

MATERIALS SELECTION AND QUALIFICATION FOR THE STABLE SALT REACTOR – WASTEBURNER

A. D. Warren¹, T. Davis², J. Musgrove² & G. Anderson¹

1. Moltex Energy Canada Inc., Saint John, New Brunswick, Canada
xanderwarren@moltexenergy.com
2. Oxford-Sigma, Harwell Innovation Centre, Didcot, Oxfordshire, UK

Abstract

Selection and qualification of materials is a key step in moving a reactor from a concept to a detailed engineering design. A range of criteria can be used to guide this process including the obvious needs of mechanical performance, but also factors such as availability and prior irradiation experience.

With the unavailability of test reactors of relevant neutron fluence, the availability and extent of this irradiation data becomes key to supporting a safety case. Further there are differences between experimentally collected data, often of limited quantity but with tightly controlled parameters, and operational experience, where hundreds or thousands of specimens will have been irradiated under a range of conditions.

In this talk the optioneering process for a fuel clad for a Gen IV molten salt reactor is discussed with consideration of the knowledge gaps and routes toward qualification.

1. Introduction

Moltex Energy's Stable Salt Reactor – Wasteburner (SSR-W) is a fast spectrum reactor design, using a fuel containing mixed lanthanide/actinide chlorides and a NaCl/MgCl₂ coolant salt. The SSR-W was selected as one of two SMR candidates for further progression by NB Power out of a field of 90 candidates and is targeting a build date of the early 2030s. Stable Salt Reactor technology uses a novel technology, where molten fuel salt is contained in fuel pins submerged in a molten coolant salt. This differs from prior molten salt reactors such as the Oak Ridge Molten Salt Reactor Experiment (MSRE) where the fuel is circulated in the coolant salt loop, and offers inherent advantages with respect to the ease of refuelling and safety. As such, the material selection of the fuel clad becomes a key factor.

With the ambitious deployment schedule there is a need for an established material that is commercially available. This is further reinforced by the limited number of available test reactors, particularly those capable of achieving representative neutron fluxes of the correct energy spectrum and operational temperatures. Thus, a further criterion is for a material with meaningful quantities of neutron irradiation data, especially of broadly comparable fluence and fast neutron spectrum at SSR-W relevant temperatures. For this it is also necessary to differentiate between experimental irradiation data (which is to say a set of tightly controlled experiments, where many/all parameters are well understood but which only produces a relatively limited number of data points) and operational experience as a fuel clad (where hundreds or thousands of fuel pins are exposed under a broad range of conditions, albeit with less granularity on the data regarding precise conditions of each pin. Ideally this is surmised in

a set of design rules by the plant operator). From this it can be seen that both types of data have value, but that if operational experience is under sufficiently close conditions to the planned plant operation then it gives a strong degree of confidence regarding the suitability of a clad material.

The SSR-W clad temperatures are at the upper limit of those of liquid-metal cooled fast reactors such as Phenix (peak clad temperatures of $\sim 650^{\circ}\text{C}$ ^[1]) or PFR (peak clad temperatures of $\sim 700^{\circ}\text{C}$ ^[2,3]). Further, fuel pins in these reactors have moderate internal pressures and experience significant dose – making these strong possible candidates. Previous liquid metal fast reactor technologies have used a range of metals for fuel clads^[4], but these can broadly be divided into ferritic-martensitic steels (HT9), austenitic steels (AISI Type 316 based steels, 15/15/Ti & Sandvik 12R72/1.4970, D9) and Ni based alloys (Nimonic PE16). For all of these materials, it should be expected that both experimental data and operational experience will exist. If experimental fuel clads are considered this list gets broader (T91^[5], FV548^[6], Inconel 706^[7], etc), however the available data for these materials reduces significantly – and in many cases these materials were never adopted for operational use either as a result of offering inferior performance to other candidates or due to encountering previously unexpected issues^[7].

Table 1 Prior operational experience of liquid metal cooled fast reactor cladding materials^[4].

Reactor	Country	Fuel clad tube material
Rapsodie	France	316 SS
Phenix	France	316 SS
PFR	U.K.	M316 SS, PE 16
JOYO	Japan	316 SS
BN-600	Russia	15-15Mo-Ti-Si
Super Phenix-1	France	15-15Mo-Ti-Si
FFTF	U.S.A.	316 SS & HT9
MONJU	Japan	mod 316 SS
SNR-300	Germany	X10 Cr Ni Mo Ti B1515 (1.4970)
BN-800	Russia	15-15Mo-Ti-Si
CRBR	U.S.A.	316 SS
DFBR	Japan	Advanced austenitic SS (PNC1520)
EFR	Europe	PE16 or 15-15-Mo-Ti-Si
FBTR	India	316 SS

This paper describes the planned operational conditions, down-selects the possible materials to give a representative of each class, assesses the suitability of the selected material candidates and identifies the next steps.

1.1 Operational conditions

The SSR-W fuel pins are thin-walled tubes, with a vent to release fission gases into the coolant salt. The vented pin design and atmospheric pressure coolant will serve to give service dwell loads of the order of 5-15 MPa (principle stress sources are cross flow from pumped coolant and gas-manifold connection), with a design deformation limit of 0.1% strain. The reactor uses on-load refuelling, and under these conditions loads aren't expected to be significantly higher than under dwell conditions. Peak clad temperatures are predicted to be in the range of 650-700°C during use, followed by subsequent storage (at the core periphery) at a lower

temperature. A neutron dose of tens of dpa is anticipated, with a target of close to 100 dpa targeted. Corrosion from both the fuel and coolant salts is managed through redox control measures. The wider plant will be a stabilised 18Cr/10Ni steel.

The greater the capacity of the material with respect to mechanical properties, post-irradiation behaviours, swelling and creep, the greater freedom it gives other disciplines during concept design and also the greater margin of safety during operation. Whilst there is obviously a desire for the longest fuel pin life achievable to maximise the potential heavy element burn-up achieved, this maximum life will be dependent on core reactivity performance and materials performance limits: the interplay of a combination of corrosion mechanisms, irradiation degradation (swelling, embrittlement, irradiation creep) and thermal creep. There is a limit on the a minimum fuel pin life, under which refuelling frequency needs to be considered - this has arbitrarily been set at two years.

2. Optioneering

For the SSR-W fuel clad, the broad range of materials was down-selected to give a representative for each class of metals. This process was based on comparisons of material properties, irradiated material properties, relevant irradiation experience and availability as well as considering the evolutionary choices of previous operators.

2.1 Ferritic-martensitic steels

HT9 steel (also known as 12Cr-1MoVW) was developed by Sandvik and trialled as a fuel clad in the US Experimental breeder reactor-II (EBR-II) and used as both a duct and fuel cladding material in the Fast Flux Test Facility (FFTF) Reactor from 1982 to 1992^[5]. Studies in FFTF suggested it was unsuitable for use at high temperatures (550-600°C), with the bulk of data falling in the range of 350-550°C^[5,8]. Further it does not have any contemporary manufacturers, although it should be noted that TerraPower are attempting to resurrect production and demonstrate that ‘new’ HT9 is comparable to ‘old’ HT9^[9,10]. With operating temperatures too low for our purposes, coupled with the lack of availability, HT9 is not considered suitable for use. By comparison T91 is a broadly comparable ferritic-martensitic steel with an ASME accreditation up to 649°C for a Section III Division 5 material^[11], better high temperature creep properties (including data for 650-700°C^[12]) than those accepted for HT9 and a current supply chain^[5]. Whilst it lacks the extensive service irradiation data of HT9, it does have irradiation data (EBR-II, FFTF, Phenix and BOR-60^[5]) to relatively high doses as part of its commissioning process for the aborted Clinch River Breeder Reactor project. EM10 steel designed in France has similar composition and manufacturing route to T91 steel and has been used as duct material in Phénix (being cited as an example of material improvements increasing burn up) and Superphénix sodium cooled fast reactors^[13]. T91 irradiation experience is limited to up to 105 dpa and between 50-550°C^[5].

2.2 Austenitic steels

Historically AISI Type 316 was supplanted from the French fast reactor program by 316Ti and then by a 15/15/Ti based on Sandvik 12R72^[1,5,14]. In the UK a modified AISI Type 316 steel (m316), was partially supplanted as the prototype fast reactor (PFR) fuel cladding in the UK by Nimonic PE16^[5,15]. Based on this AISI Type 316 L/H has been discounted from further detailed consideration as the alternatives offer historic precedence for superior performance. It

is noted that the performance of D9 in FFTF is closer to that of 316Ti in Phenix, rather than French 15/15/Ti – despite being compositionally very similar to the later^[16]. Based on this evolutionary cycle and mechanical properties^[17-20] Sandvik 12R72/DIN 1.4970/“French 15/15/Ti” was selected as the Austenitic steel candidate. It should be noted that the interrelationship of these steels is not always clear: Sandvik 12R72 was developed for use in Phenix^[21,22], and is the original of the French 15/15/Ti steels. French 15/15/Ti is distinguished from 12R72/DIN 1.4970 by many authors^[16,23], and given the subtle differences in Mo, Si and B content this is justified. Sandvik 12R72 is compliant to the German DIN 1.4970 standard^[24], although some sources distinguish the German/Dutch/Belgian use of DIN 1.4970 from 12R72^[23,25] however it is unclear of the reasons behind this. In this study the family of steels are considered holistically, but it is explicitly acknowledged that additional work assessing the compatibility of this assessment is required. 15/15/Ti fuel pin clad temperatures for Phenix range from 400-650°C with the maximum neutron dose at temperatures of around 500-550°C^[1], and the wrapper temperatures for Super Phenix of 400-550°C^[1]. By early 1989, 21,630 pins clad in 15/15/Ti (a full core loading of 100 sub-assemblies) had been irradiated in Phenix^[7], with 10 sub-assemblies discharged at end of life with peak burn up and dose of 13.4%/128 dpa^[7]. An experimental capsule achieved burn-ups of 16.6% at the same dose before discharge^[7] - however five pin failures, at doses of between 0.6-9% burn up, have also occurred^[7]. DIN 1.4970 is described as being able to survive doses of 180 dpa in the temperature range 500-600°C, and 75 dpa at 700°C, as a fuel clad^[17]. In addition to use by French and German sodium cooled fast reactor projects^[1,17,26], 12R72 was trialled for a wrapper material for the UK PFR and selected as the fuel clad for EFR (core outlet 545°C and dose of 180 dpa)^[27]. Whilst Sandvik retain the capability to manufacture 12R72 steel, it is not currently in production.

2.3 Nickel based alloys

Whilst experimental studies have considered a range of Ni based alloys (In706^[7,28], In718^[6,27], etc) the only alloy to achieve significant usage as a fast reactor fuel clad (and wrapper^[29]) material is Nimonic PE16 in the UK Prototype Fast Reactor (PFR). PFR had peak fuel cladding temperatures of 700°C^[2,3]. It has been estimated that the maximum hoop stress due to fission gas formation was <70 MPa^[30], and due to oxide pellet-clad (swelling) interaction as typically 65-130 MPa (upper bound of ~200 MPa)^[7]. Broadly the PE16 fuel cladding in PFR experienced > 1.6 x10²⁷ n/m² (80 dpa) in the temperature range 400-725°C without a single failure in the 6590 pins irradiated^[3]. At peak clad temperatures of 700°C, the pins received a dose of ~35 dpa^[2], and by the end of PFRs operation in 1994 one fuel assembly had reached a 19% burnup (155 dpa)^[31]. Further:

- A broadly similar material with somewhat inferior irradiation behaviour, EP-753, has been used in Russian fast reactors as a fuel clad^[31].
- Nimonic PE16 has also been used as the tie bar material in the UK advance gas cooled reactor (AGR) thermal reactor fleet, where approximately 800 cm from the tie bar base sees temperatures of 650 °C and approximately equal thermal and fast neutron doses totalling approximately ~3x10²⁵ n/m² each^[32]. They aren't under significant dwell loads, but experience loads of 200 MPa during routine fuelling operation, occasionally rising to 265 MPa^[33] (~300 MPa^[32]) under fault conditions^[32]. The bars require very high reliability (1 failure per 10³ years) to ensure failure doesn't occur during refuelling^[33,34].

- Nimonic PE16 was a candidate fuel clad and wrapper for the European Fast Reactor (EFR; wrapper doses of >110 dpa) and fuel clad doses of 180 dpa, core outlet temperature of 545°C^[7,35].
- A candidate fuel clad for UK Commercial Fast Reactor/Commercial Demonstration Fast Reactor (CFR, CDFR); predicted clad temperature of 670°C^[36,37], target burn up of >10% heavy atoms^[37] and internal fission gas pressures of >70 MPa^[36].
- The planned fuel clad for the UK Enhanced Gas Cooled fast reactor (EGCR) for a planned dose of 167 dpa, peak clad temperature of 634°C (with a hotspot allowance of 94°C), external core coolant pressures of 4.3 MPa^[38,39].
- Further several academic optioneering studies have identified Nimonic PE16 as a possible clad material - Jones suggests it for a CO₂ cooled fast reactor^[2], with neutron doses of <100 dpa and peak temperatures of <700°C^[2], whilst Cole^[35] considers it a suitable material for future fast reactor fuel pins for temperatures of ≤650°C.

Nimonic PE16 is still produced by the Wiggin site of Special Metals; with the UKAEA PE16 heat treatment offering slight differences to those available off-the shelf. The UKAEA fuel tubes were fabricated by Fine Tubes^[40,41], which is still in existence as part of Ametek. (The British standard BS 2HR207 specification for Nimonic PE16 includes provision for seamless tubes of OD 6-219 mm and wall thickness of 0.5-20.0 mm^[42]) Further, the AGR tie bars previously mentioned are a tube-from of PE16 in contemporary use.

2.4 Comparison of options

The full comparison of these three metals is far beyond the scope of this paper, their compositions are given in Table 2 and a limited comparison of key parameters is given in Table 3. T91 has the lowest creep resistance of the candidate metals in the operational temperature range, has very limited irradiation data at temperatures >550°C (with a degree of uncertainty regarding the availability of the data noted by Davis^[5]) and no prior OpEx as a fuel clad. Whilst its possession of an ASME Section III Division 5 code qualification to 650°C is beneficial, as a ASME code committee member has previously stated that the code is not adequate or suitable for assessing fuel pin materials such that this is a non-sequitur advantage^[43]. Both 15/15/Ti and UKAEA STA PE16 appear to represent more suitable candidates for the SSR:W clad.

Table 2 Compositions of the selected materials.

Element	Nimonic PE16 ^[7]	T91 ^[5]	12R72 ^[24]
Cr	16.5	8.0-9.5	15
Ni	43.5	0.4	15
Mo	3.3	0.85-1.05	
Mn	0.1	0.4-0.6	1.8
Si	0.2	0.2-0.5	0.4
Ti	1.3		0.4
Al	1.3		
C	0.08	0.07-0.14	0.10
N	0.03		
Co	0.03		
B	20 ppm		0.006
P			0.030 max
S		0.01	0.015 max
V		0.18-0.25	
Nb		0.1-0.6	
Fe	Balance (~33.66)	Balance	Balance

UKAEA undertook several studies comparing the performance of Solution Treated & Aged (STA) Nimonic PE16 with 15/15/Ti (including both ‘French 15/15/Ti and 12R72/DIN 1.4970) as possible candidate clads for the EFR^[7] with the following key outcomes:

- Below 800°C PE16 has superior unirradiated proof stress and ultimate tensile strength, and shows greater uniform and total elongation^[7]
- PE16 offers superior irradiated proof stress and ultimate tensile strength for a comparable level of irradiation^[7].
- Both materials show very low ductility following irradiation(<0.5%), and the report suggests that there is no evidence that PE16 offers lower ductility under the same test conditions^[7].
- STA PE16 offers superior predicted secondary creep rates to 1.4970, and comparable rates to 15/15/Ti; with the recommended minimum creep ductility values being comparable at 0.2%^[7].
- STA PE16 and 15/15/TI share broadly comparable irradiation creep properties^[2].
- STA PE16 had achieved higher maximum burns than 15/15/Ti (although significantly more 15/15/Ti pins have been irradiated)^[2].
- No STA PE16 pins had failed as of 1989 with burn ups of 20.7% (~150 dpa), despite abrupt power changes and sub-assembly rotation. Five 15/15/Ti pins have failed at burn-ups between 0.6 and 9%, with a few reaching burnups of 16.6% (128 dpa)^[7].

Table 3 Comparison of UKAEA STA PE16, T91 and 15/15/Ti. Dpa for 15/15/Ti converted using the factor for AISI Type 316 steel in EBR-II, 4.38 dpa per 10²²n/cm²^[44] to give a broad approximation.

Material	UKAEA STA PE16 (Solution treated + Aged 1080°C/10-30 min +700°C/16hr)	T91	15/15/Ti / 12R72/1.4970
Example permissible stresses (assuming 0.66% 0.2% Proof Stress)	373 MPa at 600°C ^[45] 350 MPa at 700°C ^[45]	109 MPa at 650°C ^[5] ~75 MPa at 700°C ^[46]	86 MPa at 650°C ^[24]
Creep rupture lifetime	10 ⁶ hr at 600°C and 170 MPa ^[47] 1.7x10 ⁷ hr at 650°C and 10 MPa (DEMP-3 calc) ^[47] 3x10 ⁴ hr at 750°C and 10 MPa (DEMP-3 calc) ^[47]	10 ⁵ hr at 650°C and 40 MPa ^[48] 3x10 ³ hr at 700°C and 40 MPa ^[48,49]	2x10 ⁵ hr at 650°C and 127 MPa ^[24] 2x10 ⁵ hr at 700°C and ~85 MPa ^[24]
Irradiated yield stress	691MPa at 600°C/40 dpa ^[45] ~440 MPa at 650°C/54 dpa ^[31] 300-530 MPa at 750/<35 dpa ^[47]	400 MPa at 550°C/23 dpa ^[5]	50 MPa at 650°C/180 dpa ^[24] 250 MPa at 750°C/180 dpa ^[24]
Irradiation elongation	~1.3% at 600°C/40 dpa ^[45] ~1.5% at 650°C/54 dpa ^[31]	No data found at relevant conditions	~4% /500°C/ ~15 dpa (3.5X10 ²⁶ n/m ²) ^[50] ~5.6%/700°C/ ~0.25 dpa (2.1X10 ²⁵ n/m ²) ^[51]
In reactor creep	Predicted 0.2% creep strain = 20MPa/3x10 ⁴ hrs/650°C ^[2]	No data found at relevant conditions	5x10 ³ hrs at 615°C, 175 MPa and 2 dpa (6x10 ²⁵ n/m ²) ^[52] 5x10 ³ hrs at 720°C, 44 MPa and 2 dpa (5x10 ²⁵ n/m ²) ^[52]
Swelling	0.1%/650°C/113 dpa ^[30,53] 2%/650°C/200 dpa (model) ^[7] 0%/630°C/21 dpa ^[54] 0%/700°C/100 dpa (model) ^[55] 0%/700°C/200 dpa (model) ^[7]	2%/400°C/210 dpa ^[56]	~2%/650°C/85 dpa ^[57]

Further 15/15/Ti has key risks regarding the extent of higher temperature mechanical property and irradiation data, the compatibility of data for French 15/15/Ti vs 12R72/1.4970, the possibility of swelling issues at high doses and concerns regarding the ability to produce tubes of the correct size. UKAEA STA PE16 has an excellent track record, however risks still remain with respect to the availability of irradiation data, the compatibility of irradiation data from differing heat treatments and the low ductility following irradiation in the region 650-700°C. Due to the high strength of the material, little information is available regarding its low load creep properties.

Based on the optioneering done, Nimonic PE16, in the UKAEA STA condition has been selected as the SSR:W fuel clad material.

3. Progression of candidate

Whilst Nimonic PE16 has been extensively used in the UK fast reactor programme, it lacks significant international recognition and data availability is variable. Some work on it was done in the USA at the Hanford Engineering Development Lab under the auspices of the Advanced Alloy Development Program^[58], but the majority was undertaken by the UKAEA. A large percentage of this material has been declassified and now resides in the UK National archive at Kew Gardens: between fuel clad, fuel wrapper and AGR tie-bar applications there are ~100 technical reports covering subjects as diverse as fretting, fabrication and post-irradiation examination of Nimonic PE16, in addition to over 1000 PFR fuel clad panel notes (many of which contain additional technical reports or notes on PE16) and >50 meeting minutes. It is understood that EDF Energy Ltd. holds additional information on Nimonic PE16 AGR tie bar behaviour relevant to operation of the fleet. The vast majority of this corpus of literature contain derived information rather than raw data. Descriptive equations of the mechanical properties of STA Nimonic PE16 are given in two UKAEA documents; along with their basis, valid ranges and suitability for application to irradiated material:

- J. Standring & A. M. Wilson; Descriptive Equations for the mechanical properties of LMFBR candidate cladding and wrapper alloys; PFR Cladding Panel note 1080, 1988, CP/AGT-1/39, UK National Archive AB93/287^[47]
- J Standring; Mechanical Properties of STA PE16 Cladding for use up to 950°C, PFR fuel clad panel note 1095, FRDCC/FEWP/P(89)13,1989, UK National Archive AB893/287^[59]

And material property data and operational experience are summarised in several documents, including:

- K. Q. Bagley; PE16 Performance in the context of EFR clad requirements – A UK view, PFR Clad Panel note 1086 (revision 1), Jan 1989, UK National Archive AB93/287^[7]
- K. Q. Bagley, J. Standring, J. S. Watkin & R. G. Anderson; Recommended values for the properties of STE PE16 used in pin design evaluation, PFR Clad Panel Note 1065, 1988, UK National Archive AB93/286^[45]

In a 2016 meeting between the UK NNL and CEIDEN; NNL reported having a store of components and materials irradiated in PFR^[60], Table 4, should it be necessary to undertake additional testing of irradiated materials. Additionally, some of the PE16 clad pins from EBR-II irradiation studies are retrievable at INL. A key need is to confirm that any ‘new’ material

made to the UKAEA STA specification composition and heat treatment conform to the behaviours of these prior material. Given that the fabrication plant is still in use, difficulties are not foreseen. Further, the compatibility of data unirradiated of Nimonic PE16 subject to marginally different heat treatments needs to be determined – in several UKAEA documents post-irradiation data for material with different heat treatments is mixed, and the extent to which this can be applied to needs to be confirmed. Although there is a relatively large amount of irradiation data for PE16 from EBR-II, PFR and the AGR fleet, modelling is required to compare the neutron spectrum and fluxes of these reactors to the SSR-W and in so doing inform the extent to which both operational experience and irradiation data will translate.

As already noted, irradiated ductility is a key issue in the temperature range 650-700°C, Figure 1. Neither academic nor UKAEA literature reached a consensus if this effect is solely due to He embrittlement, grain boundary γ' -Ni₃(Ti,Al,Si) formation or a convolution of the two. Material irradiated in EBR-II revealed continuous grain boundary γ' ^[58]. PE16 irradiated in the EBR-II fast reactor at 636°C to doses of up to 74 dpa showed a helium generation rate of about 1.2 appm per dpa^[61], irradiation in the DFR had a helium generation rate of 1.1 appm per dpa at 5.0x10²⁶ n/m² (21 dpa) at 630°C^[54], predicted He evolution in PFR was at a rate of 1.15 appm per dpa^[2]. More generally PE16 helium production rate was estimated to be approximately 1 appm per dpa for materials exposed up to 100 dpa^[3]. The properties of irradiated PE16 and γ' -Ni₃(Ti,Al,Si) single crystals have been noted to be very similar, and it is suggested that the recovery in ductility observed in some tests at 735°C may be a result of a reduction in the degree of segregation/ γ' -Ni₃(Ti,Al,Si) formation^[62], which supports at least some contribution from phase effects.

Table 4 Reported stored irradiated components and materials from PFR, which are available to the NNL^[60].

	No of Sub assemblies	No of pins	Burn-up (%)		Pu enrichment		Cladding
			Min	Max	min	max	
Sub-assemblies	71	20,369	2.2	19.6	15.9	33.8	M316, PE16, HL548
Radial breeders	14	1,226	0.1	2.4	-	-	M314, PE16, FV448
Clusters	33	553	0.2	11.9	0.0	33.2	M314, PE16, FV548
Loose pins		133	0.3	23.2	12.5	33.6	M314, PE16, FV548

UKAEA had been investigating a different heat treatment (AERE D-treatment) for improved post-irradiation ductility^[7,40,41]. Limited irradiation data (<10 dpa) showed superior post-irradiation ductility compared to standard routes^[40], and later investigations up to 20 dpa confirmed this improvement^[41]. It was noted that the D-treatment production route could be impractical for commercial tube production (despite being used to produce several sub-assembly tubes^[40], and may lead to too great a loss of swelling resistance^[7], however this was overcome and a simplified method was under refinement^[41] with a revised specification and tube drawing route specified for the simplified method^[62]. Compared to ‘conventional’ UKAEA STA PE16 there is relatively little detail regarding the unirradiated and post-irradiated mechanical performance of this heat treatment, making it a less favourable candidate for implementation in the initial cores of a first-of-a-kind reactor. However, it constitutes a field for development later in the reactor life. From a practical perspective, the reduced ductility of STA PE16 following irradiation still significantly exceeds that required by fuel pin design tolerances. Irradiated material (~9-36 dpa) tested at cooler temperatures (232°C) broadly

analogous of refuelling conditions, shows recovery of tensile properties, however this is incomplete and ductility remains low^[63]. Whilst qualitative, reported PFR experience of reprocessing STA PE16 pins after 15.8% burn up (116 dpa), has shown that post-irradiation the material has sufficient residual ductility to allow withdrawal from the bundle, tolerate a degree of distortion without fracture and can be successfully cropped^[7].

Questions arise regarding the suitability of the material for higher temperatures, particularly the potential for cliff edge effects during transients. There is limited unirradiated tensile and no creep test data for UKAEA heat treatment material over 700°C, showing a fall in tensile properties in the range of 700-800°C for both UKAEA STA and several other heat treatment conditions. It has been noted that γ' precipitate volume fractions begin to fall above $\sim 750^\circ\text{C}$, with a solidus temperature of $\sim 875\text{-}900^\circ\text{C}$ ^[64], which is thought to be the cause for this decline in material properties. The extent of irradiated data for many properties is 735°C at limited doses in EBR-II^[30,63]: one study shows test data at 36 dpa^[30] and cites a source, however cross-referencing the source only shows data up to 22 dpa, Figure 1b^[63]. UKAEA documents refer to additional work done during the UK-2 project in EBR-II at 710-730°C up to ~ 60 dpa, however the data from this is not available and as such it is not clear if the 36 dpa data is genuine and erroneously referenced or entirely spurious. The extant data shows a small degree of recovery in ductility up to 22 dpa^[30,63] (and no significant reduction in the disputed 36 dpa data^[63]). UKAEA published some guidelines for extrapolation of properties to 950°C^[59] (however very little of this is benchmarked with experimental data beyond 750°C and as such is not considered suitably qualified by the authors). Taken holistically, there is little to support the routine operation of the clad at these temperatures, but the indications are that there are no cliff edge effects in this region provided operation is for limited duration.

Nimonic PE16 only has very limited molten salt experience (the materials data sheet lists corrosion data for non-redox controlled NaCl-NaSO₄ and NaCl-NaSO₄-V₂O₅ mixtures in the temperature range 700-900°C, where its mass loss behaviour is comparable to a 15.0% Cr, 10.0% Ni, 6.0% Mn, 1.0% Nb, 1.0% Mo, 0.5% Si austenitic steel^[65]). Moltex is undertaking bespoke molten salt compatibility studies for both fuel and coolant salts. In addition to conventional corrosion, galvanic corrosion effects between dissimilar metals have been observed in several types of molten salts^[66-72] – whilst these are mostly focused on fluoride salts, it is highly likely that similar effects have the potential to occur in chloride salts. Based on macro-composition and the observations noted in literature, the lower Cr (16.5 wt%) and higher Ni (43.5 wt%) than 18/10 austenitic steels should result in a galvanic couple, with the PE16 as the protected cathode and the 18/10 steel as the preferentially corroded anode^[67,70-72]. Holistically assessing the composition however makes things less certain. Russian work notes that combinations of Ti and Al can limit corrosion; it is unclear if this is simply with respect to Cr leaching, with Al and Ti lost in preference^[73]. DeVan's work^[74] shows that both Al and Ti can corrode preferentially to Cr, but further DeVan notes both Al and Ti are in effect protected (in Hastelloy N) at concentrations below 2 at%^[74]. The composition of PE16 is given as wt%, converting this gives 1.2wt% Al = 2.5 at% Al, 1.2wt% Ti = 1.4 at% Ti. Further the Ti is tied up in highly stable Ti(C,N) carbonitrides and (Ti,Mo,Nb)C carbides^[61,75,76], giving additional energy barriers to leaching. As such the Ti content should remain protected. The Al content of PE16 is in excess of that which DeVan reported to be protected from leaching^[74]. DeVan does not report how the Al is distributed in the alloy^[74], although conventional Hastelloy N (Al 0.1-0.15 wt%) does not show any Al based precipitates^[77] with the Al remaining in the matrix. In PE16 a portion of the Al will be tied up in γ' precipitates (Ni₃(Al,Ti)), which typically remain stable under both irradiation and thermal aging^[78]. That these precipitates coarsen and Ni₃(Ti,Al,Si) nucleates as functions of time and irradiation^[78] strongly suggests that the matrix

still contains some level of free Al after the initial heat treatment. The NASA study of material corrosion potential specifically considers Ni₃Al^[79]: noting that whilst the precipitates lead to reduced Al activity compared to Al in solution in the matrix, it is still sufficient to corrode^[79]. However, given PE16 only contains 0.5at% Al beyond DeVan's threshold, this reduced activity due to precipitation may be sufficient to substantially protect the Al content should corrosion occur.

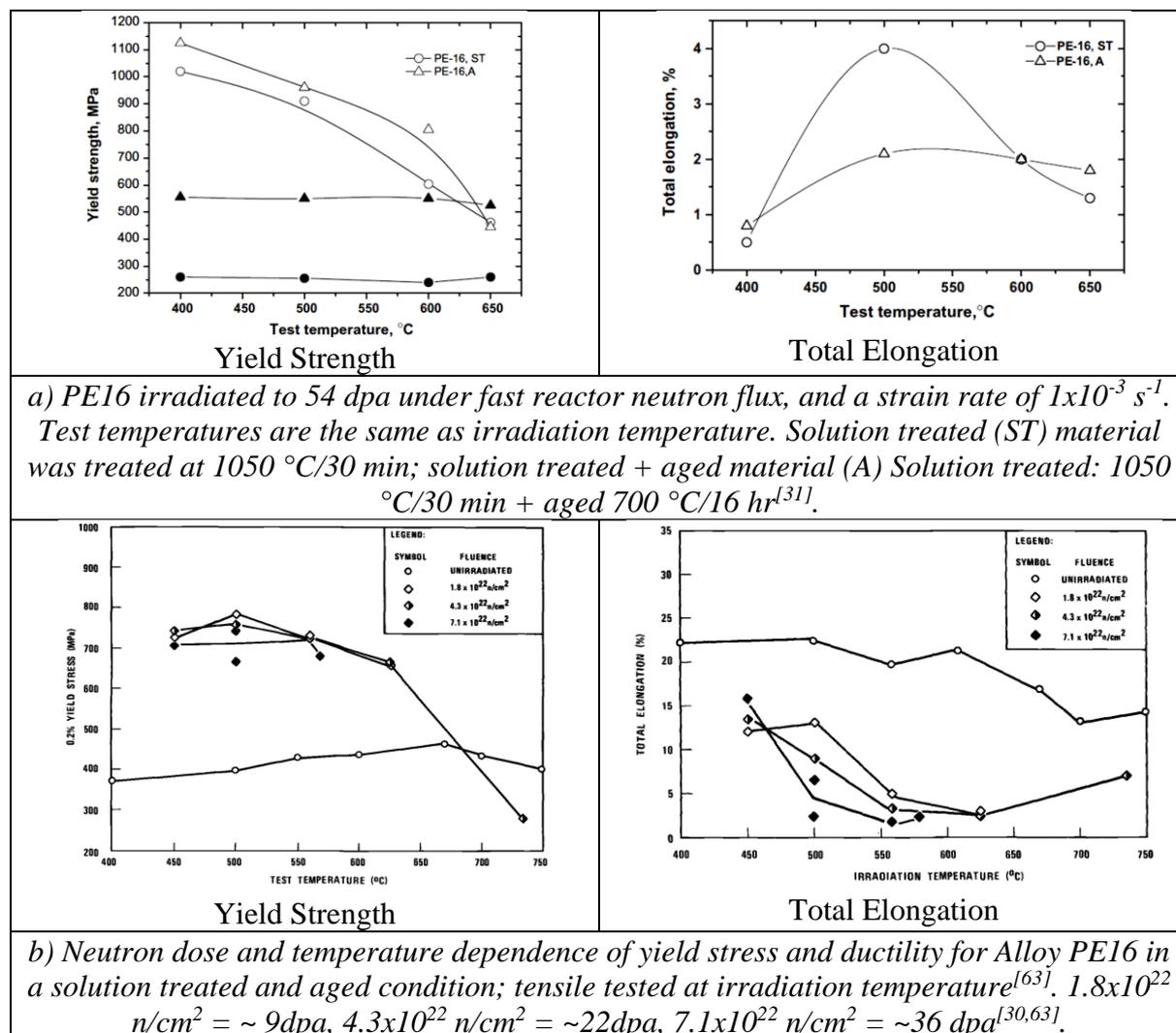


Figure 1 Tensile properties of irradiated PE16.

4. Conclusions

Following a review of previous fast reactor fuel clads, solution treated and aged Nimonic PE16 has been selected as the fuel clad for the SSR:W reactor. There is a large legacy of operational experience for the material in the temperature range of interest, to high neutron doses, from the British UKAEA archives – including design rules and material properties for as received and irradiated materials. Coupled with its high strength and creep resistance, it gives a significant confidence of success. Several qualification activities have been discussed to close identified knowledge gaps.

5. References

- [1] P. Dubuisson and D. Gilbon, "Behaviour and microstructure of stainless steels irradiated in the French fast breeder reactors", CEA-CONF-10539 Japan-France Materials Science Seminar, Paris, France, 22-25 Apr 1991, 12 pages.
- [2] R. B. Jones, "Fuel cladding for gas cooled fast reactors: a view of the sealed pin design in 1984", *Nuclear Fuel Performance*, BNES, London, 1985, 41-49.
- [3] T. M. Angeliu et al., "Assessing the effects of radiation damage on Ni-base alloys for the prometheus space reactor system", *J. Nuc. Mat.*, 2007, 366, 223-237.
- [4] K. Natesan and M. Lei, "Materials performance in sodium-cooled fast reactors: past, present, and future", International Conference on Fast Reactors and Related Fuel Cycles: Safe Technologies and Sustainable Scenarios, Paris, France, March 4-7, 2013, 32 pages.
- [5] T. P. Davis, "ONR-RRR-088: Review of the iron-based materials applicable for the fuel and core of future sodium fast reactors (SFR)", 2018. [Online]. Available: <http://onr.gov.uk/research/index.html>.
- [6] M. Kangilaski, "The effects of neutron radiation on structural materials", *REIC Report* No. 45, 1967, 253 pages.
- [7] K. Q. Bagley, "PE16 performance in the context of EFR clad requirements – A UK view", PFR Clad Panel note 1086 (revision 1), Jan 1989, UK National Archive AB93/287.
- [8] M. Toloczko et al., "Irradiation creep and swelling of the US fusion heats of HT9 and 9Cr-1Mo to 208 dpa at 400C", *J. Nuc. Mat.*, Vols. 212-215, pp. 604- 607, 1994.
- [9] C. Xu and M. Hackett, "Terrapower HT9 mechanical and thermal creep properties", In: I. Charit et al. (eds), *Mechanical and Creep Behavior of Advanced Materials. The Minerals, Metals & Materials Series*, Springer, 2018, 95-102.
- [10] K. Weaver, "Fuel development, testing, and schedule for the traveling wave reactor", DOE-NRC Second Workshop on Advanced Non-LWR Reactors, North Bethesda Marriott, June 7-8, 2016, 17 slides.
- [11] ASME, "Section III-rules for construction of nuclear facility components-division 5-high temperature reactors", in BPVC, ASME, 2019.
- [12] R. L. Nelson and A. Klueh, "Ferritic/martensitic steels for next-generation reactors", *J. Nuc. Mat.*, Vol. 371, No. 1-3, 2007, pp. 37-52.
- [13] J.-L. Seran et al., "Sodium-cooled fast reactor materials", in *Sodium-cooled Fast Reactors*, CEA, 2013.
- [14] P. Gavaille et al., "Mechanical properties of cladding and wrapper materials for the Astrid fast-reactor project", FR13 IAEA International Conference, March, 5th, 2013.

- [15] S. E. Jensen and P. L. Olgaard, "Description of the prototype fast reactor at Dounreay, Riso National Laboratory", DK-4000 Roskilde, Denmark, DK9700032, 1996, 45 pages.
- [16] P. Yvon, "Structural materials for generation IV nuclear reactors", 2016, Woodhead Publishing, 684 pages.
- [17] H.-J. Bergmann et al., "Entwicklung des werkstoffs X10CrNiMoTiB 15 15 als strukturmateriale für brennelemente", ISSN 0947-8620, Karlsruhe, 2003, 131 pages.
- [18] S. Latha et al. "Thermal creep properties of alloy D9 stainless steel and 316 stainless steel fuel clad tubes", *International Journal of Pressure Vessels and Piping*, Vol. 85, No. 12, 2008, pp. 866-870.
- [19] B. J. Makenas, "Swelling of 316 stainless steel and D9 cladding in FFTF", ASTM International, 1987.
- [20] A. L. Pitner et al., "Irradiation performance of fast flux test facility drivers using D9 alloy", Westinghouse Hanford Company, Richland, Washington, 1994.
- [21] J. Wallenius et al., "SEALER-3: A very small lead fast reactor for the Canadian market", The 19th Pacific Basin Nuclear Conference, (PBNC 2014) Hyatt Regency Hotel, Vancouver, British Columbia, Canada, August 24-28, 2014, 16 pages.
- [22] H. Guo, "Study on the compatibility of 15-15Ti with simulated fission products and lead coolant", Master Thesis, KTH Royal Institute of Technology-Tsinghua University, 2018, 73 pages.
- [23] N. Cautaerts et al., "Thermal creep properties of Ti-stabilized DIN 1.4970 (15-15Ti) austenitic stainless steel pressurized cladding tubes", *J. Nuc. Mat.*, 2017, 493, 154-167.
- [24] Sandvik 12R72 tube data sheet, S-1,780- ENG, March 1990, Sandvik, Sweden, 4 pages.
- [25] R. W. Swindeman and P. J. Maziasz, "High-strength austenitic stainless steel tubing", International conference on pressure vessel technology (ICPVT), Dusseldorf, Germany, 31 May - 5 Jun 1991, CONF-9105254-2.
- [26] K. D. Closs, XXVII. "Creep behaviour of canning material for fast sodium cooled reactors", *Gesellschaft fuer Kernforschung m.b.H., Karlsruhe (Germany)*., EURFNR-968, 1971, 22 pages.
- [27] A. Pay et al., "European fast reactor (EFR) fuel element design", *Fuel Elements and Assemblies*, C03 - Fast Reactor Fuel Elements SMiRT 10, Anaheim, CA, USA, August 22-27, 1989, 93-99.
- [28] M. M. Paxton et al., "Comparison of the in-reactor creep of selected ferritic, solid solution strengthened, and precipitation hardened commercial alloys at 595°C", Westinghouse Hanford Company, 1978, HEDL-SA-1404, 28 pages.
- [29] P. M. Boocock et al., "A review of CAGR fuel performance", *Gas Cooled Reactors Today*, Vol. 2, BNES, London, 1982, 79-84.

- [30] A. F. Rowcliffe et al., “Perspectives on radiation effects in nickel-base alloys for applications in advanced reactors”, *J. Nuc. Mat.*, 2009, 392, 341-352.
- [31] Yu. V. Konobeev et al., “Mechanical properties of high-nickel alloys EP-753 and PE-16 after neutron irradiation to 54 dpa at 400–650 °C”, *J. Nuc. Mat.*, 2011, 412, 30-34.
- [32] J. G. Gravenor et al., “CAGR tie bars – post irradiation tensile and stress rupture properties”. December 1981-March 1983, ND-R-1033(W), Dec 1984, UK National Archive AB7/26976.
- [33] P. A. Hitchcock and S. B. Fisher; “Mechanical testing of civil advanced gas-cooled reactor tie bars”, *Effects of Radiation on Materials: 15th international symposium*, ASTM STP1125, R. E. Stoller et al., (Eds), American Society for Testing and Materials, Philadelphia, 1992, 65-75.
- [34] I. K. Dickson et al., “The mechanical properties of CAGR tie-bars”, *Nuclear Fuel Performance*, BNES, London, 1985, 411-416.
- [35] G. C. Cole, “Performance analysis of a commercial fast reactor fuel pin design based on a Nimonic cladding material”, *Fast Reactor Core And Fuel Structural Behaviour*, BNES, London, 1990, 25-31.
- [36] J. Standring and A. M. Wilson, “Programme of creep and rupture studies on current and developmental fast reactor cladding materials”, UKAEA PFR Clad Panel Note 674, 1981, UK National Archive AB93/276.
- [37] R. D. Smith, “Review of the UK fast reactor programme”, International Working Group On Fast Reactors 14th Annual Meeting, Vienna, Austria, 31 March-3 April 1981, IAEA IWGFR/37/2.
- [38] W. F. G. van Rooijen, “Gas-cooled fast reactor: A historical overview and future outlook”, *Sci. Tech. Nucl. Inst.*, 2009, 965757, 12 pages.
- [39] J.T. Murgatroyd et al., “Optimisation of the CO₂ cooled fast reactor for plutonium and minor actinide management”, *European Nuclear Society - ENS-Foratom*, Brussels, Belgium, ENC 2002: European Nuclear Conference; Lille, France, 7-9 Oct 2002.
- [40] A. M. Wilson, “Optimization of PE16”, *UKAEA Clad Panel Mechanical Properties Study group: Notes of the 6th meeting held at NRL(R)*, 25 February 1988, PFR Clad Panel Note 1041, 1988, UK National Archive AB93/286.
- [41] A M Wilson, “Development of improved PE16 cladding”, *Fast reactor core and fuel progress report: January to June 1988*, PFR Clad panel note 1069, Jun 1988, UK National Archive 93/287.
- [42] “Steel grades HR207 chemical information, mechanical properties”, <https://www.steel-grades.com/Steel-Grades/specialsteel/74/5853/HR207.html>, accessed 2021-12-07.
- [43] T. L. Sham, “Advanced reactors at ASME”, NEI/EPRI Advanced Reactor Workshop, December 1, 2022.

- [44] M. Griffiths, “Effect of neutron irradiation on the mechanical properties, swelling and creep of austenitic stainless steels”, *Materials*, 2021, 14, 2622 (47 pages).
- [45] K. Q. Bagley et al., “Recommended values for the properties of STE PE16 used in pin design evaluation”, PFR Clad Panel Note 1065, 1988, UK National Archive AB93/286.
- [46] V. Sklenicka et al., “Creep behaviour and microstructure of a 9%CR steel”, Conference on Materials for Advanced Power Engineering, Dordrecht, Netherlands, 1994.
- [47] J. Standring and A. M. Wilson, “Descriptive equations for the mechanical properties of LMFBR candidate cladding and wrapper alloys”, PFR Cladding Panel note 1080, 1988, CP/AGT-1/39, UK National Archive AB93/287.
- [48] R. W. Swindeman et al., “Par 2: Stress factors for weldments”, *Verification of allowable stresses in ASME*, Section III, Subsection NH for Grade 91 Steel, 2007.
- [49] Z. Jiao et al., “Microstructure evolution of T91 irradiated in the BOR60 fast reactor”, *J. Nuc. Mat.*, Vol. 504, 2018, pp. 122-134.
- [50] K. R. Garr et al., “The effect of neutron irradiation on Types 316, 321 and Sandvik 12R72 stainless steels”, *Rockwell International Atomics International Division AT(04-3)-824*, 1974, 24 pages.
- [51] Ph. Van Asbroeck et al., “High temperature neutron-induced embrittlement of dispersion-strengthened ferritic alloys”, Symposium on the results of five years of BR2 reactor utilization, Mol, Belgium, 1973, 10 pages.
- [52] D. Closs, “Irradiation programme and results concerning in-pile creep and stress-rupture strength behaviour of canning materials (MOL-2 And MOL-5)”, in M. de Proost (ed.), *Results of five years of BR2 reactor utilization, Symposium dealing with research and development work on the liquid metal cooled fast breeder reactor, the gas-cooled reactor and the water cooled reactor*, Mol, Belgium, BLG-508, 1973 107-112.
- [53] F.A. Garner and D.S. Gelles, in: N.H. Packan et al. (Eds.), “Effects of radiation on materials”, ASTM STP 1046, *American Society for Testing and Materials*, Philadelphia, Vol. 2, 1990, p. 673.
- [54] A. Czyska-Filemonowicz et al., “Microstructure of helium-implanted Nimonic PE16”, *J. Nuc. Mat.*, 1987, 150, 24-30.
- [55] R. G. Anderson, “A brief review of irradiation creep design rules in use for PFR and CFR”, December 1978, PFR Clad Panel Note 507, PFR/FEDWP/P(79)647, 1978, National Archive AB93/272.
- [56] P. Jung et al., “Effect of implanted helium on tensile properties and hardness of 9% Cr martensitic stainless steels”, *J. Nuc. Mat.*, Vol. 318, 2003, pp. 241-248.
- [57] A. Maillard et al., “Swelling and irradiation creep of neutron irradiated 316Ti and 15-15Ti Steels”, Effect of radiation on materials 16th international symposium, ASTM STP 1175, A. S. Kumar et al., (Eds.), *American Society for Testing and Materials*, Philadelphia, 1993.

- [58] J. J. Laidler, Alloy Development Program. Quarterly technical progress letter, October, November, December 1976, HEDL-TC-160-11, Hanford Engineering Development Lab., Richland, WA (United States), 1977, 379 pages.
- [59] J. Standring; “Mechanical properties of STA PE16 cladding for use up to 950°C”, PFR fuel clad panel note 1095, FRDCC/FEWP/P(89)13,1989, UK National Archive AB93/287.
- [60] R. Stainsby, “R&D on fast reactors and their fuel cycles in NNL”, CIEDEN/NNL Meeting, 1st Feb 2016, 21 slides.
- [61] R. M. Boothby, “The microstructure of fast neutron irradiated Nimonic PE16”, *J. Nuc. Mat.*, 1996, 230, 148-157.
- [62] A. M. Wilson, “Proposals for improving the ductility of irradiated Nimonic PE16”, PFR Clad Panel Note 1038, 1989, FRDCC/FEWP/P(89)37, UK National Archive AB93/286.
- [63] R. Bajaj et al., “Tensile properties of neutron irradiated Nimonic PE16”, Effects of radiation on materials: Tenth Conference, ASTM STP 725, D. Kramer et al., (Eds.), American Society for Testing and Materials, 1981, pp. 326-351.
- [64] J. E. Palentine and J. Standring, “Correlative equations for the creep and rupture of unirradiated Nimonic PE16”, PFR fuel clad panel note 426, 1978, FRDC/FEWP/P(77)38, UK National Archive AB7/24044.
- [65] “Special metals NIMONIC® alloy PE16 data sheet”, Publication Number SMC-102, 2004.
- [66] Y. Wang et al., “Galvanic corrosion of pure metals in molten fluorides”, *J. Fluor. Chem.*, 2014, 165, 1-6.
- [67] F. Guo et al., “Corrosion in the molten fluoride and chloride salts and materials development for nuclear applications”, *Prog. Mat. Sci.*, 2018, 97, 448-487.
- [68] L. C. Olson et al., “Impact of corrosion test container material in molten fluorides”, *J. Sol. En. Eng.*, 2015, 137, 061007-1 to 061007-8.
- [69] J. W. Koger, “Alloy compatibility with LiF-BeF₂ salts containing ThF₄ and UF₄”, Oak Ridge National Lab report, ORNL-TM-4286, 1972, 46 pages.
- [70] H. Sun et al., “Interaction mechanisms of a Hastelloy N-316L stainless steel couple in molten LiF-NaF-KF salt”, *Corr. Sci.*, 2019 (preprint version).
- [71] R. S. Seller et al., “Corrosion of 316L stainless steel alloy and Hastelloy-N superalloy in molten eutectic LiF-NaF-KF salt and interaction with graphite”, *Nuc. Tech.*, 2014, 188, 192-199.
- [72] J. Qiu et al., “Galvanic corrosion of Type 316L stainless steel and graphite in molten fluoride salt”, *Corr. Sci.*, 2020, 170, 108677, 9 pages.

- [73] V. Ignatiev and A. Surenkov, “Alloys compatibility in molten salt fluorides: Kurchatov Institute related experience”, *J. Nuc. Mat.*, 2013, 441, 592–603.
- [74] J. H. DeVan, “Effect of alloying additions on corrosion behavior of nickel-molybdenum alloys in fused fluoride mixtures”, Masters Thesis, University of Tennessee, 1960.
- [75] A. Partridge et al., “The effects of long term ageing on Nimonic PE16”, *J. Nuc. Mat.*, 1992, 186, 100-116.
- [76] P. K. Rose and V.M. Callen, “The effect of thermal reactor irradiation on the microstructure of Nimonic PE16 tie bars”, *Nuclear Fuel Performance*, BNES, London, 1985, 401-409.
- [77] R. E. Gehlbach and H. E. McCoy, Jr., “Phase instability in Hastelloy N”, *Superalloys*, 1968, 346-366.
- [78] D. S. Gelles, “An example of precipitate stability in reactor-irradiated Nimonic PE16”, 1978, HEDL-SA-1395, ASTM 9th International Symposium on Effects of Radiation on Structural Materials, July 10-14, Richland, Washington.
- [79] A. K. Misra and J. D. Whittenberger, “Fluoride salts and container materials for thermal energy storage applications in the temperature range 973 to 1400 K”, NASA Technical Memorandum 89913 AIAA-87-9226, 1987, 22nd Intersociety Energy Conversion Engineering Conference, 10-14 August, Philadelphia, Pennsylvania.