materials. These papers form the basis for the PhD thesis of Merja Pukari, which was presented in June 2013.

List of publications:

- Merja Pukari, Experimental and theoretical studies of nitride fuels, PhD thesis, KTH (2013).
- Merja Pukari, Masahide Takano, Sintering and characterization of ZrN and (Dyy,Zr)N as surrogate materials for fast reactor nitride fuel, Journal of Nuclear Materials, & Volume 444, Issues 1-3, January 2014, Pages 7-13
- Merja Pukari, Masahide Takano, Tsuyoshi Nishi, Sintering and characterization of (Pu,Zr)N, Journal of Nuclear Materials, Volume 444, Issues 1-3, January 2014, Pages 421-427

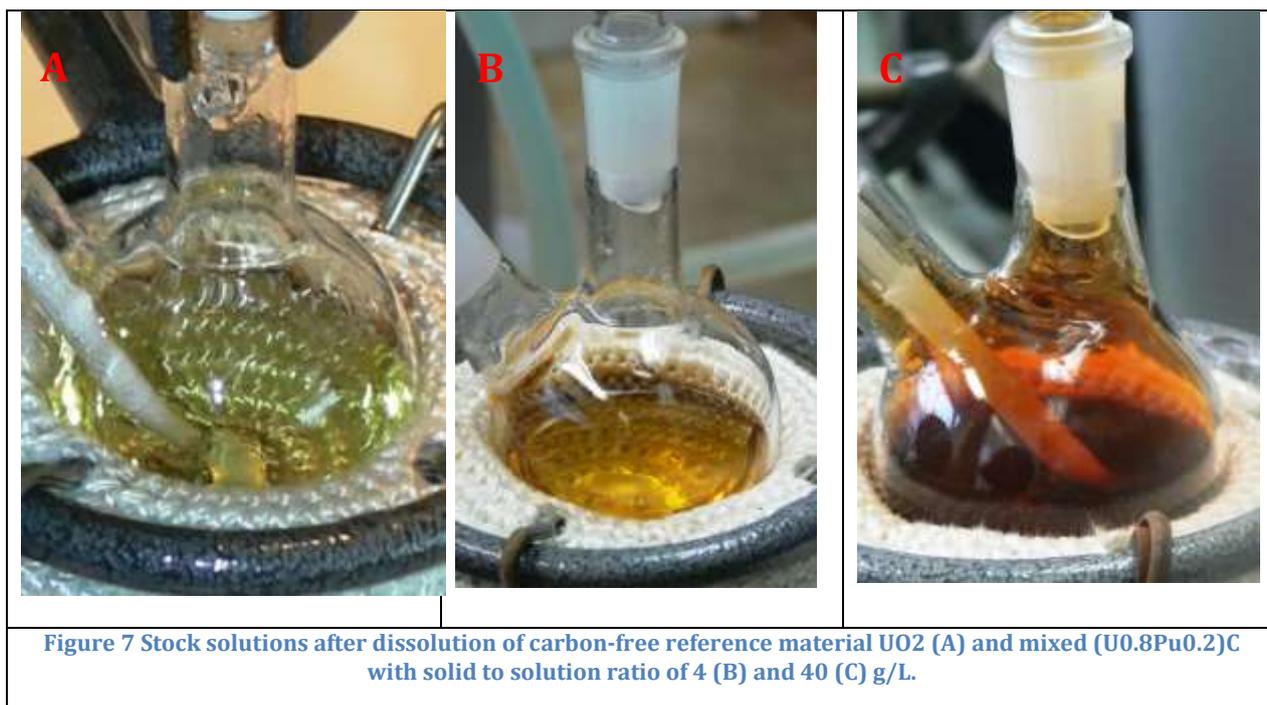
2.4 DOMAIN 4 – CARBIDE FUELS

Carbide fuel pellet design modelling has defined the dimensions and creep stress enhancement material levels resulting in a pellet that is expected to exhibit reduced fuel swelling compared to a reference material by about 50% by controlling the in reactor temperature profile of the fuel pin. FCMI is also expected to be reduced as a consequence of creep rate enhancers designed into the fuel. Sphere-pac fuel size fractions that result in reduced swelling properties are defined and will be fabricated using microwave internal gelation techniques and the equipment for these tasks is being commissioned.

Methods of producing carbide materials that reduce the temperature of carbothermic reduction and minimise the need for milling and grinding have resulted in promising results, one of which has been submitted for a patent. Other novel methods for the production of uranium carbide material have been demonstrated but require further carbon stoichiometry control. The compatibility of uranium carbide with barrier material for gas cooled fast reactors has been demonstrated to 2000°C using high temperature mass spectrometry experiments.

Studies on the pyrophoricity of finely divided uranium carbide powders have identified key reaction steps and strong exotherms that decrease as the oxygen concentration is lowered. Modelling of 3 mechanisms for uranium carbide oxidation have been made and will be compared to experimental data. A 2D and 3D chemical model of the process is being developed to provide a tool for the industry in establishing a safe operating envelope.

One option for the reprocessing of (U,Pu)C fuels is pre-oxidation of the carbide followed by dissolution in nitric acid, but the oxygen potential needs to be carefully controlled to avoid the generation of insoluble plutonium rich phases. The oxidation of (U,Pu)C in CO₂ atmosphere has been demonstrated and shown to conform to thermodynamic calculations. Dissolution tests of the resultant (U,Pu)O₂ material show lower levels of insoluble PuO₂ residues compared to MOx dissolution (Fig. 1) and the lack of highly coloured organics compared with direct dissolution of (U,Pu)C.



High temperature heating of irradiated (U,Pu)C fuel has shown how volatile fission products behave under oxidising atmospheres and allow for some quantification of the release fractions from the fuel.

The direct dissolution of carbide fuels in nitric acid is another possible reprocessing route. The generation of CO₂ and soluble organic material that can interfere with solvent extraction efficiencies has been verified. The kinetics of dissolution of titanium carbide barrier material have shown similar partitioning of carbon between the gas and liquid phases with the production of insoluble species at high nitric acid concentrations (> 12M). The mechanism and kinetics of dissolution for 70g UC fuel quality pellets is currently underway. The identification of previously unidentified soluble organic compounds has provided a breakthrough in understanding how to destroy the problematic organics (Fig. 2)

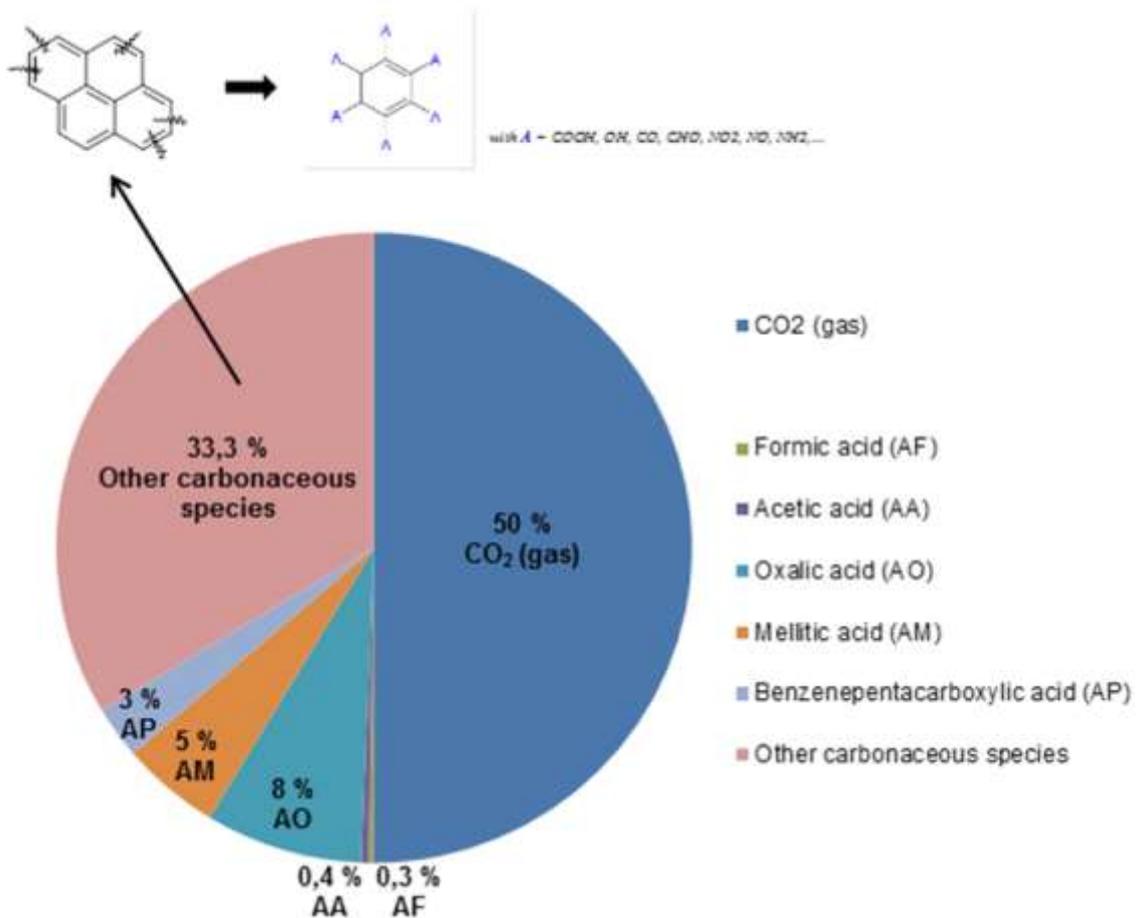


Figure 8 Mass balance of the carbon released from UC during dissolution in 8.5M nitric acid given a final solution containing 4.6M HNO₃ and 215g/L uranyl nitrate.

Molten salt processing of carbides is an alternative method to oxidative pre-treatment and has been demonstrate simply using molten salt + oxygen donor materials and the conversion of SiC and TiC to oxides has been demonstrated.

CONCLUSIONS

It can be concluded that despite the fact that the ASGARD project despite dealing with considerable amounts of radioactive material, have not encountered any significant delays or problems during its two years of operation. The robustness and flexibility of the project coordination committee and the partners was shown by the fact that despite a several moth shut down at one partners the work was divided and rescheduled in a way that no significant diversions from the original plan occurred.

The education and training activities are working smoothly and successfully and an increase in the activities are planned through a more intense collaboration with other projects in relevant areas. This has already shown to be a good and successful pooling of efforts to create an added value to all projects involved, especially creating a critical mass and a good contact net for the younger scientists in different disciplines.

The technical domains have shown significant progress in all aspects of the work. Due to the very nature of the ASGARD project the greater bulk of practical work on fuel fabrication will be during year 3 and 4 while basic studies lying the foundation for the understanding of the underlying processes was made during year two. This has been completed in a very confident way and thus the work to be performed has a very good possibility to be conducted smoothly, safely and successfully.