

TECHNOLOGY FOR MANUFACTURING DISPERSION NUCLEAR FUEL AT IPEN/CNEN-SP

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ABSTRACT

IPEN/CNEN-SP has been working for increasing radioisotope production in order to supply the expanding demand for radiopharmaceutical medicines requested by the Brazilian welfare. To reach this objective, the IPEN's IEA-R1 research reactor power capacity has been continuously increased starting from 2 MW aiming to reach 5 MW. In addition, another research reactor with power substantially greater than that of the IEA-R1 will be constructed in Brazil, the Brazilian Multipurpose Reactor – RMB. Since 1988, IPEN has been manufacturing its own fuel element, initially based on U₃O₈-Al dispersion fuel plates with 2.3 gU/cm³. To support the reactor power increase and the new research reactor, higher uranium density into the fuel had to be achieved for better irradiation flux and also to minimize the irradiated fuel elements to be stored. Uranium silicide was the first chosen option. The U-Mo alloy is considered for future use. This paper describes the results of this program and the current status of silicide fuel fabrication and qualification, as well as the status of UMo-Al dispersion fuel development.

1. INTRODUCTION

The use of radioisotopes in medicine is certainly one of the most important social uses of nuclear energy and IPEN/CNEN-SP has a special place in the history of nuclear medicine in Brazil. Due to the federal monopoly, only the Institutes that belong to CNEN (Comissão Nacional de Energia Nuclear) can produce radioisotopes and radiopharmaceuticals for use in nuclear medicine. The production of IPEN represents nearly 98% of the total demand.

There has been a significant increase in the demand of radioisotopes over the years. Between 2002 and 2004, the increase in the demand was about 30%. Distributed to hospitals and clinics throughout Brazil, the radiopharmaceuticals produced at IPEN serve 3.5 million patients per year with an annual increase in demand of about 10%. This is the scenario that drives IPEN in their goal to continuously increase its production of radiopharmaceuticals and bring the benefits of nuclear medicine to the whole country.

In 1981, this institute announced the production of ^{99m}Tc generators with its own technology, but importing the ⁹⁹Mo ever since. The use of ^{99m}Tc covers 80% of all nuclear imaging procedures for diagnostic, which represents approximately 30 million procedures annually in the world. The Brazilian demand for this radioisotope has grown significantly in Brazil,

reaching today more than 320 generators per week (450 Ci), which corresponds to 4% of global consumption of ^{99}Mo , with an import cost of U.S.\$ 20 million per year, this situation was for the year 2009 [1]. Most of the raw material (^{99}Mo) comes from Canada due to the low power of the IEA-R1 reactor, which has insufficient capacity to produce the required amounts of the primary ^{99}Mo radioisotope.

Currently, the world's supply of ^{99}Mo is centered in the operation of only five research reactors, which are aged around 40 years and, therefore, are not capable of reliable operation. Recently, the shutdown of the Canadian NRU reactor (National Research Universal Reactor) triggered a global shortage of medical radioisotopes, a situation particularly problematic in the medical point of view. This recent crisis in the supply of ^{99}Mo has profoundly affected the distribution of generators $^{99}\text{Mo}/^{99\text{m}}\text{Tc}$ in Brazil and encouraged the project's launch RMB - Brazilian Multipurpose Reactor in 2008. The Brazilian Multipurpose Reactor aims to make the country independent in the production of radioactive isotopes for medicine. The new reactor shall be six times greater in power than the main research reactor producer of radioisotopes in operation in Brazil, the IEA-R1 reactor of IPEN. The success of RMB Project will require the development of nuclear fuels with high uranium concentration. The silicide technology can reach a LEU¹ uranium density up to 4.8 gU/cm^3 . The UMo-Al dispersion is being researched worldwide and is considered the fuel of the future, with the ability to reach a LEU uranium density of 6.9 gU/cm^3 . This is the future objective.

Once the IEA-R1 reactor power was planned to be raised from 2 to 5 MW, in 1997 IPEN started research activities aiming at the elevation of the uranium loading inside the fuel, in order to allow the reactor power increase. This was accomplished with the development of the silicide technology, which allowed IPEN to fabricate fuel plates with a uranium density in fuel meat of 3.0 gU/cm^3 . Until this time, 28 silicide fuel elements (3.0 gU/cm^3) had been irradiated at the IEA-R1 research reactor. The irradiation was closely followed by visual inspections and sipping tests. After 40% burn up (average), no problems regarding to fuel performance was recorded. The enriched U_3Si_2 powder was imported from the international market until 2002, when IPEN started the development of the conversion technology to get de U_3Si_2 powder using national enriched UF_6 produced by CTMSP (Marine Technological Center in São Paulo). Nowadays, IPEN is able to fabricate the enriched U_3Si_2 powder, allowing the nationalization of all the fabrication cycle of dispersion fuel for research reactors. After mining and enrichment steps, IPEN is able to execute all the other fabrication steps. On June 26, 2007 the first fuel element fabricated with Brazilian materials and technology was put in operation at IEA-R1 reactor core. This work emphasizes IPEN's quest to acquire uranium silicide technology reaching the development level of high uranium density fuels in a worldwide standpoint.

1.1. Historical Background

The beginning of the development of the fuel element fabrication technology in IPEN is very old. The work started in 1960 aiming at fabrication of the fuel for the ARGONAUTA research reactor. Between 1964 and 1965, the fuel elements were manufactured with 20% enriched U_3O_8 powder provided by IAEA in the program Atoms for Peace. In spite of the low technological demand of the ARGONAUTA fuel (very low power), a seed was planted and

¹ LEU is the acronym of Low Enriched Uranium, containing with less than 20wt% of ^{235}U , which is the maximum level assuring no proliferation of military use.

allowed the germination 20 years later, in the decade of 80, and to bloom definitively in the 90's decade, when IPEN dominated the know-how technology and began the production of the fuel for the IEA-R1 research reactor. The relative high power (2 MW) demanded a significant technological progress in the fabrication techniques.

Starting from 1980, IPEN intensified its efforts to develop the fabrication technology of dispersion fuel element, aiming at improving the technology for manufacturing more advanced fuels, substantially superior to the old ARGONAUTA fuel type. At that time, IPEN could not acquire fuel elements from the international market in order to supply the IEA-R1 research reactor. The growing difficulty to get fuel elements in the international market acted as an initial impelling force for IPEN to deflagrate their program for fuel element fabrication. The technology previously developed in the 60's was updated starting from 1985, based in the recent technological advances in this area. Between 1985 and 1988, IPEN worked in assembling a small fuel fabrication facility at a laboratory level, with capacity to produce 6 fuel elements by year. This was enough to supply the IEA-R1 reactor operating at 2 MW and 40 hours a week.

On August 31, 1988 as part of the commemorations of its 32nd anniversary, IPEN provided the IEA-R1 reactor with the first fuel element fabricated in Brazil, only fifteen days before the exhaustion of the reactor fuel. The fissile material used was the same U_3O_8 powder previously used for the production of the ARGONAUTA fuel. There was a reserve of about 30kg of this material at IPEN's material stock. Starting from 1988, after the production of the first fuel element, IPEN began a continuous fuel element production, which has continued until nowadays.

After producing 26 fuel element, the enriched U_3O_8 powder finished in 1996. So, in 1994, IPEN started developing the processes for UF_6 conversion to U_3O_8 and for recovering the uranium scraps generated in fuel plate fabrication. In 1996, IPEN did the conversion of about 20kg of imported enriched UF_6 . IPEN was then prepared for the routine production of fuel elements starting from UF_6 as raw material. In 1997, IPEN raised the fuel production capacity from 6 annual fuel elements to 10, which was the maximum considering the available infrastructure.

In order to increase the radioisotope production of IPEN, the IEA-R1 reactor power capacity was increased from 2 MW to 5 MW. In 1997, the development of a new higher uranium density fuel started, in order to meet the reactivity needs for continuous operation and, to have a compact core for better irradiation flux and also to have a low number of irradiated fuel elements to be stored at the spent fuel pool. The new fuel was based on the U_3Si_2 -Al dispersion with uranium loading of 3.0 gU/cm^3 . In 1998, the fuel plate fabrication technology of the new silicide fuel was implanted. At that time, the U_3Si_2 powder was imported from France. Between 1999 and 2000, 16 silicide fuel elements were manufactured. Starting from 1998, the efforts to develop the U_3Si_2 powder production technology began, aiming at the nationalization of all the production process, starting from enriched UF_6 leading to the conversion to UF_4 ; its reduction to metallic uranium; U_3Si_2 powder fabrication and, finally, arriving at the fuel plate fabrication and fuel element assembly.

With the help of IAEA (International Atomic Energy Agency), in 1999 IPEN got the technology for UF_4 production using $SnCl_2$ as reducing agent. In the area of metallic uranium, IPEN had a valuable previous experience in producing 150kg natural uranium

ingots in 90's. Based on the previous experience, IPEN initiated efforts aiming at scaling down the size of the metallic uranium pieces; trying to produce pieces of about 3kg using 20% enriched material as raw material for the U_3Si_2 production. In 2002, the process for producing metallic uranium was technologically dominated, which allowed the development of the U_3Si_2 intermetallic. In 2004, IPEN obtained the first lot of natural U_3Si_2 powder, manufactured with national technology, dominating then the "uranium silicide cycle". In 2006, IPEN consolidated the fabrication technology of the silicide fuel by manufacturing the first fuel element with full Brazilian technology. This fuel element was put in the IEA-R1 reactor on June 26, 2007.

In 2008, with the support of FAPESP (Fundação de Amparo à Pesquisa do Estado de São Paulo), IPEN started efforts to develop and qualify high loaded fuel elements with U_3Si_2 -Al dispersions, aiming at obtaining the maximum uranium concentration for silicide technology, which is 4.8 gU/cm^3 . This goal has achieved at the present (2011) by fabricating 4 miniplates containing 4.8 gU/cm^3 using 20% enriched uranium (LEU) [2]. The irradiation of two miniplates started at IEA-R1 reactor on June 2011 for fuel qualification. In addition, two full sized silicide fuel plates with 4.8 gU/cm^3 were also fabricated for complete demonstration of the technology. These fuel plates will be assembled in a partial fuel element to be irradiated in the IEA-R1 reactor, in order to complete the qualification of the new fuel.

Also in 2008, under the same FAPESP Project, IPEN started studying the UMo-Al dispersion fuel. Because the ductile characteristic of the UMo alloy, the powder production is the most challenging step of the fuel production. The studies for powder fabrication started in 2007 [3] and was successfully concluded in this year [4]. The produced UMo powder (400g) allowed the fabrication of the first UMo-Al prototype dispersion miniplate with natural uranium. The next step is to conclude the fuel development and produce miniplates and full sized fuel plates with LEU enriched uranium in order to qualify this fuel under irradiation.

Due to the emergent increase in radiopharmaceuticals demand and the consequent increase in the IEA-R1 power, the reactor needed an increase in fuel production, from 6 (U_3O_8 -Al) to 18 elements (U_3Si_2 -Al) a year. In addition, the new RMB research reactor would consume about 50 annual U_3Si_2 -Al fuel elements. Therefore, a demand for about 70 annual fuel elements seems to be quite realistic. Based on this forecast demand for fuel elements, IPEN began a project in 2001 seeking to adapt the production facilities in order to improve the producing capacity. This project is nowadays under way aiming at replacing the current researching laboratory facility by a fully production one with industrial characteristics. That will meet the fuel element demand requests. The producing capacity of the new facility should reach 80 annual fuel elements, which would supply also the new RMB research reactor. The conclusion of this new fabrication facility is foreseen for 2012-2013.

2. FUEL DEVELOPMENT

The IEA-R1 Reactor of IPEN/CNEN-SP is a pool type reactor operating since 1957. This reactor uses MTR type dispersion fuel element in a 5 X 5 core arrangement. The Nuclear Fuel Center of IPEN is responsible for the production of the necessary nuclear fuel to keep the continuous operation of the reactor. Development of new fuel technologies is also a permanent concern. The Nuclear Fuel Center had produced 92 fuel elements until now, including 14 control fuel elements.

The fuel meat is fabricated according to conventional powder metallurgy techniques. The fuel element contains 18 fuel plates, each one 1.52 mm thick. Cladding and frame plates are made with the ASTM 6061 aluminum alloy. The fuel assembly is performed by a well-known picture-frame technique. The fuel element results from mechanical assembling of 18 fuel plates and other structural components. Fig. 1 shows the details of the plate fabrication and fuel element geometry.

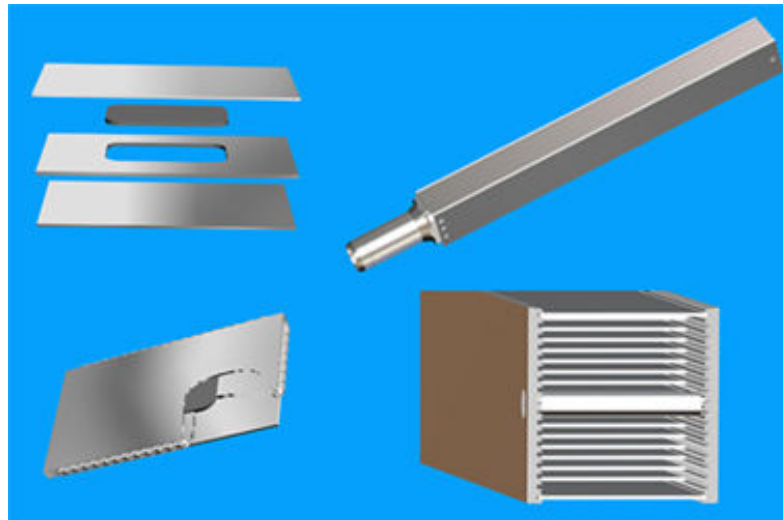


Figure 1. Fuel element fabricated at IPEN.

As, in Brazil, hot cell laboratories to test irradiated fuels are unavailable and as the irradiation tests abroad would be very expensive, IPEN decided to risk the testing and evaluating in pile its own fuel, checking the performance under reactor operation. This was possible since IPEN fuel specification is conservative for dispersion fuel and the reactor power is relatively low. In July 1985, a partial fuel element with only two fuel plates (the external plates) and with 16 aluminum plates was placed in the core to start fuel qualification. After this, other partial fuel element with 10 fuel plates and 8 aluminum plates was also placed in the core (November 1985). These two fuel element were identified as the precursor fuels. A periodic monitoring and evaluation was done on them. After successful results with these precursor fuels, it was decided to start loading standard fuel elements in the reactor core (August 1988), with $1.9 \text{ gU/cm}^3 \text{ U}_3\text{O}_8\text{-Al}$. The adopted criteria was that each IPEN fuel element had to start irradiation at peripheral positions of the core, with lower power densities, up to 4% burnup (almost one year of irradiation) and then it can be placed at higher power density positions in the core. It was decided that the precursor fuels should stay in core during the same time that a complete fuel element would stay. All precursor fuel assemblies were taken out from the reactor core without any operational problems. Previous paper detailed about this fuel qualification program [5]. To qualify the $\text{U}_3\text{Si}_2\text{-Al}$ fuel the same strategy was adopted. In this case, the volume fraction of the fissile material in the dispersion was kept with the same amount, around 27% in volume, resulting in a uranium loading of 3.0 gU/cm^3 .

The program for silicide fuel development was made, as a primary purpose, to develop the whole fabrication process of $\text{U}_3\text{Si}_2\text{-Al}$ dispersion fuel plate with its irradiation test at the IEA-R1 reactor followed up by post-irradiation analysis. This program proposal would give the

necessary background to IPEN to produce and qualify its own U_3Si_2 powder and silicide based dispersion fuel plates for the IEA-R1 fuel element fabrication. Minor changes in this program were necessary to develop the UMo fuel, including alloy production and powder fabrication. Fig. 2 shows a flow-sheet of the main program activities. The program steps to achieve the objectives are:

- to develop the process for producing UF_4 starting from UF_6 ;
- to develop the process for producing metallic uranium starting from UF_4 ;
- to develop the process for producing U_3Si_2 powder;
- to produce miniplates with 20% enrichment for irradiation tests;
- to irradiate miniplates at the IEA-R1 reactor and to perform non-destructive analysis on the irradiated fuel miniplates inside the spent fuel pool.

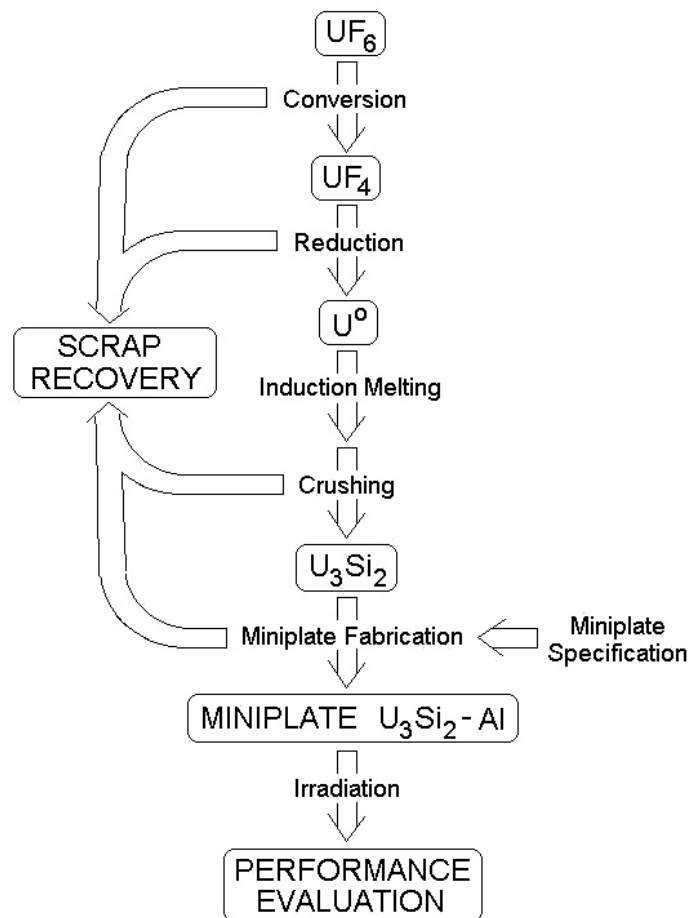


Figure 2. Main development program activities.

2.1. UF_6 Conversion Towards UF_4

Essentially, the process to get UF_4 from UF_6 uses the reduction of hexavalent uranium present in aqueous solution to the tetravalent state and its precipitation as UF_4 by means of the HF solution. The preparation of UF_4 using chemical reduction has been carried out starting from UO_2F_2 solution resulting from UF_6 hydrolysis. The solution is heated under continuous

stirring to reach a temperature set point and then the reducing agent added. Next, the hydrofluoric acid HF precipitating agent solution is slowly added. Tests have been carried out using different reducing agents, such: SnCl_2 , CuCl , FeCl_2 , and $\text{Na}_2\text{S}_2\text{O}_4$. The reducing agent SnCl_2 was the one that showed the best results and achieved a level of UF_4 precipitation in the range of 98%. However, during the UF_4 preparation an amount of water is absorbed by the UF_4 crystal. This water could interfere in the other process steps. To avoid problems during magnesiothermy to produce metallic uranium it must be removed.

The elimination of crystallization water from the UF_4 is carried out in temperatures near 400°C , under a constant flow of argon over the surface of the powder, which avoids the UF_4 oxidation to UO_2F_2 or UO_2 and drag the water vapor released. The material, in a powder form, is placed inside a small Monel boat and put in the heating chamber. After purging with argon for 1 hour the system is heated to 400°C under flow of gas. The UF_4 powder remains in this temperature for 1 hour, which is sufficient time to complete the water elimination. The subsequent cooling is carried out keeping the flow of argon till the temperature reaches 100°C . Details of the UF_4 precipitation procedures were presented in a previous work [6]. Fig. 3 presents a typical representation of the UF_4 morphology.

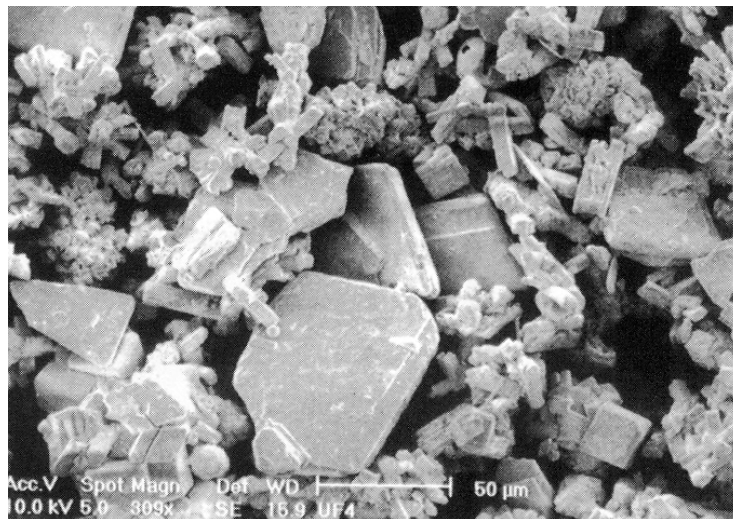


Figure 3. UF_4 crystals (SEM micrography).

2.2. UF_4 Reduction to Metallic Uranium

The intermetallic U_3Si_2 is produced from metallic uranium. To produce metallic uranium for the required quantity (up 80 kg/year), IPEN has developed a process to produce small quantities of uranium (1000g) through magnesiothermic routine. The 1000g ingot of metallic uranium is rather small if compared to previous practice of IPEN. In 1980's, ingots of natural uranium ranged up to 100kg. The downscaling was difficult, since not only the reduced scale for the crucible was conceived, but also lower material amount to be reduced implied in difficulties to get reasonable metallic yields. The crucible itself has been planned several times to achieve the final format using a bottom extractable system to help removing the cast ingot from the crucible. The thermal profile of the furnace has already been achieved by

experimentation and calculations, but to reach optimized yields it was necessary to identify the exact moment of the reaction. It was got by means of an accelerometer in order to recognize the sound waves perturbation during the reaction moment.

Optimization of this method was met only after 25 experiments with natural uranium simulations. Once settled down this technology, IPEN started to produce the 20% low enriched uranium (LEU). The yield of this system is around 82-85% of metallic uranium. The thermal cycle used nowadays is indicated in the diagram of Fig. 4. The enriched material was already produced with good operation and quality results. Fig. 5 shows the enriched ingots produced by our IPEN methodology.

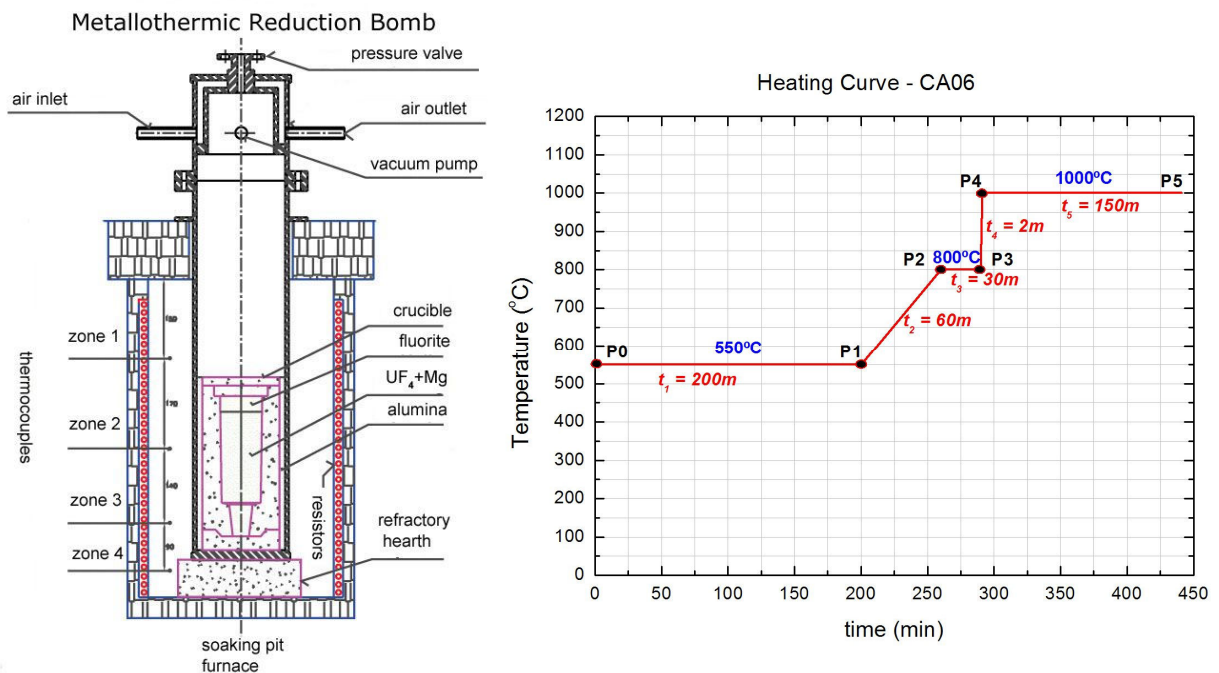


Figure 4. Magnisothermic bomb for metallic uranium reduction and reaction thermal cycle.

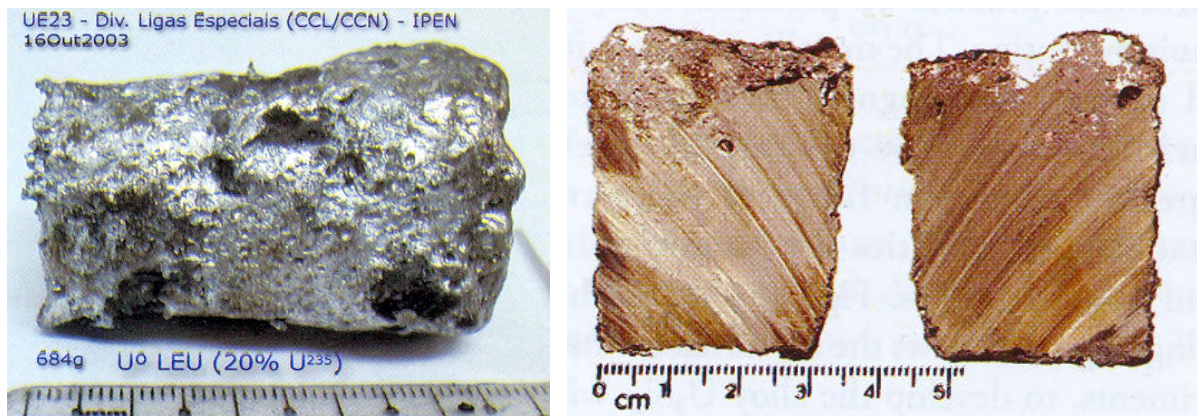


Figure 5. IPEN enriched metallic uranium pieces.

2.3. U_3Si_2 Fusion and Powder Fabrication

Since 2000, the Nuclear Fuel Center of IPEN has been dedicating great efforts to achieve expertise in production of intermetallic alloy U_3Si_2 . After facing some difficulties, as reported previously [7], in 2004 IPEN has arrived to the full experimental route to produce, in production scale, the necessary alloy for nuclear fuel.

From the produced uranium ingot, the metal was melted inside an induction furnace with silicon addition, with an adequate vacuum instrumentation and facilities for handling and melting uranium and uranium alloys. The zirconia crucible was specially designed to reach temperatures higher than $1750^\circ C$ and to support the aggressive environment created by uranium chemical attack. The load arrangement inside the crucible was studied to help the sequence of melting in the several stages of that molten alloy, before reaching the final intermetallic composition, as shown in Fig. 6. More than 20 trials were carried out, using natural uranium, before the first LEU U_3Si_2 were successfully made. It was produced 3 enriched U_3Si_2 melting in 2005, which consisted the first own produced load of fuel plate fabrication in IPEN. In general terms, the quality of this intermetallic has fully met the needs postulated by the requirements for a routine nuclear material. The X-ray diffractogram presented in Fig.7 attests the presence of the expected phases in the produced powder of this alloy.

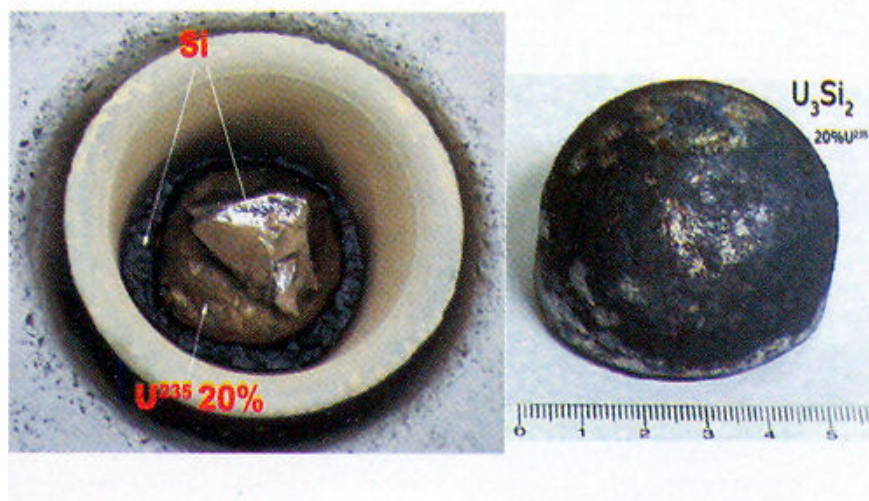


Figure 6. Assembling of U_3Si_2 crucible load before melting and the resulting ingot after induction melting.

2.4. Miniplate Fabrication

As previously mentioned, IPEN started manufacturing its own fuel element by using U_3O_8 -Al dispersion fuel plates with 1.9 gU/cm^3 . This uranium loading represents a U_3O_8 volume fraction of 27 %. If this same volume fraction is used, the direct substitution of the U_3O_8 by U_3Si_2 would result in an increasing of uranium loading to 3.0 gU/cm^3 .

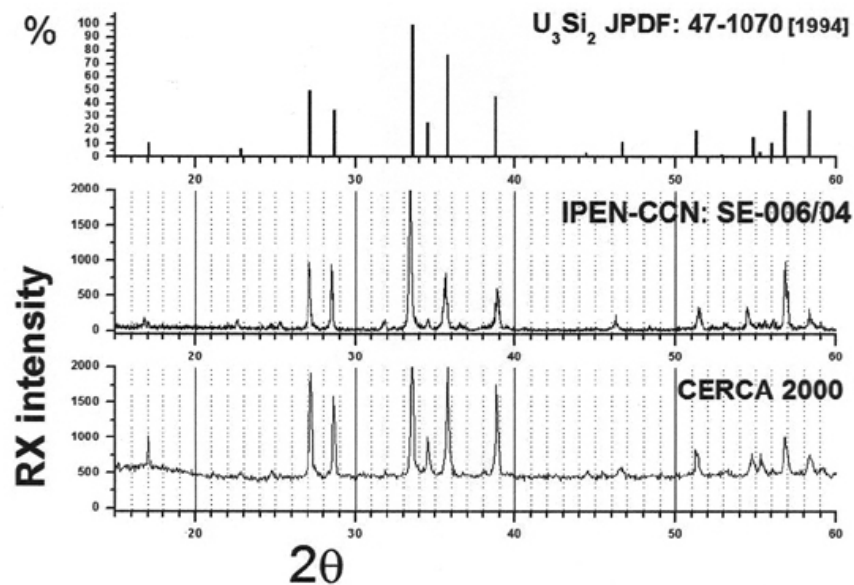


Figure 7. X-ray Diffractogram showing the U_3Si_2 produced by IPEN contrasted to the already used imported powder (CERCA).

The increasing of U_3Si_2 volume fraction in the dispersion has a technological limit of 45 %, in order to allow fabrication of fuel plates. This means maximum uranium loading of 4.8 gU/cm^3 . This is the objective to be reached. The increase in the volume fraction of both U_3O_8 and U_3Si_2 to 45 % implies in implementing modifications in the fabrication procedures in contrast to the currently adopted ones for LEU fuel. The necessary research work to adequate the fabrication procedures have been done under the IAEA and FAPESP research projects. The first results are presented elsewhere [2]. It was fabricated 22 miniplates with approximately 120 mm length and 42 mm width fuel meats. The compacts were 20 X 40 mm. Aluminum alloy 6061 was used for the frames and covers in fuel plate assemblies. The hot rolling temperature was 440°C . The fuel plates were fabricated by hot and cold rolling. The cladding thickness over the defect zones (dog-boning) was 0.28 mm (specification states a minimum of 0.25 mm). All the fabricated miniplates showed good metallurgical bonding. In the first fabrication tests it could be observed some oxidation of U_3Si_2 particles located near the interface core/cladding. The volume fraction of the oxidized phase was quantified by means of image analysis and the results indicated that a volume fraction between 2 and 4 % of the U_3Si_2 particles has been oxidized. The presence of the measured volume fraction of this oxide phase, within the fuel core, was considered to be no deleterious to the fuel performance during irradiation. However, the study the oxidation phenomenon was carried out to eliminate or minimize the oxide formation during the fabrication. The cause was determined to be the welding procedure. A modification in the welding device was done to minimize the air entrapment in the weld assembly. This was possible with the use of a glove-box with argon atmosphere. The oxidation problem was no more observed ever since. Fig. 8 shows a radiography taken from the miniplates fabricated with 4.8 gU/cm^3 U_3Si_2 -Al dispersion. Fig. 9 shows a radiography illustration with the final dimensions of the miniplate.

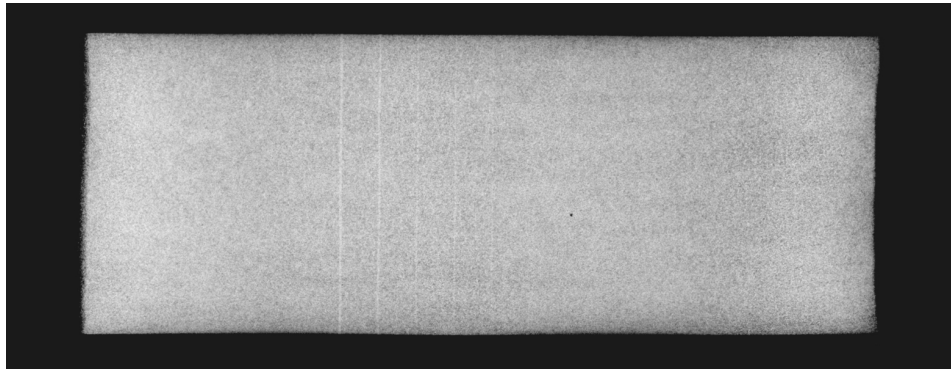


Figure 8. X-ray radiograph taken from U_3Si_2 -Al fuel meat (4.8 gU/cm^3).



Figure 9. X-ray radiograph taken from a finished miniplate.

2.4. Miniplate Irradiation at IEA-R1 Research Reactor

An irradiation device was placed at the IEA-R1 core support plate. Its overall dimensions were similar to the fuel assembly dimensions. It was projected to contain miniplates for irradiation and also to allow their removal for post-irradiation NDT to be performed at the spent fuel pool. The project assumed the possibility to irradiate up to 10 fuel miniplates at once. It was designed an internal support for the miniplates, which could be assembled inside the irradiation box. Fig. 10 illustrates the irradiation device components (miniplates; miniplate support, irradiation box).

The post irradiation examination includes miniplate thickness measurement, visual inspection and sipping. The fuel miniplate thickness measurement device is used inside the reactor pool, in the spent fuel storage area. It is operated from the reactor pool border and is able to measure the fuel miniplate thickness variation along its surface. This device is based on a mechanical structure for positioning the miniplate and to perform the scanning along the miniplate surface. The thickness measurement is performed by electronic probes (LVDT probes). The results are obtained by measurement instrumentation connected to the probes

and the data are stored and processed by a laptop computer. Fig. 11 shows the support structure and close-ups of the miniplate thickness measuring system.

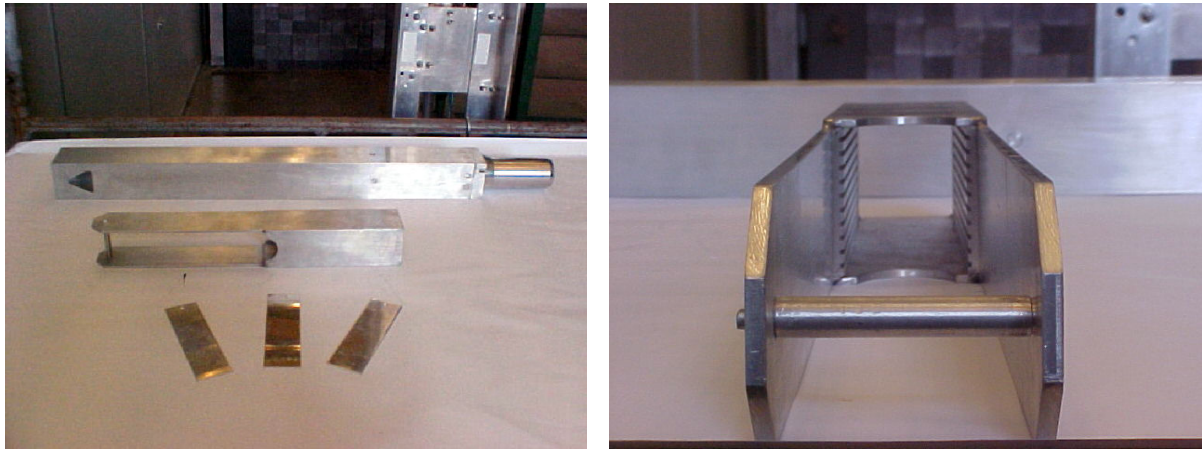


Figure 10. Irradiation device components: irradiation box, miniplates and miniplate support.



Figure 11. Thickness measurement device: support and miniplate measurement system.

3. CONCLUSIONS

The developments involving fuel fabrication was satisfactorily completed for U_3Si_2 -Al and U_3O_8 -Al dispersion fuels, which allows uranium densities of 4.8 and 3.2 gU/cm³, respectively. These are the maximum uranium loadings (45% in volume) possible by using these two uranium compounds. The next step is to develop and fabricate full sized UMo-Al dispersion fuel plates with 6.9 gU/cm³. This work was already started.

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