ANNEX 6: NEA NUCLEAR INNOVATION 2050

R&D COOPERATIVE PROGRAMME PROPOSAL

Advanced structural materials for GenIV systems

Lorenzo Malerba

Executive Summary

The sustainability of nuclear energy will be ensured by the deployment of GenIV reactors, along with the facilities to close the fuel cycle: these (i) can produce more fuel than they consume, guaranteeing low-carbon energy production for millennia, (ii) offer much higher thermal efficiency than current reactors and (iii) provide higher standards of passive safety, resulting also economically attractive. However, materials will be exposed to high levels of temperature and irradiation, in contact with potentially aggressive non-aqueous coolants, and the design needs to be extended to 60 years of lifetime. **The development, screening and qualification of suitably performing and affordable structural materials are thus crucial to make GenIV reactors an industrial and commercial reality, with positive impact on safety, waste, economy and sustainability of nuclear energy**. Modelling is an essential enabler to accelerate materials deployment, as is more efficient international cooperation. The cross-cutting nature of materials issues through different technologies, on the other hand, gives investing on materials research high potential for economical return.

Only a few classes of materials promise to be able to sustain the harsh GenIV reactor operating conditions entail. To facilitate the licensing of reactor components, these **materials need to be fully qualified for the expected service conditions**. This requires, especially in view of the goal of 60 years lifetime design, the development of **innovative methodologies of lifetime assessment**, based on **robust extrapolation procedures**, that integrates accelerated testing with the support of validated models, to be **applied first to current industrial materials**, already selected for the GenIV prototypes. At the same time, **current materials cannot guarantee that the GenIV targets** of sustainability, waste minimisation, optimal use of resources and very high safety standards **will be all fully achieved**: the **severity of the conditions of operation expected in GenIV systems**, especially for cladding and other core materials, **strongly motivates the development of innovative materials solutions**.

Consistently, the present proposal flags two objectives:

1. Develop robust design rules for existing industrial materials, enabling reactor design for 60 years lifetime already in the case of prototypes
2. Develop industrially manufactured innovative materials with superior resistance to temperature, corrosion and irradiation, for use in future GenIV commercial reactors

The **gaps addressed** are:

- Timely complete the full codification of the materials now selected for GenIV prototypes, collecting sufficiently large and consistent data set, to derive design rules supported by models.
- Select and optimize the most suitable materials solutions to guarantee the highest performance level of truly GenIV commercial power reactors.
- Set solid bases for physical and engineering model development and application, in support of design rules and materials development.

Three case studies are accordingly proposed, namely:

1. Nuclear materials for a 60 years design lifetime: high temperature properties, compatibility with coolants, irradiation.
2. Development of promising high temperature, radiation and corrosion resistant core materials: F/M steels and SiC_f/SiC.
3. Nuclear materials modelling and experimental validation: example of low temperature embrittlement of F/M steels

To close the gaps **proper international cooperation is advocated**, with the support of NEA, **concerning data sharing, harmonized materials testing and characterization procedures, shared infrastructures and facilities, and agreement on model development**. In addition, **the involvement of TSOs and regulators** for an early transfer of information on materials and to receive guidelines, **is suggested as a way to accelerate the licensing of advanced nuclear systems**.

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List of abbreviations

ADS	Accelerator driven system
AFA	Alumina forming austenitic (steels)
APT	Atom probe tomography
ASTRID	French SFR prototype
ATF	Accident tolerant fuel
CSE	Creep-strength enhanced
CVI	Chemical vapour infiltration
EERA	European Energy Research Alliance
ESNII	European Sustainable Nuclear Industrial Initiative
FIB	Focused ion beam
FP7	7th Framework Programme (7 years research programme of the European Union)
F/M	Ferritic/martensitic
GFR	Gas-cooled fast reactor
H2020	Horizon 2020 (8th Framework Programme of the European Union)
HLM	Heavy liquid metals (liquid lead, liquid lead-bismuth eutectic, …)
HTGR	High Temperature Gas Reactor
HTR	High temperature reactor
IF	Internal friction
JPNM	Joint Programme on Nuclear Materials
LBE	Lead-bismuth eutectic
LME	Liquid metal embrittlement
LWR	Light Water Reactor
MAE	Magnetic after-effect
MYRRHA	Belgian ADS prototype
NPP	Nuclear Power Plants
ODS	Oxide dispersion strengthening/strengthened (steels)
PAS	Positron annihilation spectrscopy
PIE	Post-irradiation examination
SANS	Small angle neutron scattering
SFR	Sodium-cooled fast reactor
TEM	Transmission electron microscopy
TSO	Technical Safety Organization
TRISO	Tristructural-isotropic (fuel for (V)HTR)
(V)HTR	(Very) high temperature reactor
WPFC	Working party on fuel cycle (NEA body)
WPMM	Working party on multiscale modelling (NEA body)

1. Justification of the selection

The *sustainability* of nuclear energy will be ensured by the deployment of **GenIV fast reactors**, along with the facilities needed to close the fuel cycle. By **pushing the burnup to high values**, fast reactors can **produce more fuel than they consume** and the closed fuel cycle requires then only a short-term storage before reprocessing and re-use. Increasing the thermal *efficiency* (ratio between electricity and heat produced) is also key to improve the *sustainability*: not only more electricity is produced for a given thermal power, but also waste heat and environmental impact when cooling decrease. The thermal efficiency depends on the temperature of the reactor core and on the performance of the conversion system: **the higher the operating temperature, the higher the thermal efficiency**. High temperature operation also allows production of heat usable for several industrial applications. The **use of non-aqueous, non-moderating coolants will further enable fast reactor operation at high temperatures**, with efficiencies well-above those of LWR (~33%), close to coal or natural gas plants (~38-42%) or even combined-cycles (in excess of 50%). With liquid metals this can be achieved close to atmospheric pressure, with a design largely based on *passive safety systems*. This provides designers with **an important tool to make breeder reactors *safe and economically attractive*** and competitive with respect to LWRs and other energy resources. Finally, if high burnups are reached, fast reactors may transmute minor actinides into short lived fission products, so the radiotoxicity of the waste can be reduced to time scales below 1000 years, which more easily fulfil man made engineered repository licensing, their capacity being also increased by a factor of 10 or more.

To reach these goals, however, **materials in GenIV systems will be exposed to significantly higher levels of temperature and irradiation than in today's LWRs**, some components (e.g. fuel pins) experiencing also massive temperature gradients. Moreover, **the compatibility of materials with non-aqueous coolants needs in several cases to be demonstrated.** At the same time, to be economically competitive with other low-carbon energy systems, including current LWRs, the high capital cost of NPP construction needs to be compensated by **designing GenIV reactors for a *60 year lifetime***. This is a **challenge** because of the **harsh conditions** that both structural and fuel materials will be exposed to, that **call for new design guidelines**. The **<u>development, screening and qualification of suitably performing and affordable structural materials</u>** in terms of **resistance to high temperature and irradiation**, and **compatibility with non-aqueous coolants,** are thus **<u>crucial to make GenIV reactors an industrial and commercial reality</u>**, with ***impact on safety, waste, economy and sustainability of nuclear energy***. **Modelling is here an essential *enabler* to accelerate** materials deployment, **as is more efficient international cooperation**. Also, materials issues are often *cross-cutting* through different reactor systems and even non-nuclear energy technologies, thus **investing on materials research has a high potential for economical return**, even beyond nuclear applications.

2. The issue to tackle and objectives to reach

A lifetime of 60 years entails that the vessel and other non-replaceable components of the reactor core should preserve their ability to guarantee appropriate response to service and off-normal conditions after up to six decades of exposure to high temperature coolants (from 400-550°C in the case of liquid metal-cooled reactors, to 850°C in the case of gas-cooled systems), as well as to maybe mild, but continuous, neutron irradiation. Cladding materials, on the other hand, while regularly replaced during core reshuffling, are subject to the harshest conditions in terms of high temperature and temperature gradients, high irradiation dose and interaction with flowing coolants (as well as with the fuel inside): the longer they can sustain them, the higher the burnup that can be achieved. **Only a few classes of materials promise to be able to keep their properties for a sufficient amount of time against the unavoidable degradation of properties that these operating conditions lead to**. These classes are listed in the rows of **Table 1**.

Table 1 - Summary of materials of interest and relevant issues to be tackled.

<table>
<tr><th colspan="2">Type of related issues
Materials</th><th>Exposure, qualification & codification</th><th>Development of models</th><th>Innovative materials solutions</th></tr>
<tr><td rowspan="3">Austenitic steels</td><td>316L(N) (prototype irreplaceable components)</td><td colspan="2">Thermal ageing, thermal creep, compatibility with heavy liquid metals (HLM)/gas: increased database (including welds), accelerated testing, models describing micro/macro evolution → refinement of existing, or elaboration of new, design rules</td><td>Improve compatibility with coolants, apply high temperature protective barriers</td></tr>
<tr><td>15-15Ti (cladding)</td><td colspan="2">Irradiation creep and swelling, thermal creep, compatibility with coolants & fuel: increased database, models describing micro/macro evolution → refinement of existing, or elaboration of new, design rules</td><td>Increase swelling resistance and compatibility with coolants</td></tr>
<tr><td>Alumina forming austenitic (AFA) steels</td><td>Exposure needed for screening between candidates</td><td>Thermodynamic models for composition optimisation, microstructure evolution models</td><td>Addition of Al increases compatibility with coolants (protective alumina layer), but causes embrittlement at low T, although improves high T creep strength (NiAl precipitates): compromise searched</td></tr>
<tr><td rowspan="2">Ni-based alloys (heat exchangers, valves, coaxial pipes)</td><td>Alloy 800 (high Ni austenitic steel)</td><td>Design properties are available for Alloy 800H from existing Codes and Standards;
Need to validate the properties of thin-section under extreme conditions due to strength reduction.</td><td rowspan="2">To identify mitigation strategies, models of radiation-induced microstructure evolution in connection with predictions of embrittlement and swelling (He production). Creep and creep-fatigue engineering models in support of design correlations and rules.</td><td>Improve compatibility with alternative coolants and high temperatures; increase strength properties</td></tr>
<tr><td>Actual Ni-based alloys (eg Inconel 617, Haynes 230, …)</td><td>Exposure to high temperature in environment needed for screening between candidates and then for qualification.
Not suitable for irradiation field environments due to swelling and embrittlement.</td><td>Compatibility with coolant at high temperatures; manufacturing and joining</td></tr>
<tr><td rowspan="3">Ferritic / Martensitic (F/M) steels (cladding and core)</td><td>9-14 %Cr</td><td colspan="2">Low temperature irradiation embrittlement, irradiation creep, thermal ageing/creep, creep-fatigue (cyclic operation softening) compatibility with coolants, liquid metal embrittlement: increase database (including welds), models → define design rules and develop models in support</td><td>Need solution to minimize embrittlement, improve creep resistance (e.g. by thermomechanical treatment) and improve compatibility with coolants</td></tr>
<tr><td>Oxide dispersion strengthened (ODS)</td><td rowspan="2">Exposure needed for screening between candidates, suitable treatments for recrystallization to eliminate anisotropy after powder metallurgy production of bars and tubes by extrusion.</td><td>Oxide formation/stability, microstructure evolution, modes of deformation</td><td>ODS steels (tubes) have better creep resistance, but manufacturing and joining are issues (optimization needed); toughness and compatibility are also issues</td></tr>
<tr><td>FeCrAl alloys (also ODS)</td><td>Thermodynamic models for composition optimisation, microstructure evolution models</td><td>Addition of Al increases compatibility with coolants (protective alumina layer), but worsens mechanical behaviour: compromise searched</td></tr>
<tr><td>Refractory metallic alloys</td><td>Molybdenum alloys (including ODS)</td><td colspan="2">Exposure needed for screening between candidates. Irradiation creep and swelling, thermal creep, compatibility with coolants & fuel: increase database, models describing</td><td>Prospective materials, mainly for cladding, studied also in the past, with problems of</td></tr>
</table>

(cladding and core)	Vanadium alloys	micro/macro evolution → refinement of existing, or elaboration of new, design rules, supported by models		manufacturing, compatibility with coolant and mechanical behaviour
	High Entropy Alloys	Prospective metallic materials with potentially excellent mechanical properties, coolant & radiation resistance, need extensive investigation for screening, including understanding of origin of properties through modelling, before applications are identified		
Ceramics (cladding and coating)	SiC/SiC (also C/C) composites (cladding)	Mechanical test standardization, radiation resistance (thermal conductivity, hermeticity, swelling, …) and corrosion resistance → define design rules	Microstructure evolution models under irradiation, finite element models for composite architectures, X-ray tomography techniques	Liners to guarantee hermeticity of cladding, or other techniques to guarantee hermeticity. Limit thermal conductivity degradation under irradiation.
	Graphite	Irradiation effects on oxidation resistant graphite, irradiation creep, Codes & Standards development	Dependence of properties on porosity, graphite structure dynamics (stress states)	SiC/Graphite "composites"
	Non-metallic core support structures (*ad hoc* ceramics)	Screening of candidates. Test standardization (mechanical & thermophysical properties)	Microstructure evolution models under irradiation	Protection against oxidation
	Al_2O_3 coatings	Applied with different techniques on different substrates to protect against coolant attack and temperature: exposure for screening and qualification		
	Max phases	Prospective ceramic materials with excellent mechanical properties (for ceramics), coolant and radiation resistant, need extensive investigation for screening, including understanding of origin of properties, before applications are identified. Usable as coatings.		

Before suitably selected materials and components can be licensed for use in actual reactors, even within a conducive regulatory framework, **qualification is needed for the specific expected conditions**, via elaboration of design correlations and rules. These should guarantee the integrity of components fabricated for the 60 years' target (in the case of irreplaceable components), including sufficient resistance when exposed to off-normal conditions. While for some materials and conditions return of experience exists from their prolonged use in, e.g., sodium fast reactors built and operated in France in the past (or the AGR fleet in the UK), in most cases the lack of prior operational data needs compensation by performing appropriate **pre-normative research**. This implies developing an ***innovative methodology of lifetime assessment, based on robust extrapolation procedures, that integrates accelerated testing with the support of validated models***. The latter need to be **rooted in the deep understanding of the complex physical processes** that take place during prolonged and simultaneous exposure to high temperature, irradiation and aggressive coolants. This procedure should lead to an **<u>acceleration of the codification of materials and components in design standards</u>**.

In this framework, although the interaction with regulators is mainly the duty of the system designers, because of the need to refer to specific designs and service conditions, it is argued that the **involvement of TSOs and regulators to follow the procedures used for materials qualification and possibly guide them is likely to further accelerate the licensability of nuclear components**.

The qualification for GenIV applications concerns materials that have already been selected based on return of experience. However, the **severity of the conditions of operation expected in GenIV systems**, especially for cladding and other core materials, **strongly motivates the <u>development of innovative materials solutions</u>**. These should offer superior properties in terms of resistance to temperature and irradiation in contact with the selected coolants with respect to existing industrial materials. **<u>Only new materials will allow the the GenIV targets to be fully achieved</u>**, namely: optimal use of resources, waste minimization, highest efficiency, cost effectiveness and highest safety standards. The relevant materials and solutions are listed in **Table 1**, as well.

In short, **two key objectives supported by modelling can be flagged for this proposal**:

1. Develop robust design rules for existing industrial materials, enabling reactor design for 60 years lifetime already in the case of prototypes
2. Develop industrially manufactured innovative materials with superior resistance to temperature, corrosion and irradiation, for use in future GenIV commercial reactors

3. What is done/exists already, who is doing what, what are the means (resources and infrastructures)

Work performed and ongoing in Europe

Work on the subjects described in section 2 has been done or is ongoing within European projects, especially those linked to the EERA-JPNM in the 7th Framework Programme (FP7) and those recently started (2017) within Horizon 2020 (H2020). **Table 2** provides a summary.

Table 2 – Summary of past and ongoing European projects of relevance for the current proposal.

Project	Highlights
FP7/GETMAT (GEN IV & TRANSMUT-ATION MATERIALS – **2008-2013**) Total budget: 14 M€ EC grant: 7.5 M€	Objective: investigation, both in the theoretical and experimental domains, of selected material properties that are cross-cutting among the various Generation IV and Transmutation reactor designs. Target materials: 9Cr, 12Cr and 14Cr ODS (powder metallurgy) alloys + 9-12 Cr Ferritic/Martensitic (F/M) steels Results: An important post-irradiation experiment (PIE) program contributed considerably to better understand the behaviour of these classes of materials for nuclear applications. Other technical areas were welding and joining of ODS alloys and the development and first characterisation of corrosion protection barriers for structural materials to be used in HLM-cooled systems. The fabrication of ODS (thermal treatments, reproducibility of heats …) turned out to be a key issue that deserved more effort. The theoretical programme was devoted to the study of Fe and Fe-Cr alloys, with emphasis on atomic and microstructural level modelling studies and experiments to identify and validate physical models and mechanisms that explain irradiation hardening and embrittlement of these alloys, with seminal results for Fe-Cr steels. The results led to the identification of further R&D activities defined within the EERA Joint Program for Nuclear Materials (JPNM) and its related projects MATTER and MatISSE (see next).
FP7/MATTER (MATERIALS TESTING AND RULES – **2011-2014**) Total budget: 12.3 M€ EC grant: 6.0 M€	Objective: support the design of the ESNII reactors, in particular the ASTRID prototype SFR and the MYRRHA ADS, addressing material test procedures and design rules Target materials: Emphasis on the tempered F/M steel Grade 91; some activities on ODS fabrication. Results: Two main topics were addressed for tests procedures: miniature tests and tests in heavy-liquid metal. Miniature tests, such as small punch test and nano-indentation, can be used to screen on key design parameters, such as the ductile-to-brittle transition temperature, and to assess liquid metal embrittlement (LME) and irradiation effects on materials' properties. An important result is that contact with lead-bismuth eutectic (LBE) reduces the fracture toughness of P91. Corrosion in LBE was studied in both 15-15Ti and 316L austenitic steels and Grade 91 F/M steel, developing test procedures and addressing specific relevant issues. The Grade 91 steel softens under cyclic loading and the ultimate strength/yield ratio is much lower than for 316 stainless steels. To use Grade 91 for high temperature applications, design rules need to be revised or developed. Updated design rules for RCC-MRx were proposed for ratcheting, creep-fatigue and negligible creep, supported by an extensive test programme. Failure in metallic components has mainly been observed in welds. Hence proper manufacturing and design rules for welds are crucial. New fatigue joint coefficients were defined for both P91 and 316H steels. Code requirements on welding and different consumables and processes for welding were evaluated.
FP7/ARCHER (ADVANCED HIGH-TEMPERATURE REACTORS FOR	Objective: address generic and cross-cutting V/HTR-related R&D issues, supporting European contributions towards international demonstration of HTR and VHTR technology. It continued the work of the RAPHAEL project (completed in April 2010).

COGENERATION OF HEAT AND ELECTRICITY R&D **2011-2015**) Total budget: 9.72 M€ EC grant: 5.4 M€	Target materials: Mainly alloy 800H, including welded plates, and graphite; marginally addressed: Haynes 230, 20MnMoNi55, Alloy 617, Hastelloy X (Ni-based alloys), as well as Gr 91 (F/M) and 316SS. Results: R&D were organised in four Sub-Projects (SP): SP1: System Integration, SP2: Safety Aspects, SP3 Fuel and fuel back end, SP4 Materials and Components. SP4 also dealt with cross-cutting gas-cooled system related R&D, such as the coupling to industrial processes: designing and validating challenging high temperature, high performance heat exchangers and steam generators or solving the materials issues related to the development of these systems and components, such as corrosion by impurities, high temperature alloy application assessment and characterization, with specific focus on welding and joining, and high temperature instrumentation. The project results range from a better fundamental understanding of the behaviour of the system, such as the detailed safety studies that are performed on flammability levels, dust behaviour, fuel particle properties and graphite, to added operational experience on large components such as the intermediate heat exchanger.
FP7/MatISSE (MATERIALS' INNOVATION FOR SAFE AND SUSTAINABLE NUCLEAR – **2013-2017**) Total budget: 8.65 M€ EC grant: 4.75 M€	Objective: support the research done in the four subprogrammes on structural materials of the EERA-JPNM Target materials: F/M steels including ODS, austenitic steels (316 and 15-15Ti), SiC/SiC Results: R&D were organized in 4 work-packages. WP2: Modelling of irradiation embrittlement and creep in F/M alloys: models of microstructure evolution under irradiation developed in GETMAT for the Fe-Cr system were refined and extended to include the effect of other elements present in steels, such as C, Ni, Si, P, Mn, … Corresponding irradiation strengthening was modelled. Bespoke neutron and ion irradiation experiments were performed, followed by partial PIE. Effect of strain on atomic-level properties was addressed with a view to producing better irradiation creep models. WP3: SiCf/SiC composite tubes with metallic liner (sandwich tubes) as clad for GFR were fabricated and a broad evaluation of their behaviour was performed, including key properties of safety concern, investigated at relevant GFR operating temperatures: mechanical behaviour including gas tightness, thermal conductivity and corrosion/erosion resistance in impure flowing helium coolant with predefined levels of impurities. WP4: ODS: The stability of the ODS steels microstructure after heat treatments or ion irradiation, and the evolution of their mechanical properties, were studied by combining microstructural examination, hardness nanoindentation and small punch tests. Deformation mechanisms, especially interaction of dislocations with nano-clusters, were studied by in-situ neutron diffraction during thermo-mechanical tensile loading, in-situ TEM and under cyclic loading. The fabrication of ODS cladding tubes, both ferritic (14Cr) and F/M (9Cr), has been demonstrated. Innovative characterization techniques like mandrel tests, small punch, pin-load-tests have been used to study the behavior of the cladding tubes. Internal pressurized specimens were successfully fabricated from the ODS tubes, and a creep tests campaign was carried out. WP5: Creep-fatigue was studied, with emphasis on prediction cyclic softening for P91 and crack propagation for P91 an 316L. Functional coatings and modified surface layers were addressed to evaluate the compatibility of some specific and specially designed coatings primarily for claddings but potentially also for other components, e.g. steam generator or heat exchangers with Pb alloys as working fluids. Environment Assisted Degradation of materials in liquid lead alloys was addressed, in particular to investigate the mechanisms of crack initiation and propagation under constant and cyclic load conditions for ferritic-martensitic and austenitic steels.
EERA JPNM pilot projects (2016-onwards) **In-kind coordinated work of European research centres**	Content: R&D activities involving materials exposure and qualification, modelling and development of new materials. Examples: - A large test programme for welded 316 components has been launched to assess the extent of environmental effects on mechanical properties and corrosion, as well as to assess residual stresses after welding by neutron diffraction and modelling. - Multi-scale approaches are used to better understand and predict hardening and loss of ductility for austenitic and ferritic-martensitic steels at low levels of irradiation and also to address the issues of compatibility with HLM. As part of the modelling activities, ion irradiation as screening tool for novel materials is investigated to establish best practices in the design of the experiments, with the support of models. - Alternative routes to powder metallurgy are explored to produce ODS; small specimens are standardized to assess their fracture properties and the improvement of the creep resistance of F/M steels is pursued via composition-tuning and thermo-mechanical treatments. Concerning ceramics, test standardization and joining for SiC/SiC are addressed, while pulsed laser deposition alumina coatings are being tested on several substrates.
H2020/GEMMA (GENIV MATERIALS MATURITY– **2017-2021**) Total budget: 6.6 M€ EC grant: 4.0 M€	Objective: to qualify and codify the structural materials selected for the construction of GenIV reactor prototypes, as envisaged within the European Sustainable Nuclear Industrial Initiative (ESNII). Target materials: Those selected by the designers of the ESNII systems for fuel cladding and, in some cases, for the main vessel and the internals, namely austenitic steels. In particular AISI 316L(N) and DIN 1.4790 (15-15Ti), including their welds and coated versions. Workplan: The proposal is prepared under the auspices of the EERA JPNM, based on the contents of 7 of its Pilot Projects, dealing with environmental characterizations of materials and welds, numerical modeling accompanied by dedicated experiments and development of mitigation measures towards materials degradation. The qualification of the target materials implies assessing their resistance to harsh exposure conditions of high temperature, highly corrosive environment and intense flux of fast neutrons, including development of numerical models. The applicability of materials to the reactors' construction implies also that relevant welded joints will be tested, as well as corrosion protection treatments: surface treatments and new AFA steels and will be tested in representative conditions. The codification of the project results entails that a

	large amount of experimental data will be generated and that such data will be transformed to useful rules, for system and component designers, to be expressed in a suitable way for eventual inclusion in the Design Rules of the RCC-MRx code.
H2020/M4F (MULTISCALE MODELLING FOR FUSION AND FISSION MATERIALS- **2017-2021)** Total budget: 6.5 M€ EC grant: 4.0 M€	Objective: Bring together the fusion and fission materials communities working on the prediction of microstructural-induced irradiation damage and deformation mechanisms of irradiated ferritic/martensitic (F/M) steels. Target materials: F/M steels Workplan: This is a multidisciplinary modelling project where computer simulation and experiments at different scales are be integrated to foster the understanding of complex phenomena associated with the formation and evolution of irradiation induced defects and their role on the deformation behaviour in F/M steels for fusion and fission applications. Three main areas will be addressed: - Modelling of microstructural evolution under irradiation, with emphasis on the effect of C in Fe-Cr alloys and the specificities of ion irradiation as an affordable tool to study these problems; - Modelling of the onset and effects of radiation-induced plastic flow localization on the mechanical behaviour of F/M steels - Standardization of use of nanoindentation as a tool to evaluate mechanical properties in materials irradiated with ions and other charged particles, characterized by limited penetration.
H2020/GEMINI+ (Research and Development in support of the GEMINI Initiative **2017-2020)** Total budget: 4.37 M€ EC grant: 3.9 M€	Objective: Provide a conceptual design for a high temperature nuclear cogeneration system for supply of process steam to industry, a framework for the licensing of such system and a business plan for a full scale demonstration. Target materials: advanced HT materials Workplan: To create a licensing framework (WP1) for a configuration of HTGR cogeneration system (WP2) satisfying the needs of industrial process steam supply and the relevant safety requirements; to have an acceptable approach defined for safety demonstration and a feasible plan for demonstration of high temperature nuclear cogeneration (WP4)

Infrastructures and facilities used in European projects

- Materials test reactors: BR2 (Belgium), HFR (The Netherlands), OSIRIS (France - now shut down), LVR-15 (Czech Rep.) and relevant hot cells – in FP7/GETMAT an irradiation in BOR60 (Russia), the Lexur-II campaign, has been performed;
- Ion irradiation facilities: Jannus (France), HZDR ion beam centre ;
- Loops for exposure to (heavy) liquid metals and helium gas : CVR, ENEA, KIT, SCK•CEN, …;
- Mechanical tests in environment/at high temperature: several organisations, including universities;
- Microstructural characterization: APT, IF/MAE, PAS, SANS, TEM, other … at several organisations, including universities.

Work performed and ongoing in non-EU OECD countries

In the US much effort has been dedicated to the development of VHTR with gas outlet temperatures between 750°C and 950°C. Significant work has been performed within ASME in the last 10-15 years, to develop the ASME BPVC codes for the HTGR needs. Several technical issues have been identified that are now being considered for development and inclusion in the ASME code, such as: weldments, ageing and coolant compatibility issues; creep and fatigue; multi-axial loading; material allowables, in particular in view of 60-years' service-life; failure criteria. Activities are underway to extend the allowable lifetime from 300,000 to 500,000 hours for Type 304 and 316 stainless steels. In addition, roadmaps are being developed for the Nuclear Regulatory Commission to consider the new ASME Section III Division 5 design rules for high temperature reactors.

Several activities are running under the US Advanced Reactor Technology Program in support of VHTR and fast reactor technology. There is a graphite irradiation campaign using the Advanced Test Reactor at Idaho National Laboratory (INL). A Code Case for qualification of the nickel based Alloy 617 is currently under ballot with the ASME for nuclear construction up to 950°C and 100,000 hours. In addition, this program supports development of elastic-perfectly plastic design methods and the corresponding experimental validation. There are experimental programs to address potential issues with high temperature reactors beyond the design code, including characterization of the influence of notches on base and weld metal properties of Alloy 617.

The fast reactor structural materials program is addressing issues with advanced ferritic-martensitic materials and beginning characterization of the advanced austenitic material Alloy 709. The later activity has purchased a large heat of the alloy and is currently carrying out scoping tests to identify the optimum processing and annealing schedule to result in the most beneficial combination of creep rupture and creep-fatigue properties.

This section, partially filled in thanks to R. Wright, DoE, should be expanded based on input from non-EU representatives, still to be identified. It is important because knowledge on projects underway creates a strong basis to identify links.

Advisable to use a Table like Table 2

Remaining gaps addressed in this proposal

- Timely complete the **full codification of the materials now selected for GenIV prototypes**: face the challenge of 60 years lifetime design (high temperature operation under irradiation, contact with liquid metals/gas, including potential synergy with irradiation …) by putting together **sufficiently large and consistent data set, to derive design rules supported by models**.
- **Screen among the proposed advanced materials solutions**, especially for cladding, **to select and optimize the most suitable ones** to guarantee the highest performance level of truly GenIV commercial power reactors[1]: the most promising solutions need to be worked on to **move towards full optimization and codification**.
- Set solid bases for physical and engineering model development and application, in support of design rules and materials development, by working on the **establishment of modelling paradigms**, ensuring mutual **compatibility between codes** addressing different scales and developing **standard procedures and suitable programmes for their validation,** including assessment procedures that integrate information from lower length and time scales.

Among the **bottlenecks to close gaps** is the current **lack of proper international cooperation concerning data sharing, harmonized materials testing and characterization procedures, shared infrastructures and facilities, agreement on model development**: this is highlighted in section 4.

4. What can be done to improve/accelerate through cooperation

The qualification and development of materials for GenIV systems has positive impact on both safety and economics of nuclear energy and enables its sustainability. It is an essential activity, because **the design of licensable innovative reactors is impossible without qualified materials**. Luckily, this is largely a cross-cutting activity, so developments for one technology can also be useful for others.

[1] These will be designed and operated in the future based on the return of experience from the prototypes, but will need better materials to actually reach the objectives of GenIV systems in terms of safety, waste reduction, optimal use of resources and ultimately sustainability. Materials will be qualified in the prototypes themselves.

Breakthroughs are possible, though not easily foreseeable: **materials development & qualification is a continuous learning process that requires extensive availability of specific and specialised R&D infrastructures** (for exposure to irradiation, temperature, coolants, and relevant testing and microstructural examination). The **results need to be then efficiently transferred to both industry and TSOs/regulators**. To support design correlations and so define design rules for qualified materials and develop innovative materials solutions of improved performance, **modelling in the sense of combination of simulation and characterization for advanced understanding is a crucial enabler**.

Such a *knowledge-seeking activity* is **very suitable for international cooperation and can significantly benefit from it**. The overview given in section 3 shows that all over the nuclear world extensive work is ongoing, funded through national or supranational schemes, to address structural materials issues of relevance for GenIV reactors, as summarized in Table 1. **Provided that sufficient funds continue to be earmarked for these activities**, the **keywords** below are **essential in order to accelerate progress and innovation in this field, through cost-effective international cooperation**:

1. **Optimized/harmonized use of infrastructures**, making unique facilities transnationally accessible whenever needed and, even more importantly, adequately planning their use to avoid duplications and redundancies, while making the best use possible of the specificities of each particular infrastructure: this should be the rational approach to make materials qualification complete and affordable.
2. **Harmonization of procedures and methodologies** to test and characterize materials, especially innovative materials in specific environments, including protocols to perform microstructural examination with advanced techniques and to analyse the results. In several cases, completely new tests need to be designed and standardized.
3. **Data collection and sharing**, through suitable databases that should be eventually made available for reactor designers (industry and not only), TSOs and regulators: the more complete and extensive the databases, the safer the corresponding design rules and the more conducive the action of the regulators. Importantly, data collection and sharing makes sense provided that tests are homogeneous as to procedures used, so this point links strongly with point 2.
4. **Synergy on modelling**: modern modelling approaches inherently require interdisciplinarity, computer resources, theoretical developments and, crucially, extensive experimental effort for the characterization of materials at all scales after exposure to a variety of conditions: structured international coordination and exchange can only be beneficial. The three points above enter here, too. In addition, there is a need to make modelling approaches more compatible and complementary with each other including upscaling to engineering models, in order to better focus the development towards platforms of linkable codes and models, as well as to agree on protocols for experimental validation.

The present cooperative programme proposal hinges on a plan of actions that aims at increasing international cooperation on specific nuclear materials case studies, based on the above overarching principles.

The EERA JPNM is, in Europe, the main actor in this programme. EERA JPNM is connected to ESNII via its MoU with SNETP, and to Euratom via JRC. Another international actor that is expected to contribute to such programme, under the coordination of NEA/OECD and in connection with its relevant expert groups and working parties (e.g. the WPMM, the WPFC, etc), is GIF, specifically the working group on

VHTR materials, particularly in its cross-cutting configuration. As said, although the involvement of regulators is especially important in connection with the (pre)-licensing of the prototypes, and is therefore mainly the responsibility of designers, it is believed that an early transfer of up-to-date information on nuclear materials to TSOs and regulators, including procedures for data production and familiarization with models, can contribute to accelerate licensing of advanced nuclear systems.

NB: The existence of ear-marked funding of national and international origin is here a pre-requisite. In addition, the identification and deployment of effective international cooperation schemes to move towards optimized use of infrastructures, harmonization of test and characterization procedures, enhanced synergy on modelling, and more effective data sharing, are crucial parts of the plan and are the responsibility of international organisations and member states.

5. Plan of Actions and necessary means (resources and infrastructures)

5.1 Case study 1 – Nuclear materials for a 60 years design lifetime: high temperature properties, compatibility with coolants, irradiation

Technical issues and objectives

Exposure to high temperature leads to microstructural changes in materials, generically known as thermal ageing, that may lead to mechanical property degradation. Materials subjected to load at high temperature will also experience time-dependent deformation via creep processes and cyclic loading, which may lead to the formation and propagation of creep-fatigue cracks and component failure. Weldments are notoriously the weak link when subjected to these processes.

Exposure to even only mild (low flux) irradiation for long times, with subsequent production of matrix defects such as vacancies, self-interstitials and their clusters, enhances thermal ageing processes and also induces microstructural modifications that would not take place without irradiation. Already at low dose these may lead to loss of ductility and fracture toughness, due to the increase of yield and ultimate strength, especially at low temperature (<0.3 T_m, where T_m is the melting point). At higher temperature ($0.3T_m<T<0.6T_m$) and higher irradiation doses, swelling and irradiation creep may threaten the dimensional stability of components, a problem especially serious for cladding and fuel element materials. Finally, the possible production of helium by transmutation may pose potential threats to the mechanical properties of the materials even at high temperature.

In addition, flowing liquid metals and gases may be corrosive, inducing or accelerating material failure both under static loading (brittle fracture) or under a time-dependent loading (fatigue and creep). Lead and lead-bismuth eutectic (LBE), jointly called heavy liquid metals (HLM), present particular challenges for component design and safety demonstration. Guaranteeing resistance to corrosion, dissolution and erosion is the main factor limiting the operating temperature and the number one safety issue for HLM-cooled systems. Thus, particularly when in contact with HLM, protections in the form of ceramic coatings (Al_2O_3) may be necessary and need to be tested and qualified, as well.

The **objective** of this R&D case study proposal is then to **develop an accelerated methodology for the design and life assessment of structural components for GenIV reactors that should be in service at high temperatures and subjected to aggressive environments for 60 years or more, as a prerequisite for the successful deployment of commercially competitive GenIV systems**. This

involves the prediction of the long-term processes of material degradation and how they affect the integrity and performance of nuclear components fabricated **according to industrial practices**. The methodology is intended to be implemented into future industrial design and assessment codes and standards.
Effective international cooperation as described in section 4 is here a crucial enabler.

List of actions

The qualification of the materials selected for **prototype GenIV reactors, essentially commercial austenitic steels**, in order to derive suitable engineering design rules and to identify the appropriate operating condition windows, requires an intensive research programme.

The programme, in its first stage, would focus on high-temperature degradation, i.e. thermal ageing, creep, fatigue and their interactions, in baseline and welded material. Studies on effects of contact with coolants (corrosion, dissolution, erosion, …) would be going on as well, based on the available resources and in complementarity with ongoing projects. For example the European H2020 project GEMMA (see Table 2) comprises extensive activities on compatibility of austenitic steels with HLM and He, including welds and also coated and alumina forming austenitic steels. These activities could be further supported by the present project, via international cooperation. The logical continuation of this effort would eventually be the assessment of the combined effect of irradiation and exposure to specific coolants, as well as long-term low-dose irradiation at high temperature, compatibly with the availability of resources and relevant facilities (mainly MTR offering specific capabilities of dose reached and conditions simulated). This is detailed in the following points:

1) Assess historical data: the main bottleneck for the development of long-term design and assessment codes and procedures is the lack of data on material properties and degradation mechanisms representative for the long-term operational conditions. Compilation, review and (re)assessment of historical data is thus essential.

2) Test on service exposed materials: long-term creep, accelerated creep and creep-fatigue tests on representative materials including welds should be planned and started as soon as possible. Tests with no stress that represent "pure" thermal ageing should also be included as reference.

3) Characterise microstructure in thermally aged and creep- or creep-fatigue deformed materials.

4) Map corrosion rate and perform static and dynamic mechanical tests in stagnant and flowing coolants under a range of service and off-normal conditions, controlling temperature, velocity of the fluid and impurities, especially oxygen content.

5) Plan and then perform irradiation experiments to generate data representative of the combination of high temperature, coolant attack and neutrons, for baseline and weld materials.

6) Develop models, from mechanistic-based, to engineering-based correlations for long-term service conditions, accounting for key degradation mechanisms, to extrapolate accelerated test results.

7) Health monitoring of materials and components' structural integrity with respect to the above properties and damage state, by means of non-destructive techniques.

Timeline and resources

Due to the need for long-term tests, a programme of this type is by default long-term. However, the goal here is to accelerate progress applying the methodology detailed above. The programme could start as a five-year project which would allow compilation and analysis of existing data and models and planning and start-up long-term tests and accelerated tests, to be then extended for another five years, with follow-ups for the most long term tests. An illustrative timeline is given in **Table 3**.

No completely new test facilities are needed for this phase, but the large test programme advises coordination of tasks based on available facilities. In a second stage, when irradiation campaigns are designed, it is not sure that those that are currently available will be (i) still available and (ii) sufficient for the task, especially in terms of accessible doses. A wide range of stake-holders, from reactor designers to industry and regulators, as well as basic and applied researchers, should be involved.

The cost of this action can be estimated between 5 and 10 M€/yr, although the launch of irradiation campaigns may increase this estimate.

Table 3 – Timeline of case study 1

1. General High Temperature	1	2	3	4	5	6	7	8	9	10
1.1 Review & assessment of existing HT design rules, assessment procedures and models										
1.2 Analysis of operational conditions for components and associated load conditions										
1.3 Compilation and review of historical test data (all HR degradation mechanisms)										
1.4 Planning & implementation of test programme for in-service exposed materials										
1.5 Synthesis Code Evolution High Temperature Long-term operation										
2. Creep										
2.1 Review and assessment of methodologies for accelerated tests										
2.2 Planning and implementation of overall test programme										
2.2.1 Long & medium-term test: preparation, implementation, post-test analysis										
2.2.2 Accelerated tests: preparation, implementation & post-test analysis										
2.3 Development and implementation of creep models at different length scales										
2.4 Validation of models										
2.5 Recommendations for code evolution										
3. Creep-fatigue										
3.1 Planning and implementation of test programme										
3.2 Development creep-fatigue models at different scales (initiation & crack propagation)										
3.3 Validation of models										
3.4 Implementation of health monitoring strategies for component structural integrity and materials										
3.4 Recommendations for code evolution										
4. Thermal Ageing										
4.1 Accelerated tests for thermal ageing planning, implementation and post-analysis										
4.2 Model development for microstructural evolution										
4.3 Model for impact on mechanical properties										
4.4 Model validation by mechanical tests of service-exposed and artificially aged material										
4.5 Recommendations for code evolution										
5. Environmental effects: corrosion, dissolution, erosion, liquid-metal embrittlement										

5.1 Planning and execution of test programme (stagnant and loops)										
5.2 Microstructural characterization										
5.3 Development of models, improvement, validation										
5.4 Recommendations for code evolution										
6. Irradiation effects										
6.1 Planning of irradiation campaigns in suitable facilities										
6.2 Execution of irradiation										
6.3 First post-irradiation examination										
6.4 First code recommendations										
7 Welded components (also integrated with 2-6)										
7.1 Review of code rules for welds										
7.2 Characterization of welds (residual stresses, micro-structure and defects)										
7.3 Damage and defect assessment welds										
7.4 Code evolution welded components										

5.2 Case study 2 – Development of high temperature, radiation and corrosion resistant core materials: F/M steels and SiC_f/SiC

Technical issues and objectives

The use of austenitic steels for cladding is penalized by their susceptibility to swelling: it is unlikely that these materials can withstand doses in excess of 100 dpa, because above this dose the volume increase and especially the embrittlement that ensues become inacceptable. Pushing the burnup to the highest level possible requires more swelling resistant materials to be used. However, in order to increase energy efficiency, these materials should also withstand the highest temperature possible. Finally, since the thickness of the cladding is necessarily limited (thin-walled components), they should also prove to resist the attack of liquid metals and gases used as coolants. **Here the attention is focused on the two most promising classes of materials with these features**, which are **of interest also for ATF and fusion applications**, namely **(i) creep-resistance enhanced and/or alumina-forming F/M steels, including oxide dispersion strengthening (ODS), and (ii) SiC_f/SiC composites**.

F/M steels are inherently much less prone to swelling than austenitic steels, allowing doses up to 200 dpa to be safely attained, without experiencing any major volume change. They are, however, penalized by a relatively low operating temperature upper limit of 550°C, and therefore require creep-resistance enhancement. This can be achieved in two ways: (a) By optimizing the thermomechanical treatments of composition-tuned, nuclear grade F/M steels (where "nuclear grade" means that for example elements like Co that induce very high activation or others that are known to promote radiation embrittlement should be minimized), for the rest produced via conventional metallurgy: these are called creep-strength enhanced (CSE) F/M steels and are targeted to withstand service temperatures up to 650°C; (b) By oxide dispersion strengthening (ODS), by means of powder metallurgy techniques,which should allow operating temperatures of 700°C or in excess of it. The production of cladding tubes is in the first case not an issue; in the second one the feasibility is demonstrated, but the technology remains far from producing a fully satisfactory final product. A problem that penalizes ODS steels is the scarcity of industrial providers engaged in wide production of either tubes or other relatively large components. However, ODS F/M steels are worth being pursued because they bear the promise of being not only high temperature resistant, but also highly resistant to irradiation, including to the effects of helium produced by transmutation, even more than conventional F/M steels, thanks to the sink effect of the interfaces between oxides and matrix. The research plan should, in any case, include activities aimed at reducing the fabrication costs of these steels, possibly finding alternative fabrication routes.

Another problem is that F/M steels, including ODS, are prone to corrosion and erosion, especially in contact with HLM, and are also affected by a loss of mechanical performance due to liquid metal embrittlement (LME). Protection against the former can be provided in the form of alumina surface layers. These can be either deposited, e.g. by pulsed laser deposition, or their formation may be induced by implanting aluminum on the surface, so as to change only the local composition (surface treatment). In both cases, the stability of the protection needs to be proven, but the advantage is that they apply equally to CSE and ODS F/M steels, as well as to any steel, without changing their bulk properties. Alternatively, Al can be added to the whole steel (FeCrAl steels), providing full self-healing capacity. The problem with the latter solution is that it changes the properties of the whole material, which therefore needs to be optimized to provide sufficiently good mechanical performance, including high temperature and irradiation effects, and then specifically qualified. In order to guarantee good radiation and temperature resistance, the ODS FeCrAl solution seems especially attractive. It should be noted that ODS FeCrAl is a candidate material for ATF cladding in Japan. Unfortunately, however, there is no certainty that any of these corrosion/erosion protection strategies provide also immunity against LME in HLM: this will need to be tested and it is considered that mitigation strategies against this problem will require specific investigations to understand the mechanisms whereby LME operates, that to date remain unclear.

To operate above 800°C no steel is suitable. Of all refractory materials, the main candidate for core components and cladding is currently the SiC_f/SiC pseudo-ductile ceramic composite. SiC material is already used as ceramic layer retaining fission products in Tristructural-isotropic (TRISO) fuel for V/HTR concepts. The fabrication of SiC_f/SiC thin-walled tubes has been demonstrated by using textile methods, where fine filaments (~ 10 µm) are combined into fibre tows and woven or braided into a tubular preform and bound into place by additional bulk SiC, deposited by carbon vapour infiltration (CVI): this is an expensive and time-consuming, but well established fabrication method. Ways of reducing time and cost of production exist, for example microwave CVI, but these methods need to further developed to provide nuclear grade materials properties. One of the main problems of this material is the scarce hermeticity of SiC/SiC tubes to fission product percolation, which is exacerbated by irradiation and service cracking. To remove this problem the "sandwich" concept has been recently developed (CEA patent), where a suitable metallic liner is incorporated between two structural SiC_f/SiC shells, allowing the matrix to remain fully sealed even if the ceramic matrix cracks through. This solutions needs further testing and qualification. Another issue that needs further study is the decrease of thermal conductivity of SiC_f/SiC during irradiation at low temperatures (≤500°C), due to introduction of lattice defects. Nevertheless the radiation stability of this material is overall satisfactory, exhibiting temperature dependent swelling that remains below 1-2% in the whole 300-1600°C range. Finally, in contact with gas or liquid metal coolants under very low oxygen conditions and temperature above 1300°C, a volatile compound (SiO) is produced and the reaction proceeds until consumption of the SiC.

The **objective** of this R&D case study proposal is then to **take the TRL of promising GenIV core reactor materials such as F/M steels and SiC/SiC from <5 to >5, as a condition for the deployment of nuclear systems that fully achieve the expected target of sustainability, safety and economy**. This involves pursuing targeted parallel developments on these two classes of materials. For steels, the different research lines should eventually converge to a single high performance material, resistant to both high temperature and corrosion. In the case of SiC/SiC the goal is to identify an affordable and qualified route to produce clads. Accelerated screening procedures based e.g. on ion irradiation and modelling are considered. Coordination at international level and cooperation as described in section 4 are expected to be key to accelerate progress along this route.

List of actions

1) CSE F/M steels: The actions are very much in line with standard metallurgical research: optimization of composition and thermo-mechanical treatments, guided by thermodynamic models to reduce to the minimum the trial and error part of the developmental process, followed by screening between several heats and finally full qualification for the selected material.

2) ODS F/M steels: optimization of the tube production procedure by removing the anisotropy through suitable intermediate thermal treatments leading to controlled recrystallization; ensure the reproducibility of the final product, also through a detailed knowledge of the deformation behaviour; possibly reduce costs via alternative fabrication routes that also offer easier industrial production upscaling; develop appropriate methods for welding or joining without losing the painstakingly achieved good features of the microstructure; screening between different possibilities and eventually full qualification of the selected material.

3) Alumina-protected F/M steels, including ODS: the protection effectiveness and stability of the surface layers needs to be verified under different conditions of temperature, fluid velocity and impurity level, with emphasis on the worst conditions, including the effect of service loading (also cyclic) and allowing for the existence of cracks or for the accidental failure of the protection, that should not lead to uncontrolled or uncontrollable component failure, as well as for the effect of irradiation.

4) Alumina-forming (FeCrAl) F/M steels, including ODS: optimization of the fabrication process through screening of the most suitable compositional choice (reduce Cr and Al as much as possible, while guaranteeing stable Al_2O_3 protection by adding opportune reactive elements like Hf, Zr, …), via characterization of the properties of these materials: stability of the oxide surface layer (see above), mechanical properties of FeCrAl in environment, etc. CSE or ODS should be routes to be followed to make these steels also resistant to high temperature operation.

5) Liquid metal embrittlement: specific tests (e.g. slow strain rate tests in environment) are needed to verify the immunity or susceptibility of the above materials to LME; fundamental investigations involving microstructural investigations and modelling are also needed to unravel the mechanisms of this process, in support of the identification of mitigation strategies.

6) Small/miniaturized specimen testing, microstructural characterization and ion irradiation: all the above implies the ability to screen among potentially large quantities of different heats, most likely not corresponding to large amounts of material: to accelerate the screening the use of small or miniaturized specimen testing will help, both to spare material and to allow the use of ion irradiation. The latter may be an affordable screening tool for radiation effects if miniaturized specimen methods to extract mechanical property information are applied, such as small punch test or nanoindentation,

coupled with advanced microstructural local characterization. Also micromechanical tests on FIBbed micropillars can be of relevance. These methods require however still effort for full standardization.

7) SiC/SiC composites: response to irradiation of non-stoichiometric fibers, separately and in composites, especially in connection with mechanical properties and loss of thermal conductivity; compatibility of composite with liquid metals and helium gas in presence of impurities in the coolant, (potential need for protective coatings); evaluation of mechanical properties of tubes after standardization of relevant mechanical tests, to complete a consistent full characterization for the specific conditions of use; verification of stability and hermeticity under irradiation of, in particular, sandwich tube solution; solutions to limit thermal conductivity degradation; suitable joining techniques; identification of alternative and cheaper production routes.

Timeline and resources

A possible timeline is provided in **Table 4**. As can be seen, there is a common group of activities related to the development and optimization of innovative materials that include screening techniques.

For the different family of materials considered here (advanced F/M and SiC/SiC), the timeline is more or less shifted, but common activities are identified: high temperature deformation mechanisms studies; thermal stability; radiation resistance (experiments & modelling); corrosion and LME assessment; joining and welding techniques.

The cost assessment for innovative materials development is around 10-15 M€/yr, taking into account irradiation and PIE campaigns.

Table 4 – Timeline of case study 2

Activity	1	2	3	4	5	6	7	8	9	10
1. Screening techniques: develop and optimization of innovative materials										
1.1 Advanced microstructural techniques										
1.2 Development of small specimen tests techniques										
1.3 Standardization activities										
1.4 Data Management										
2. ODS cladding tubes										
2.1 Optimization of the fabrication route: Intermediate heat treatments										
2.2 Reduce fabrication cost										
2.3 High temperature deformation mechanisms studies										
2.4 Thermal stability										
2.5 Corrosion and Liquid metal embrittlement assessment										
2.6 Irradiation resistance (experiments & modelling)										
2.7 Joining and welding techniques										
3. Advanced F/M steels (CSE, FeCrAl)										
3.1 Optimization of the chemical composition										
3.2 Optimization of thermo-mechanical treatments										
3.3 Up-scale to industrial production (ingots)										
3.4 Fabrication of thin-walled tubes feasibility										
3.5 Fabrication of thick components feasibility										
3.6 High temperature deformation mechanisms studies										

3.7 Thermal stability										
3.8 Irradiation resistance (experiments & modelling)										
3.9 Corrosion and Liquid metal embrittlement assessment										
3.10 Joining and welding techniques										
4. Alumina surface layers										
4.1 Optimization of deposition/induction procedure to achieve maximum stability										
4.2 Qualification under worst case scenarios										
5. SiC/SiC composites										
5.1 Optimization of the fabrication route (hermeticity)										
5.2 Reduce fabrication costs										
5.3 Joining and welding techniques										
5.4 Mechanical test standardization										
5.6 High temperature corrosion/oxidation mechanisms										
5.8 Irradiation effects - experiments & modelling										

5.3 Case study 3 – Nuclear materials modelling and experimental validation: example of low temperature embrittlement of F/M steels

Technical issues and objectives

An integrated modelling approach is crucial to properly interpret, categorize and extrapolate the results of materials exposure and testing, in view of producing robust design correlations and codes, as well as of developing optimized materials. This approach, that combines advanced characterization with computer simulation, lies at the core of the shift from the *observe and qualify* to the *design and control* paradigm. This shift will boost and accelerate progress towards innovation in nuclear energy.

The development of models that describe the microstructural and microchemical evolution of differently exposed materials, based on the fundamental physical mechanisms responsible for it, is one of the main goals of the modelling effort, because macroscopic properties are reflections of microscopic features. In F/M steels, macroscopic effects of major concern for nuclear applications are for example radiation-induced hardening and embrittlement (loss of ductility and plastic flow localisation). These result from the hindered motion of dislocations due to the creation of radiation defects: key is to understand which defects are mainly responsible for this and how they form.

Atomic-level and atomistically-informed mesoscale models that satisfactorily describe microstructural and microchemical processes in model materials for F/M steels (binary or ternary alloys) do exist. They need, however, further refinement to include the chemical complexity of real materials. Moreover, the length and especially time scale limitations of current modelling tools need to be overcome.

The microstructural and microchemical description is then expected to inform mesoscale and then macroscale mechanical behaviour models. The former, for the purpose of modelling hardening and embrittlement, are mainly of dislocation dynamics type. The latter are continuum plasticity models for the description of the deformation behaviour under load, from the level of grain aggregates to, through homogenization procedures, the scale of the component. In this respect, for the prediction of the tensile behaviour under irradiation, while progress has been made, existing models are still limited. Even stronger limitations exist for the description of crack initiation/propagation processes and therefore to predict properties such as fracture toughness.

The use of charged particles (ions, electrons, …) is very suitable for modelling purposes, because it allows the effect of parameters such as dose and temperature, and to a certain extent also dose-rate, to be affordably studied over wide ranges, that would be prohibitive to explore using neutron irradiation. Modern microstructural characterization tools allow very precise characterization as a function of, for example, particle penetration depth, including the assessment, to a certain extent, of local mechanical property changes. However, the design of appropriate charged particle irradiation experiments requires being well aware of the associated artefacts, e.g. limited ion penetration, damage gradients, wanted and unwanted injected atomic species, spectral differences, etc. Even though charged particle irradiation experiments have been performed for decades, there is a need to develop microstructural and microchemical evolution models that take into account the specificities of charged particle irradiation. This is necessary to optimise the design of such experiments, beyond the standard use of binary collision approximation models for particle penetration and dpa profile prediction, and to define best practices for the performance of this type of experiments.

Eventually, however, neutron irradiation experiments are unavoidable. These are multi-annual types of research activities, because the preparation and performance of the irradiation take time, and even more time (several years) takes the PIE which, for modelling purposes, requires thorough characterisation, based on the combination of several examination techniques, all of them to be applied to active materials. Costs are also high, though they can be reduced making use of already designed larger experiments (“piggy-back” irradiation in MTR).

Irrespective of the type of irradiation, shared protocols need to be established for the analysis of the results of microstructural characterization techniques (TEM, PAS, APT, SANS, …), because different procedures may make results from different laboratories incomparable with each other, which hampers the validation of models. Likewise, protocols for a fair comparison between simulation and experiment results need to be established. These will benefit from the development of methods that simulate how a given experimental technique would see a given microstructural feature.

In order to concretely address all the above methodological problems, it is necessary to choose one prototypical case on which to focus international cooperation, to apply the *observe and control* paradigm in all its aspects. The case chosen should provide a good framework to define best practices and standards concerning characterization techniques and use of charged particle irradiation as screening method. For this purpose it should comply with a series of conditions, namely it should correspond to an important operation-limiting process, it should require understanding over different scales, and it should have already reached a sufficient level of development to make it possible that in a decade the whole set of modelling tools can be fully developed and deployed, envisaging possibly also industrial use.

The **objective** of this R&D case study proposal is to **use the problem of embrittlement and loss of ductility of F/M steels irradiated at low temperature (~300°C) as a paradigmatic case to develop in its entirety an example of integrated modelling, as support to robust design correlations and codes, as well as to the development of optimized materials**. This involves pursuing targeted parallel model developments that should eventually converge to a single integrated approach, in a properly coordinated framework of international cooperation.

In Europe, the Euratom-funded project M4F provides partial support to activities related to this issue, thereby providing a basis for concrete collaboration with other OECD countries, that should only be extended to allow and enhance international cooperation.

List of actions

1) Development of radiation-induced microstructure and microchemical evolution models: These models should be the upgrade of existing ones and the main goal is to increase the chemical complexity that they can treat, i.e. to include the effect that, separately or in synergy, each alloying element has on the defect production and evolution (typically in F/M steels C, Cr, Si, Ni, Mn, P, …). Another issue concerns the computational efficiency of complex models. The use of artificial intelligence methods is expected to be of help to address the chemical complexity and bridge through scales, as well as eventually to by-pass them, reducing also the problem of long computing times, thereby boosting progress.

2) Development of plasticity models at single grain and aggregate scale: Here too an important issue concerns the transfer of the complexity of the microstructure that develops under irradiation, in terms of defects and solute rearrangement, to dislocation dynamics models where suitable local rules need to be defined to translate the effect of the underlying microstructure on their motion. Subsequently, dislocation dynamics is expected to inform crystal (gradient) plasticity models to treat the mechanical behaviour of aggregates. Of special importance in this context is the challenging problem of slip localization.

3) Development of continuum mechanics models up to component scale: Here actions should be devoted to identify suitable homogenization methods. These should be able to take the effect of the largely localized processes that occur under load in irradiated F/M steels, *in primis* slip localization, to the scale of relevance for the calculation of the stress and strain state of components. Quasi-continuum models may in some cases be of use.

4) Performance of modelling oriented experiments: These experiments should be guided by the needs of models in terms of identification of mechanisms and need to have data for calibration/validation. Several experiments on F/M steels and model alloys have been already executed for this purposethat need to be exploited, but new ones should be designed to provide currently unavailable data. Ion irradiation can be a suitable tool to explore wide ranges of conditions, but neutron irradiation reference experiments should always be included as well. A wide range of characterization techniques should be combined, in a consistent way (TEM, APT, SANS, PAS, …). For dislocation dynamics model validation on single crystals tests on FIBbed micropillars are an essential tool. The models should, however, always pursue the target of predicting quantities of engineering interest, thus essentially predict the full tensile curve and provide indications about fracture toughness.

5) Establishment of protocols: This problem, that concerns ion irradiation, analysis of microstructural characterization techniques data, extraction of mechanical properties from miniaturized tests, and model calibration/validation, needs to be addressed essentially by performing benchmarks and round-robins and by agreeing on best practices within suitably created committees of experts. The development of tools to simulate the response of the technique to a given microstructure are expected to be of crucial support in the process of model calibration/validation.

Timeline and resources

Table 5 illustrates the list of actions and their tentative timeline over a perspective of ten years since the start. The estimated resources needed to adequately funding these activities can be estimated to be around 5 M€/y, with additional costs associated with neutron irradiation experiments, if any.

Table 5 – Timeline of case study 3

Activity	1	2	3	4	5	6	7	8	9	10
1. Models for microstructural evolution under irradiation										
1.1 Supportive ab initio calculations	■	■	■	■	■	■	■	■		
1.2 Cohesive models for progressively more complex alloys	■	■	■	■	■	■	■	■		
1.3 First approximation microstructural evolution models (limited chemical complexity)	■	■	■	■	■					
1.4 Model validation and identification of gaps (using established protocols)				■	■	■	■			
1.5 Advanced microstructural evolution models (richer chemical complexity)						■	■	■	■	■
2. Plasticity models at single grain and aggregate scale										
2.1 Atomistic study of interaction of dislocations with specific microstructures (input to higher scales)	■	■	■	■	■	■	■	■		
2.2 Dislocation dynamics models using combined microstructural information (experiments/models)	■	■	■	■	■	■	■	■		
2.3 Derivation of crystal plasticity equations and models from dislocation dynamics			■	■	■	■	■			
2.4 Slip localization continuum models				■	■	■	■	■		
2.5 Performance of micromechanical experiments on selected materials (as received and irradiated)				■	■	■	■	■		
2.6 Validation and refinement of models						■	■	■	■	■
3. Plasticity models at polycrystal scale and transfer to component scale										
3.1 Polycrystalline homogenization			■	■	■	■	■			
3.2 Development of physically based constitutive equations			■	■	■	■	■			
3.3 Simulation of the damage initiation process during tensile loading						■	■	■	■	
3.4 Experimental validation							■	■	■	■
4 Modelling oriented experiments										
4.1 Identification of model materials and conditions for experiments: available and to be produced	■	■								
4.2 PIE of relevant materials irradiated with charged particles and neutron in the past	■	■	■	■	■					
4.3 Coordinated performance of new irradiation/ageing experiments for further validation				■	■	■	■			
4.4 PIE of newly irradiated materials							■	■	■	■
5. Protocols for charged particle irradiation experiments										
5.1 Identification of materials conditions and existing experimental results to be used as reference	■	■								
5.2 Adaptation and use of microstructural evolution models to simulate charged particle irradiation			■	■	■	■				
5.3 Definition of new experiments to test models and provide new insight				■	■					
5.4 Performance of experiments and relevant PIE/modelling						■	■	■	■	
5.5 Elaboration of protocols for best practice charged particle irradiation experiments									■	■
6. Protocols for performance and analysis of microstructural characterization and model validation										
6.1 Inter-laboratory comparisons on reference specimens	■	■	■	■						
6.2 Elaboration of best practices for examination performance and analysis			■	■	■	■				
6.3 Dissemination of best practices							■	■	■	■
6.4 Development of models for the simulation of the response of the technique to given microstructures	■	■	■	■	■	■	■	■		
6.5 Definition of protocols for the comparison between experiments and simulations			■	■	■	■				

6.6 Development of tools in support of the validation of models by comparison to experiments										
6.7 Dissemination of tools										
7. Protocols for performance and analysis of micromechanical tests										
7.1 Inter-laboratory comparisons on reference specimens										
7.2 Elaboration of best practices for examination performance and analysis										
7.3 Dissemination of best practices										

5.4 Enablers in terms of international cooperation schemes

Expert committees should be enabled to work on the issues listed here below, which are key for the success of the case studies of the previous sections. In several cases **NEA could act as facilitator**, but efficiency can only be guaranteed if (whenever needed) suitable **agreements are signed and appropriate funding is provided** (not only to cover meeting and travel expenses of experts, but also for the work performed in preparation of them):

- In several cases it is necessary to develop specific procedures for testing and characterization of materials, e.g. in the case of testing in liquid metals, of performing mechanical tests on tubes, especially ceramic composite tubes, or of testing small or miniaturized specimens. Likewise, shared standard protocols need to be applied to validate microstructural and microchemical evolution models, for the treatment of the results of microstructural characterization techniques, as well as for the way in which model results, especially when the atomic-level is involved, should be analysed, for a fair comparison to experimental results and between simulation results. Thus one important issue is the **harmonisation of test and characterisation methodologies and procedures at international level**, as pre-normative step to standardization by bespoke bodies (ASME, CEN, ISO, …): support to **round-robin exercises** addressing non-standardized mechanical tests and development of best practices for microstructural characterization and data analysis, with **exercises of inter-comparison between laboratories for specific microstructure examination techniques** (TEM, APT, SANS, PAS, …).
- Experimental data are the basis for design curves and rules and should therefore remain available for reassessment in the context of new models, different operating conditions, different regulatory requirements, etc. It should therefore become standard practice for the data to be collected, preserved, and made easily discoverable, **especially for industrial benefit**, using an appropriate database and data management system. Other crucial issues are then: (i) the **identification of unified international databases of reference** for the collection of materials testing data; (ii) the establishment and distribution of relevant **data templates compatible with selected reference database(s)**; (iii) the establishment of **international rules and implementation review protocols concerning protection and disclosure of data collected in databases** (e.g. 10 years embargo on proprietary data); (iv) **encourage data upload** (this is always the bottleneck in the case of databases, thus grants should be accorded to support financially data seekers and collectors; if enforceable, data upload should be mandatory), etc.
- The quantity of materials data to be produced and collected for whichever purpose (design rules, modelling, …) is staggering. The exposure, testing and characterization of materials requires wide availability of specific and often costly infrastructures. In this respect, given the limited amount of resources, it becomes important to address the problem the construction, maintenance and exploitation of facilities in a rational way at international level. Thus it is important to **oversee the**

use of major research facilities worldwide (mainly materials test reactors, but also facilities for the exposure of materials to high temperature and coolants), based on existing lists and maps, designing **joint experimental programmes of (possibly) cross-cutting interest, using available large facilities in a coordinated way**. For example plan irradiation exposure jointly, taking advantage of available space in reactors on the occasion of certain experiments, especially for modelling purposes ("piggy back" irradiation) and then share the intensive cost in time and resources of the microstructural characterisation. It would be also desirable, as further step, to **optimise the complementarity of facilities**, driving similar facilities to non-overlapping uses.
- Because of the wide spectrum of possible modelling approaches, <u>progress in this field would be boosted if simulation tools were developed to be compatible with each other in terms of format</u>, input and output, and as much as possible complementary with each other, or providing consistent input to each other towards integration at different scales. Without of course posing limits to the development of new codes, in some cases it may be useful to <u>establish standards and reference simulation codes, promoting inter-code comparison as an essential benchmarking exercise</u>. This implies, however, **international agreement about broad goals and coordination in terms of development of tools, fostering harmonized development of tools for modelling and simulation compatible with, and complementary to, each other**. Effort should be therefore made towards better mutual complementarity and compatibility, possibly based on the consensual identification of gaps, ensuring also modelling data collections (according to criteria similar to the above ones applying to testing and characterisation).
- Finally, in connection with all the above, i.e. harmonisation of testing procedures, population of reference databases, rational use of infrastructures and coordinated development of models, researchers must be enabled to work together and cross-fertilise, thus **funded schemes for trans-national and also trans-continental mobility of researchers** are essential.

5.5 Facilities and infrastructures

The realisation of the above programmes requires:
- Facilities for the exposure of materials to
  - High temperature for prolonged time, under load and not;
  - Specific coolants, stagnant and flowing (loops), under load or not;
  - Irradiation facilities: materials test reactors, ion beams, …
- Infrastructures to handle irradiated materials:
  - Hot cells
  - Nuclearised experimental characterisation facilities
- Materials characterisation techniques (nuclearized and not):
  - Wide range of mechanical testing facilities
  - Microstructure examination equipment
- Computational facilities
- Instruments to enhance international cooperation on the points in 5.4