PROJECT FINAL REPORT

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4.1 Final publishable summary report

4.1.1 Executive Summary

The objective of the GETMAT project was the 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. Since the design requirements of the innovative reactors are beyond those for the LWR currently in operation, the selection of the properties to be investigated has been performed by identifying relevant conditions of key components as cores and primary systems. In fact, high temperature, high irradiation doses, corrosive environments as well as high thermal and mechanical loads are features that pose challenges on all materials foreseen for these components. Taking into account this framework and the goals defined by the Generation IV and the waste transmutation approaches as e.g. sustainability in terms of optimisation of use of resources and waste minimisation, high safety standards, economic viability and efficiency, it turned out that innovative materials might be a better choice with respect to conventional nuclear grade steels for the design of the core components and the primary system. Therefore, ODS alloys and 9-12 Cr Ferritic/Martensitic (F/M) steels have been selected as reference for the GETMAT project. In particular, even if ODS alloys are known since several decades, data for nuclear applications are quite scarce. The scientific results produced within the GETMAT project on 9Cr, 12Cr and 14Cr ODS alloys fabricated via the powder metallurgy route as well as the results obtained on the 9-12Cr F/M steels through an important PIE program, contribute considerably to a better understanding of the behaviour of these classes of materials for nuclear application. Moreover, the results have been found to be highly relevant for the identification of further R&D activities defined within the EERA Joint Program for Nuclear Materials (JPNM) and the related projects MATTER and MatISSE supported by the EURATOM FP7. In concordance with the experimental investigation on F/M steels and Ferritic / F/M ODS alloys the theoretical program was devoted to the study of Fe and Fe-Cr alloys. Emphasis has been put on first principle modelling studies and experiments to identify and validate physical models and mechanisms that explain irradiation hardening and embrittlement of these alloys. The results obtained are very promising and supportive in explaining phenomena occurring on Fe-Cr steels. Moreover they have been the basis for further R&D activities included in the JPNM and the MatISSE project.

The GETMAT results contribute also to the development of other technical areas as welding and joining of ODS alloys and the development of corrosion protection barriers for structural materials to be used in HLM-cooled systems.

Finally, within the GETMAT project high priority has been given to Education and Training. The scientific program of GETMAT has allowed the involvement of about 20 PhD students with experimental and theoretical work being part for their respective dissertations. Moreover, educational activities have been further supported through the organisation of two summer-schools and two dedicated workshops. The schools were organised in cooperation with other EU projects like PERFECT60 and MATTER and the workshops had a wide international participation, beyond the European boundaries. These educational activities have been very successful in terms of attraction of both well renowned lecturers in the field of materials science and of a quite significant number of young students.

4.1.2 List of all beneficiaries

Beneficiary Number *	Beneficiary name	Beneficiary short name	Country
1(coordinator)	Karlsruhe Institute of Technology	KIT	Germany
2	Commissariat à l'Energie Atomique	CEA	France
3	Studiecentrum voor Kernenergie/ Centre d'étude de l'Energie Nucléaire	SCK-CEN	Belgium
4	Ente per le nuove Tecnologie l'Energia e l'Ambiente	ENEA	Italy
5	Paul Scherrer Institute	PSI	Switzerland
6	Nuclear Research and Consultancy Group	NRG	Netherland
7	Centro de Investigaciones Energeticas Medioambientales y Tecnologicas	CIEMAT	Spain
8	Electricité de France SA	EDF SA	France
9	Helmholtz Zentrum Dresden-Rossendorf	HZDR	Germany
10	Université Libre de Bruxelles	ULB	Belgium
11	Kungliga Tekniska Högskolan	KTH	Sweden
12	The University of Liverpool	UL	United Kingdom
13	The University of Edinburgh	UEDIN	United Kingdom
14	University of Alicante	UA	Spain
15	University of Helsinki	UH	Finland
16	Materialpruefungsanstalt Universitaet Stuttgart	MPA.USTUTT	Germany
17	Consiglio Nazionale delle Ricerche	CNR	Italy
18	Centre National de la Recherche Scientifique	CNRS	France
19	Ústav jaderného výzkumu Řež a.s	UJV	Czech Republic

20	Joint Research Centre (IE, ITU)	JRC	Belgium
21	Technical Research Centre of Finland	VTT	Finland
22	Chalmers University	CHALMERS	Sweden
23	Universidad Politécnica de Madrid	UPM	Spain
24	Ricerca Sistemi Elettronici	RSE	Italy

4.1.3 Summary description of project context and objectives

4.1.3.1 Project context

The key elements at the origin of the GETMAT project have been identified through a thorough analysis performed on innovative reactors that are of relevance within the European Framework. In particular, reactor systems developed for advanced nuclear fuel cycles as well as advanced LWR were essential. Advanced nuclear fuel cycles include technological approaches that allow an optimised use of resources and a safe and optimised radioactive waste management. Nuclear reactors that are able to respond to these objectives are those with a fast neutron spectrum. Two categories of fast neutron reactors have been gaining attention in Europe, i.e. the sub-critical accelerator driven system (ADS) and the fast reactors (FR). Moreover, Supercritical Light Water Reactors (SCWR), considered as technological advancement of current LWR technology, were included in the analysis. A number of EURATOM supported projects (e.g. EUROTRANS, ELSY/LEADER, ESFR, GoFastR, HPLWR) that were addressing the design of these reactor systems and related materials as well as technological issues have been included and evaluated. The analysis was conducted in order to identify key reactor components for which advancement in the area of nuclear materials resulted to be crucial for the envisaged innovative design solutions. It turned out that the cores and primary systems are the most challenging components for what concerns materials requirements, thus materials selection and qualification. In general, all innovative reactor concepts foresee operational temperatures which are higher with respect to the temperature ranges of LWR (the current technology). Therefore, it can be assumed that all components of these reactors would be exposed to more severe conditions as compared to the established technology. However, the highest temperatures as well as dose rates can be expected at core components and primary systems levels. Therefore, the focus of the GETMAT project has been on these components. The analysis has also shown that candidate materials for cores and primary systems of almost all reactor concepts considered were austenitic and 9-12Cr Ferritic/Martensitic (F/M) steels. However, limits of these two classes of steels could have been identified. These limits impose design constraints that might not be in line with the original objectives declared for an enhanced sustainability and for innovations. Exemplary of these limits and their consequences especially for the fast neutron systems is the selection of the fuel clad materials. The austenitic steel option imposes an upper fuel burn-up limit due to irradiation swelling and embrittlement phenomena that occur on this class of steel for high irradiation doses (as discussed in past FR development programs). The limitation of the fuel burn-up might considerably impact strategies related to sustainability as e.g. the optimisation of use of resources. Limits can be as well identified for the 9-12Cr F/M steels as clad material. Indeed, the temperature window of these steels in an irradiation environment might be limited in the lower range to temperatures above 350°C and in the upper range to temperature below 550°C. The lower limit is due to irradiation hardening and embrittlement phenomena that can occur below 350°C (as discussed e.g. in the SPIRE project) and the upper limit is due to poor thermal creep and stress-to-rupture resistance of these steels above 550°C (as discussed e.g. in the EUROTRANS project). These temperature limits might impact the realisation of innovative design solutions as well as efficiency and economic viability of the reactor systems.

Therefore, within GETMAT the possibility to use oxide dispersed strengthened (ODS) alloys with a ferritic or F/M steel matrix has been proposed as an alternative option to the conventional steels. The focus on ODS alloy was based on data reported in the scientific literature, where it is discussed that these alloys show improved irradiation swelling and improved thermal-creep resistance in the high temperature range. The envisaged applications of ODS alloys were the fuel cladding for all considered reactor systems, i.e. ADS, FR and SCWR, and some structural components of the core as e.g. core support plate. Within this context properties of the ODS alloys that are of relevance for the given applications (and for all reactor systems) have been investigated. The preliminary assessment of the ODS alloys as materials for nuclear application, has been rounded up with an investigation of possible techniques able to join/weld these materials. Moreover, specific tasks related to environmental effects (corrosion and potential mechanical properties degradation) and options for mitigation of these effects through e.g. the development of corrosion protection barrier have been addressed as well. The environmental effects have been analysed for molten lead, helium with controlled impurities and supercritical water.

The development, optimisation and qualification of ODS alloys for nuclear application is considered as a long-term activity due to the fact that knowledge and relevant data on these materials for the nuclear applications are quite scarce. Therefore, selected needs for short- and medium-term applications were taken into account as well. The short- and medium-term plans foresees the availability in Europe of prototype and demonstration FR and ADS systems. For these systems conventional steels as 9-12Cr F/M and the austenitic steels AISI 316L, 15Cr-15Ni/Ti are candidate materials for both core components and primary systems. The GETMAT contribution to the design and construction of the prototype and demonstration systems occurred through the behaviour assessment of these candidate materials in different irradiation fields and in presence of relevant environments (heavy liquid metals (HLM) Pb and Pb-Bi eutectic, sodium and gas atmosphere). The activities were defined so as to take advantage, through post irradiation examinations (PIE), of relevant available and completed irradiation campaigns that were performed in past European projects and national programs. Moreover, it was also possible to include in the project a new irradiation campaign in a fast neutron spectrum and HLM environment and the related PIE.

A further topic considered as cross-cutting for all innovative reactor systems is the physical understanding of materials behaviour in terms of microstructure and mechanical properties evolutions in irradiation, temperature and stress fields. The physical understanding of material behaviour foresees the prediction of basic mechanisms, from atomic to the microscopic level, which determine materials response to applied thermal and mechanical loads, while being exposed to neutron irradiation. This research area includes the development of both physical models and computational tools for the different scales (from atomic to macroscopic level). Moreover, the theoretical activities are supported by dedicated model experiments. Since the multiscale modelling and experimental validation of nuclear material is a quite wide area, modelling and experimental activities included in the GETMAT project have been defined on the basis of the research direction in terms of composition and properties identified for the candidate alloys and steels. Indeed, the focus of the modelling activities was on Fe and Fe-Cr alloys (representative of F/M steels) and key aspects included in the investigation were thermo-chemical data (phase diagram of Fe-Cr) and irradiation hardening and embrittlement of these alloys. The theoretical activities were accompanied by dedicated irradiation experiments (in both ion and neutron fields) to assess assumption and models.

The overall challenges and research area of the GETMAT project can be summarised as follows:

- ODS alloys procurement and their characterisation
- Assessment of joining and welding techniques for ODS alloys
- Development and definition of corrosion protection barriers
- Improvement and extension of conventional steels (9-12 Cr F/M and austenitic steels) qualification
- Improved physical understanding of Fe and Fe-Cr alloys in irradiation and temperature fields and validation experiments.

4.1.3.2 Project objectives and S&T results

To address these challenges the GETMAT project has been structured into four work-packages and for each work package specific objectives have been identified. These objectives and the description of the main S&T results are hereafter summarised.

WP1 - Metallurgical and mechanical behaviour

The main objective of this work package is to procure and distribute the GETMAT ODS alloys and evaluate the basic physical and mechanical properties of these alloys.

Fabrication of ODS alloys is performed following classical powder-metallurgical (PM) processes that involve different steps as mechanical alloying (MA), consolidation by HIP and/or hot extrusion, and final thermo-mechanical treatments to achieve required mechanical performance. This fabrication procedure is expensive and the final quality depends strongly on a large amount of parameters (e.g. mechanical alloying time and velocity, consolidation temperature, and final heat treatments) that gives some uncertainties for what concerns the reproducibility of heats. Within the WP1, ODS alloys were produced by PM processes but other techniques that are based on more conventional steel production, as melting and casting, were explored. Unfortunately, these melting techniques do not allow the production of quality ODS alloys mainly due to the agglomeration of the oxide particles during the fabrication process.

Three ODS alloys with different Cr contents (14Cr, 12Cr and 9Cr) where produced by MA and hot extrusion see Figure WP1.1 and chemical composition reported in table WP1.1. However, first trials to produce a P91-ODS alloy consolidated by HIP do not reached the expected results, due to the porosity of the heats that leads to the cracking of the slabs during hot-rolling. Therefore the heat was procured through the KOBELCO company.

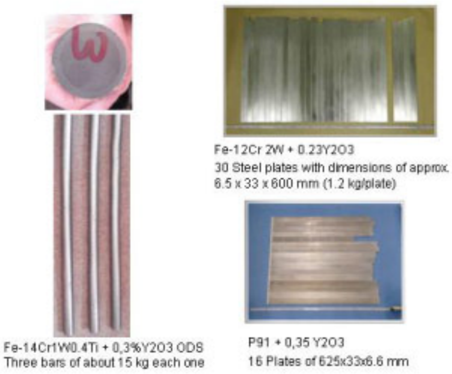


Figure WP1.1: ODS alloys produced within GETMAT project

The microstructure of the hot-extruded 14Cr ODS bar is constituted by small elongated grains (cigar shape) along the extrusion direction (ED) with a preferential crystallographic orientation of the grains along <110> in ED. This anisotropic microstructure is typical of ODS alloys and affects the mechanical properties as it will be explained later on this report. Nano-phases (Y-Ti-O) are uniformly distributed all over the grains and Cr-rich phase decorating some grain boundaries were also observed. The 12Cr ODS plates show a quite different microstructure, with a bimodal grain size distribution: small grains size < 1 μm heavily deformed and some elongated grains size up to 200 μm. The biggest grains have not got a preferential orientation, while the smallest grains seem to have a preferential orientation along <110> in the rolling direction (RD). Both the 12Cr ODS and the P91-ODS show elongated pores along the extrusion direction.

Table WP1.1: Chemical composition of the GETMAt ODS alloys

9Cr ODS	Wt.%	Fe	Cr	N	Mo	C	Y	Y_2O_3	
		base	8.9	0.016	0.76	0.095	0.28	0.36	
12 Cr ODS	Wt.%	Fe	Cr	N	Mo	Ti	Y	Y_2O_3	
		base	12.2	0.009	<0.01	0.31	0.17	0.22	
14 Cr ODS	Wt.%	Fe	Cr	W	Mn	C	Si	Y	Ni
		base	13.5	0.9	0.27	0.09	0.32	0.2	0.16

Tensile properties have been determined up to 850ºC by different laboratories involved in the GETMAT project, see Figure WP1.2. In general the results from the different laboratories are consistent among them. Moreover, the GETMAT 14Cr-ODS and 12Cr-ODS alloys show lower strength than the well-known 14YWT and 12YWT fabricated by ORNL (see figure WP1.2). Interesting to note that the three GETMAT ODS alloys show a peak in the elongation at 600ºC, typical for similar ODS alloys. This enhanced ductility at 600ºC can be attributed to a change on the deformation mechanism from intraganular to intergranular mechanisms, as is reported in the scientific literature and it can also be related to an inverse strain rate sensitivity showed by the 14Cr ODS alloy.

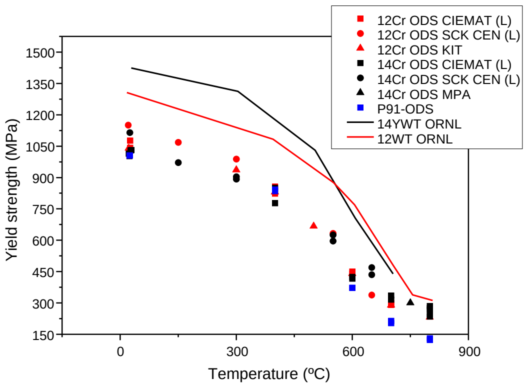


Figure WP1.2: Tensile properties of GETMAT ODS alloys in comparison to the ORNL ODS 14YWT and 12YWT

The effect of the microstructural anisotropy on the tensile strength is not so evident for temperatures up to 600ºC. However, for higher temperatures, the transverse orientation is weaker than the longitudinal one. The effect on ductility has been measured through the total elongation and the reduction in area. Moreover, the loss of reduction in area for the T orientation in respect of the L is around 70% at room temperature and around 50% at 700ºC.

Impact properties of the three ODS alloys has been obtained by testing small KSLT specimens. In general the DBTT and absorbed energy are strongly dependent from microstructural features, showing the effect of the microstructural anisotropy on the mechanical properties. The observed difference in the values of absorbed energy is a consequence of the relative orientation of the notch with respect to the elongated microstructure. When weak interfaces are present parallel to the longitudinal direction of the impact test bar, the interaction between the weak interfaces and the stress field that is generated by the localized plastic constrain at the notch and/or the crack tip can cause splitting. The two basic geometries are termed ‘‘crack divider’’ and ‘‘crack arrester”. In the crack arrester geometry, the delamination is thought to relax the triaxial stress conditions and to blunt the crack tip. To fracture a material, crack re-initiation is necessary and occurs under conditions of nearly uniaxial tension, which is an unfavourable cleavage. Hence, high absorbed energy is obtained through the delamination in LS and TS samples by the well-known delamination toughening mechanisms. The load-displacement curves obtained from instrumented Charpy tests are very useful to investigate the effect of splitting on the DBTT, see Figure WP1.3. As shown in this figure the load-displacement curves of LT and TL samples present similar behaviour regardless of the test temperature; while the load-displacement curves for LS and TS samples present an unusual behavior. For LT and TL samples, at which ductile fracture surfaces were observed, the load gradually decreases after a maximum value Pm is reached. The LS and TS samples, at which extensive delamination was observed, the load-displacement curves exhibit load drops that can be associated to crack arrest, and loads plateaus that can be due to re-initiation of new cracks. In other words, the crack is blunted by delamination. As a result, the absorbed energy becomes larger by the delamination effects. Moreover, because of the blunted crack by delamination, the sample behaves as an un-notched sample during the test.

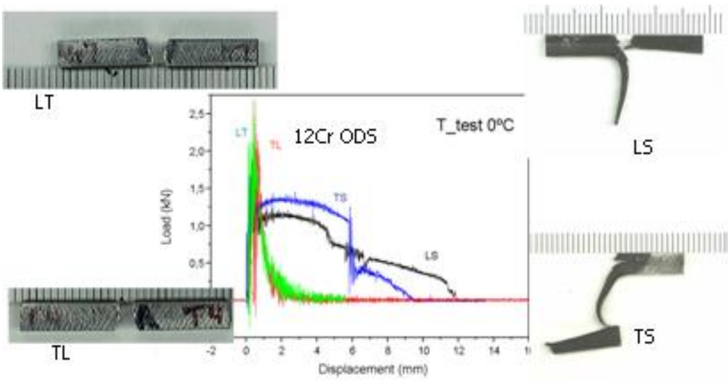


Figure WP1.3: Anisotropy on the impact behaviour of the 12Cr-ODS alloy (Courtesy M. Serrano, CIEMAT)

Anisotropic behaviour of the ODS alloys have been also observed on the fracture toughness tests, where the TL specimens show very low fracture toughness values at 550ºC and 650ºC.

Impact and fracture toughness resistance are properties normally not considered during the design of high temperature components. However, from a safety point of view it is mandatory to demonstrate that the components perform as expected under all conceivable conditions. For this purpose properties as impact and fracture toughness are relevant.

Uniaxial creep tests, small punch creep tests and low cycle fatigue tests under fully reversed conditions (R = -1) in strain control with a strain rate of 0.2%/s in air and in inert atmosphere have been conducted on the 14Cr and 12Cr ODS alloys.

The creep curves for the GETMAT 14Cr ODS alloy are characterized by very slow secondary creep and sudden rupture at very low deformation with little evidence of tertiary creep. Similar observations have also been reported for other ferritic ODS steels in the scientific literature. For a given stress level, the rupture times obtained from the creep tests were found to exhibit considerable scatter with deviations reaching more than one order of magnitude, see figure WP1.4. This large scatter in the rupture times might not be ascribed only to the stochastic nature of a brittle final fracture event, as the documented absence of tertiary creep might suggest, but rather by scatter already in the steady state creep rates.

Fatigue tests have been carried out at 650 ºC and 750 ºC. The alloy is under cyclic load very stable with practically no hardening/softening effects. Results from the tests at both temperatures can be described by a common power law, see figure WP1.5. An increase in the test temperature has little influence on the fatigue ductility exponent. For a given total strain level, the fatigue life of the alloy is reduced with increasing temperature. Creep fatigue tests (with 10 min hold-time) show smaller number of cycles to crack initiation, there is nearly no difference between results of tests carried out under inert and air environment.

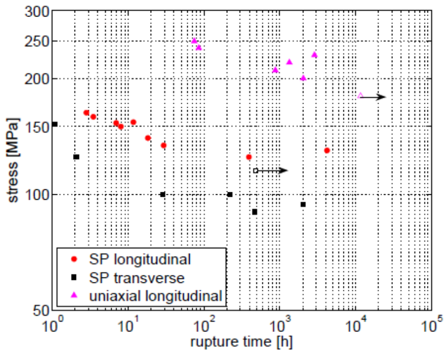


Figure WP1.4: Creep Stress-rupture data for small punch creep and uniaxial creep tests of the 14Cr ODS alloy at 650 C. The open symbols with arrows indicate specimens that did not rupture. (Courtesy M. Bruchhaussen, JRC-IET)

Irradiation creep and microstructural changes of the 12Cr-ODS and 14Cr-ODS alloys have been studied by homogeneous implantation of helium under uniaxial tensile stresses from 40 to 300 MPa. The maximum dose was about 1.2 dpa (5000 appm-He) with displacement damage rates of $1{\cdot}10^{-5}$ dpa/s at a temperature of 300 ºC. Irradiation creep compliances were measured to be $4.0{\cdot}10^{-6}$ $dpa^{-1}{\cdot}MPa^{-1}$ and $10{\cdot}10^{-6}$ $dpa^{-1}{\cdot}MPa^{-1}$ for 12Cr and 14Cr ODS, respectively. No remarkable effects of Cr content, grain size and dispersoid size on irradiation creep properties are observed. The apparently slightly higher compliance value of the 14Cr ODS cannot be unambiguously related to any material property or microstructural evolution.

Microstructural evolution was studied in detail by TEM observations. Dislocation loops and helium bubbles are distributed homogenously in the matrix. In the case of high density fine dispersoids, most bubbles are attached to oxide particles. This may suppress loop formation as well as growth of bubbles, thereby increasing the resistance of ODS ferritic steels against helium embrittlement.

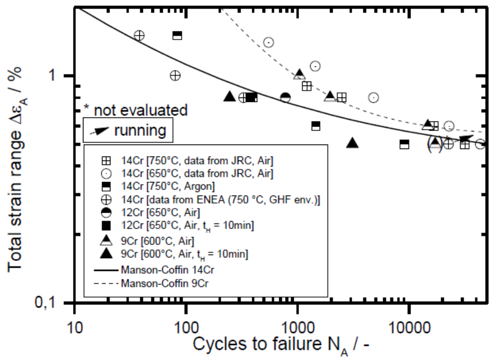


Figure WP1.5: Number of cycles to failure (N_f) as a function of total strain (Courtesy MPA, JRC and ENEA)

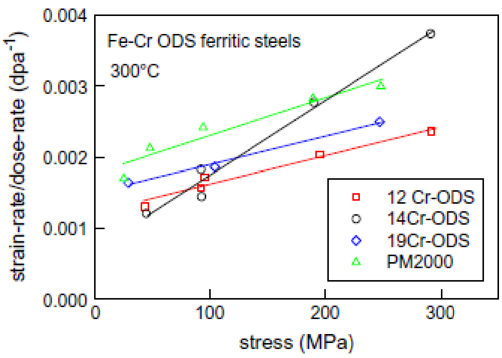
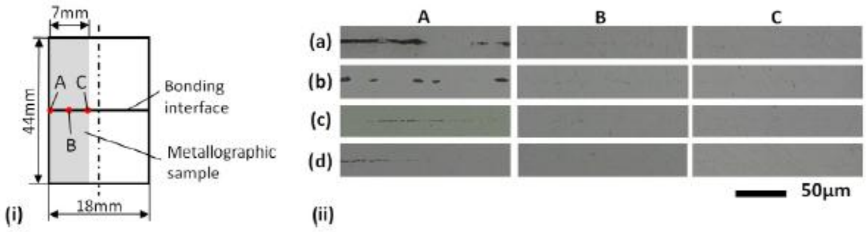


Figure WP1.6: Irradiation creep rates per displacement rates of 12Cr ODS and 14Cr ODS steels as a function of tensile stress under He-implantation at 300 ºC. Linear fits give irradiation creep compliances. The 19Cr ODS and PM2000 data are included for comparison. (Courtesy J. Chen, PSI)

The welding techniques explored within the GETMAT project were the electro-magnetic pulse technique, diffusion bonding, explosive and friction stir welding. Electron beam and TIG welding techniques have also be investigated for mixed welds.

Due to limitations of the GETMAT ODS alloys quantities, the diffusion bonding activities have been performed on PM2000. In this framework it has been seen through hardness measurements that heat treatment of PM2000 above a temperature of 1200 ºC causes a degradation of mechanical properties. TEM investigations as well as optical light microscopy revealed that neither a constant particle size distribution nor the changes in the grain sizes of small grains can be identified as a clear reason for the temperature induced loss in hardness. The diffusion bonding model of Hill and Wallach was adapted by using the creep equation for ODS materials introduced by Rösler and Arzt. The influence of pressure, temperature and surface roughness on the bonding time were investigated, see Figure WP1.6. For a diffusion temperature of 1100 ºC, a pressure of 50 MPa and a surface roughness of b = 2.5 µm and h = 1.2 µm similar UTS and Charpy impact energies compared to the bulk material were achieved



Diffusion bonding parameters used for bonding experiments.

	p (MPa)	T (°C)	t (h)	b (µm)	h (µm)
(a)	30	1300	2	3.5	1.5
(b)	20	1300	2	3.5	1.5
(c)	50	1100	2	2.5	1.2
(d)	50	1100	2	4.2	1.6

Figure WP1.7: Light microscopy pictures of diffusion bonded PM2000 samples. (i) Sketch of the location of the investigated spots. (ii) Microscopic images of bonding seam. The bonding conditions are marked by (a–d) (Courtesy W. Sittel, KIT)

Electromagnetic pulse technique has been used to weld T91 cladding tube and to assess to use this technique in hot cells. The investigation focussed on the angle of collision between the moving component (the tube) and the static central end cap. This angle has a strong influence (supplier's information) on the interaction between the components during the deformation but it also determines the flight distance of the tube prior to collision. This distance is critical in allowing an optimization of the acceleration achieved by the tube as a result of the electromagnetic pulse. The objective of defining a parameter window for the joining of advanced cladding materials (T91 and ODS steel) has been achieved. A joined sample with refined geometry is shown in Figure WP1.8.

Figure WP1.8: EMPT joined sample (Courtesy A. Cambriani, JRC-ITU)

Linear Friction Welding (LFW) is a solid state joining process in which a stationary material part is forced against a part that is linear oscillating to generate friction heat. Important parameters are oscillation frequency and amplitude, friction and forge pressure and burn-off. The joining of 12Cr ODS alloy by LFW has been successfully demonstrated. A clear thermo-mechanically affected zone (TMAZ) with deformed and more refined microstructure containing uniform dispersion of particles was observed in both longitudinal and transverse directions. The micro-hardness changed from 370 HV in the parent material to 330 HV in the TMAZ. The minimum hardness values, 170-190 HV, occur very close to the weld interface (probably due to dynamic recrystallization), see Figure WP1.9.

Explosive Welding (EW) is a solid state process where welding is accomplished by accelerating one of the materials parts at extremely high velocity through the use of chemical explosives. Final explosive welds of 14Cr ODS alloy are performed to investigate the welding window for these type of materials (see figure WP1.10). A wide weldability window (between 2000 and 4000 m/s, collision point (welding) velocities) exists for welding 14Cr ODS steel. Due to extremely high strength and low ductility, explosive welding should be done with a pre-heating at about 200°C and with increased impact velocity (height of explosive layer) compare to conventional steels.

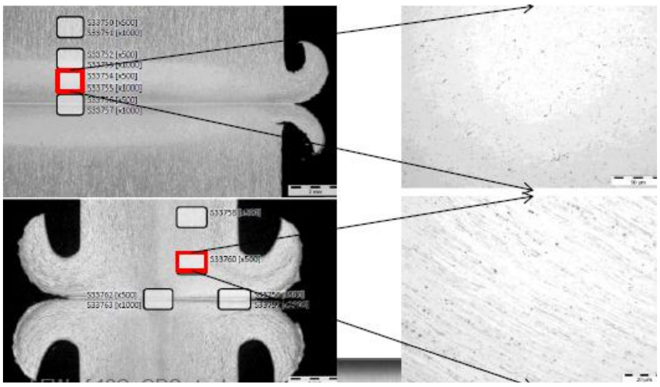


Figure WP1.9: LFW of 12Cr-ODS alloy (Courtesy M. Kolluri, NRG)

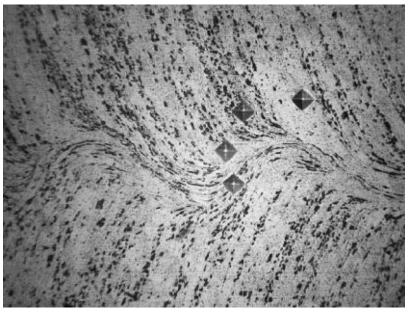


Figure WP1.10: explosive welding of 14Cr-ODS (Courtesy N. Luzginova, NRG)

TIG (Tungsten-Inert-Gas) and EB (Electron Beam) welds of P91, P91-ODS, P92 and PM2000 were performed, see Figure WP1.11. Dissimilar (P91/PM2000) EB welds have been obtained. However, Charpy and stress-to-rupture test results showed that the welded part is more brittle than each of the base material showing that this technique might not be the optimal for dissimilar weld with ODS. Moreover, EB welding of ODS P91 with ODS P91 failed.

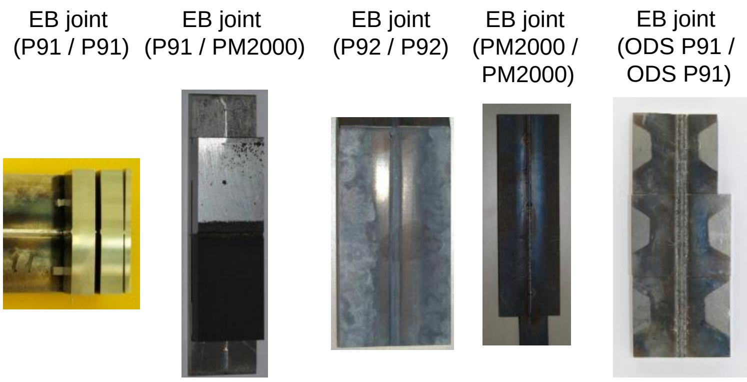


Figure WP1.11: EB joints of F/M and ODS alloys (Courtesy V. Widak, KIT)

WP2 – Materials' compatibility with coolant

This work package is divided into three main thematic areas, i.e. compatibility of ODS alloys and conventional steels with different reactor coolants, corrosion barriers development and impact of coolant chemistry on the mechanical properties of ODS alloys and conventional steels. The reactor coolants considered were molten Lead (Pb) and Lead-Bismuth Eutectic (LBE), impure He (impurities were defined on the basis of assumed GFR reference atmosphere) and supercritical water (SCW). These coolants were selected to contribute the development of Lead Fast rectors and Accelerator systems (LFR and ADS) both cooled with Pb and LBE respectively, to GFR and SCWR. In the following the results obtained in the three areas are summarised.

Compatibility of ODS alloys and conventional steels with different coolants

The compatibility test matrix performed in molten Pb and LBE is reported in the table WP2.1 where "Stag." stays for stagnant. As shown in this table the durations of the tests were different. In addition further differences in the experimental conditions and materials tested by the involved laboratories are:

- ENEA: flowing tests in the LECOR with oxygen concentration of 10^{-6} wt.%; materials tested: 15Cr-15Ni Ti stabilised austenitic steel up to 8000 hours, ODS alloys, up to 5000 hours
- CIEMAT: stagnant tests in Pb; oxygen concentration of 10^{-5} wt. % and 10^{-7} wt. %;
- CNR: stagnant tests in Pb at a temperature of 650°C and 700°C; In addition CNR has also performed wetting tests.
- KIT: stagnant tests in Pb; oxygen concentration of 10^{-6} wt. % and 10^{-8} wt. %.

Table WP2.1: Test matrix of the compatibility tests performed in Pb

ENEA	Flow. 1m/s	500°C	Up to 8000 hrs	ODS, 15-15Ti
CIEMAT	Stag	600°C, 700°C	2000 hours	ODS
CNR	Stag	650°C, 700°C	2000 hours	ODS
KIT	Stag.	550°C	2000hours	Welds

Considering the wide amplitude of the experiments and the different approaches in performing them, the outcomes and experimental data were surprisingly similar. Indeed, surface oxidation on all tested samples could be observed. This is shown in figure WP2.1, where the cross section of the 12Cr ODS alloy tested in the different laboratories are reported.

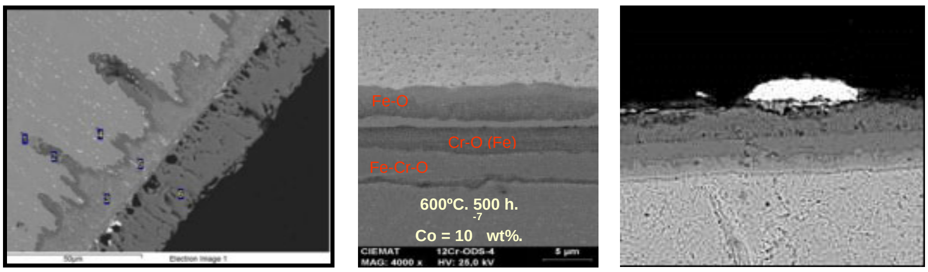


Figure WP2.1: Cross section of samples tested in Pb. All samples show surface oxidation. The oxide layers from external towards the bulk are mainly made of magnetite, spinel oxide and internal oxidation. Micrographs are from CNR, CIEMAT and ENEA.

Considering the above depicted examples on the 12 Cr ODS alloys, a first qualitative observation is possible: the oxidation mechanism of the material appears very similar between the flowing and the stagnant experiments. However, the appearance of the oxide layer, their thickness and their compactness were function of various parameters as e.g. the alloy composition, the oxygen control in the testing devices and the temperature. Moreover, all alloys tested by different laboratories, do not show any mass loss due to massive corrosion phenomena. In the high temperature range (above 600°C) even if the oxidation behavior of the three ODS alloys are very similar, some peculiarities of each sample can be highlighted. These are summarised in the scheme in Figure WP2.2:

- 9Cr ODS alloy shows an internal oxidation associated to the growth of a thin Fe-Cr-O layer on the surface.
- 12Cr ODS alloy shows a Fe-Cr-O layer growth on the steel surface and some spots of Cr-O oxide. Internal oxidation could not be detected.
- 14Cr ODS alloy shows a double oxide layer; an outer Fe-Cr-O layer and an inner Cr-O layer.

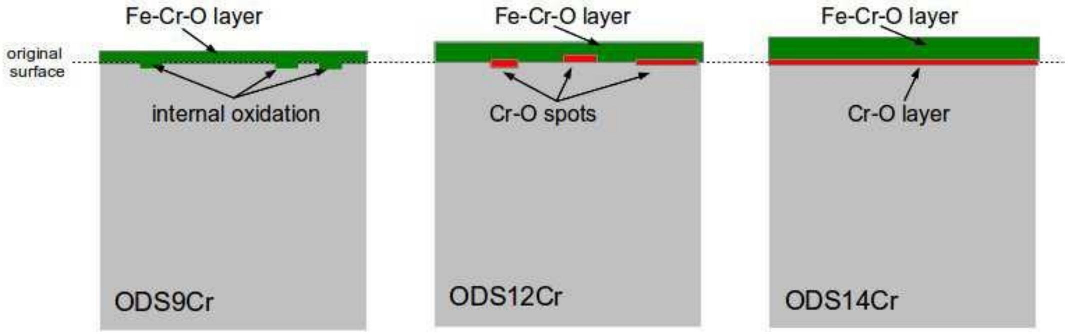


Figure WP2.2: schematics of ODS' oxidation behaviours in the high temperature range.

The thickness, the composition and the compactness of the Fe-Cr-O layer show a slight dependence on the Cr-content in the steel matrix. In fact 14Cr ODS presents a more compact and continuous layer (with a thickness of about 10 µm) than the other two ODS alloys.

The internal oxidation observed in the 9Cr ODS steel can be associated to its fine grained structure as already reported in the scientific literature.

Compatibility tests in Pb have been carried out as well on welded materials. These tests have shown that the chemical composition and the grain size in the weld region (due to a heat treatment) impact the corrosion/oxidation behavior. When these parameters in the welded area are similar to the bulk (as e.g. it was the case for the 9Cr F/M steels welded with EB, TIG and the friction stir welds) also the corrosion/oxidation behavior is comparable. While, for smaller grain size in the welded zones (as e.g. observed for friction stir welding) thicker oxide layer are observed. Moreover, precipitations of elements at the grain boundaries and/or voids can lead to a fast growing oxide or a stronger dissolution attack, like the one observed on the P91/PM2000 EB welded specimens. Finally, changes in the grain orientation and dislocation also may lead to a different behavior of the welding region, like observed at the specimens welded by explosive welding.

For what concerns the compatibility tests performed in He with controlled impurities, these have been done only the 14Cr ODS alloy by CEA, CIEMAT and ENEA. The controlled impurities in the He flow was for all experiments set as indicated in table WP2.2.

ENEA and CIEMAT manufactured dedicate facilities to perform the tests, whilst CEA adapted the Coralline loop. The approaches were quite different: ENEA and CIEMAT obtained the reference atmosphere by means of commercially available tanks, CEA mixed the impurities in situ with a dedicated system.

Table WP2.2: reference GETMAT controlled impurities in He.

Impurities	Helium composition [ppm]
H_2	180 – 200
CO (CO_2)	30- 40
CH_4	10 – 15
H_2O	0,2 - 2
O_2	0,1 - 1

The exposure times for all experiments were 250, 500 and 1000 hours, at 750°C, 850°C, 900°C, with a flow rate of 100 l/h ca. In figure WP2.3 cross-section of exposed specimen at the three temperatures are given

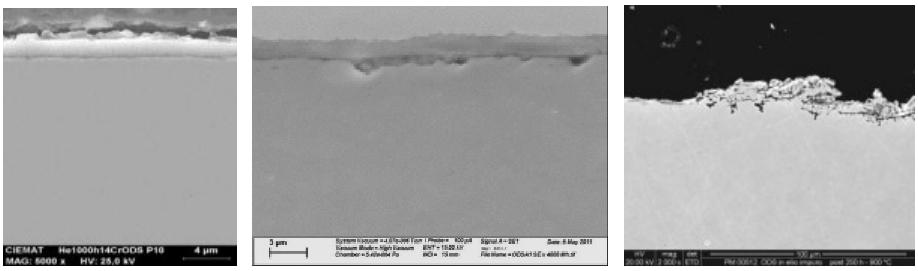


Figure WP2.3: 1000 hours exposures of 14 Cr ODS, at 750°C (left) (Courtesy CIEMAT), 850°C (middle) (Courtesy CEA), 900°C (right) (Courtesy ENEA).

The results gained in the three experiments, validate that the 14Cr ODS did oxidize in the test conditions as expected with the selected helium impurities and composition. Comparison of the measured mass gains and scale adhesion for Fe-14Cr steels with and without Y is consistent with a smaller oxidation rate and higher quality scale for the tested ODS alloys.

Based on these preliminary results, 14Cr ODS steel exhibits a promising corrosion behavior in oxidizing impure helium at 750, 850, 900°C. This comment is important if one thinks that the alloy has yet not been optimized regarding its oxidation properties. However, significant work would still be needed in order to validate the environment compatibility of this material for use in a GFR. An experimental qualification program would at least comprise: parametric studies to establish oxidation kinetics as a function of the environment parameters based on medium to long term oxidation tests, understanding of the oxidation mechanisms to get inputs for lifetime modelling including material aging and long term effects, experimental validation on long duration oxidation tests, investigation of the corrosion behavior in off-normal conditions (temperature outburst, out-of-specification coolant chemistry) and possibly an optimization of the alloy as regards its corrosion performances.

SCW was the third reference environment considered. The tests were performed only by the project partner VTT. The tests were done in SCW at two temperatures, 550°C and 650°C. The dissolved oxygen content in the feed water was between 100 and 150 ppb and the conductivity was below 0,1 µS/cm. The specimens were exposed to SCW at the pressure of 25MPa in an autoclave connected to a recirculation water loop. The environmental control and monitoring included temperature, pressure, inlet and outlet water conductivity, inlet water dissolved oxygen content, and flow rate.

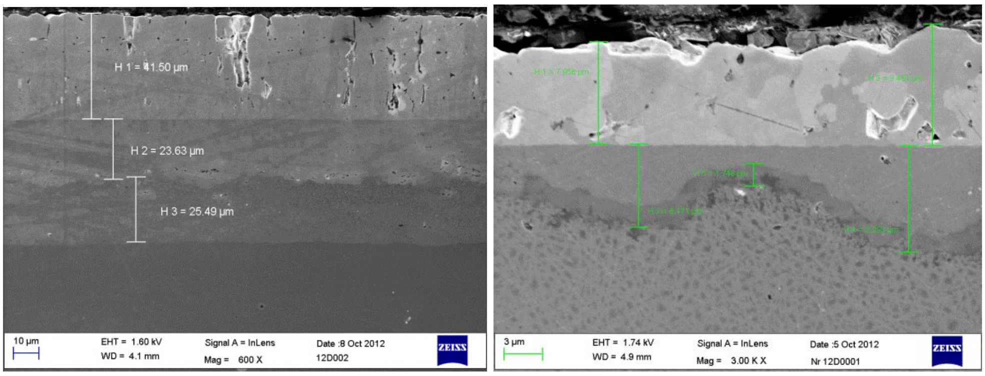


Figure WP2.4: Cross sections of 12Cr and 14Cr ODS samples after exposure to SCW at 550°C for 3000 hours (Courtesy VTT)

Based on the weight change tests and data analyses, the ODS alloys PM2000 and MA956 have superior corrosion resistance when compared to the GETMAT ODS alloys. The oxidation performance of PM2000 resulted from the formation of a protective Al–Cr–Fe-rich surface oxide layer. Based on this work, it has been deduced that in SCW conditions at 550 °C the Cr-content for thin walled ODS components must be higher than that of the 14% in order to produce protective and compact oxide layers.

Advanced corrosion barriers development

Surface modified layers based on FeCrAl coatings have shown in general their capability to protect steels against the influence of liquid lead alloys by the formation of slowly growing stable oxide scales. The goal of the activities was to perform a critical analysis of two different coating methods, and to identify the optimal composition of the corrosion protection barrier. The methods compared were HVOF and Laser treatment and LPS/VPS and GESA (pulsed electron beam) treatment. Both systems would need a very careful investigation in terms of parameters, alloy composition etc.

However, due to the fact that the laser treatment induces volumetric heating while the GESA concentrate the heating on the surface, it appears that GESA would produce corrosion protection barrier with superior mechanical performance. The next step was the identification of the most suitable composition of the coated layer to be applied in order to use the VPS/GESA approach. For this purpose, model alloys were produced by Arc- Melting to evaluate the Al and Cr content required for protective oxide scale formation.

The phenomena occurring during the Fe-Cr-Al alloys exposure to HLMs, containing very small amounts of dissolved oxygen is scarcely documented. A systematic investigation of the corrosion behaviour of the Fe-Cr-Al system, when exposed to HLM was performed, in order to find the minimum Al content for the formation of an alumina scale. Therefore a systematic study concerning the corrosion behaviour of Fe-Cr-Al-base model alloys during their exposure to oxygen-containing (10^{-6} wt. %) lead in the temperature range 400 - 600 °C has been performed. A test matrix considering the alumina stability domain for this temperature range has been drawn in Fe-Cr-Al ternary diagrams and the type of alumina polymorph, formed at different temperatures, was determined (see Figure WP2.5).

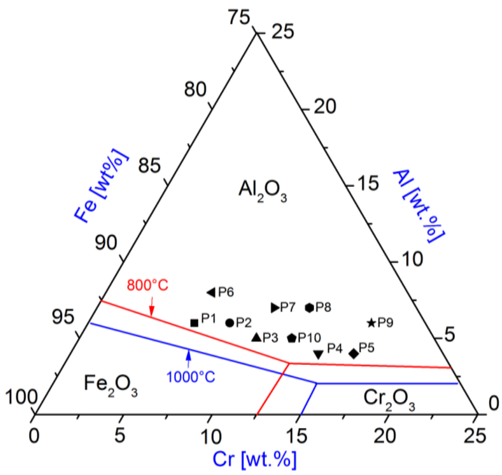


Figure WP2.5: Ternary phase diagram with compositions of prepared alloys and the oxide-maps drawn at 800 °C and 1000 °C (Courtesy A. Jianu, KIT).

Based on the state of the art oxide maps concerning oxidation behaviour of Fe-Cr-Al-base alloys at 800 and 1000°C in oxygen atmosphere, 10 compositions, belonging to this alloy system, were designed in order to tap the borders of the alumina stability domain, during their exposure to oxygen (10^{-6} wt.%) containing lead, at 400, 500 and 600°C. Experimental results obtained at 400°C are summarised in Figure WP2.6.

Eight alloys, Fe6Cr6Al, Fe8Cr6Al, Fe10Cr5Al, Fe14Cr4Al, Fe16Cr4Al, Fe6Cr8Al, Fe10Cr7Al and Fe12Cr5Al, were found to be protective against corrosion in oxygen containing lead, either by a duplex layer (Fe_3O_4 + $(Fe_{1-x-y}Cr_xAl_y)_3O_4$) or by $(Fe_{1-x-y}Cr_xAl_y)_3O_4$, depending on the temperature at which they were exposed.

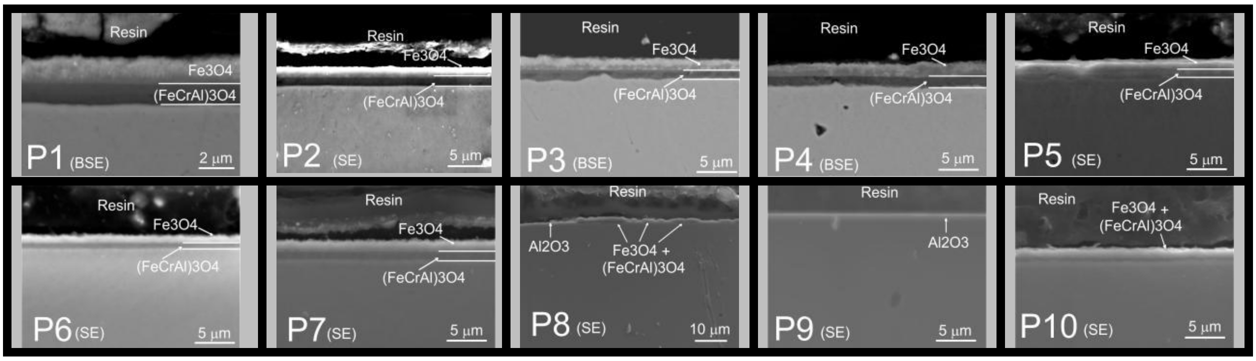


Figure WP2.6: cross sections example of samples made of Fe-Cr-Al-base alloys exposed to oxygen containing liquid lead for 840 hours at 400°C: P1 (Fe6Cr6Al), P2 (Fe8Cr6Al), P3 (Fe10Cr5Al), P4 (Fe14Cr4Al), P5 (Fe16Cr4Al), P6 (Fe6Cr8Al), P7 (Fe10Cr7Al), P8 (Fe12Cr7Al), P9 (Fe16Cr6Al), P10 (Fe12Cr5Al). P1 to P7 and P10 are covered by duplex scale (Fe3O4+Fe(Cr,Al)2O4). P8 is covered by a mixture of duplex and thin Al-rich oxide layers, while a thin Al-rich scale is grown on P9 (SE: secondary electrons image; BSE: back-scattered electrons image) (Courtes A. Jianu, KIT)

Two alloys namely Fe12Cr7Al and Fe16Cr6Al were found to form transient aluminas, $\kappa-Al_2O_3$ (at 400 and 500°C) and $\theta-Al_2O_3$ (at 600°C), as protective oxide scale against corrosion in oxygen containing lead. An oxide map illustrating the stability domain of alumina, grown on Fe-Cr-Al-alloys when exposed to molten, oxygen containing lead, was drawn. The alumina stability domain border shifts with lower temperatures to higher chromium and aluminium concentrations. When the concentration of Cr is in the range of 12-25 wt. %, the minimum concentration of Al, required to form alumina scale on Fe-Cr-Al alloys exposed at 400-600°C in molten Pb, can be calculated with the following equation: *C Al =1.523978 – 0.80805 (CCr) + 0.01561 (CCr)2 [wt%]*.

For the temperature range and exposure times used during the current evaluation, the growth rate of the alumina scale was low. No area with detached scale was observed and no trace of $\alpha-Al_2O_3$ was detected. Lower aluminium concentrations leads to the formation of Fe-base spinel-type oxides protrusions on extended area. However, this can also happen at high aluminium concentration (7.6 wt. %) if the chromium content is lower than 10 wt.%.

Mechanical testing in environment

Mechanical tests on the GETMAT ODS alloys were performed in Pb, He and SCW. The tests in Pb were creep-to-rupture and fretting tests; moreover several mechanical tests were performed on Pb corroded specimens. In He LCF tests and in SCW Slow strain rate tests were performed. In general the preparation and execution of mechanical tests in the different environments leads to very different approaches and obstacles.

Creep tests in contact with Pb were carried out by CIEMAT and KIT. Figure WP2.7 depicts, as an example, the role of HLM on the creep properties of the 9Cr ODS alloy. The tests have been performed in air and in liquid lead under non-controlled oxygen atmosphere at 600 °C (CIEMAT) and at controlled oxygen condition at 650 °C (KIT). The results show an important effect of liquid lead in the creep behaviour, principally lowering the rupture time of the specimens in liquid Pb under non-controlled conditions. On the contrary, creep test performed at KIT under oxygen controlled conditions show similar tendency in air and in Pb.

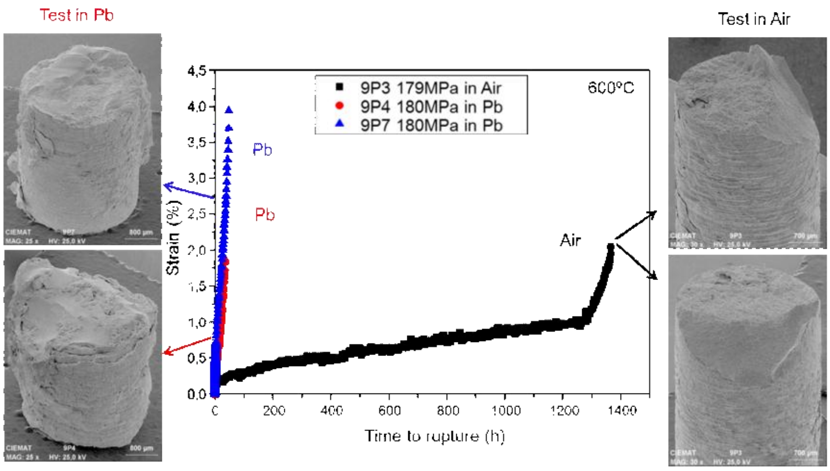


Figure WP2.7: comparison of 9Cr ODS creep properties in Pb without oxygen control and air (Courtesy M. Serrano, CIEMAT).

A possible reason could be the formation of a thick, porous and brittle oxide layer that is formed when the oxygen content in the liquid metal is not controlled. This layer breaks during the tests and allow an intimate contact between the liquid and solid metal, leading to a liquid metal embrittlement mechanisms.

Fretting experiments with the main goal to simulate the interaction between the spacer grid and the fuel cladding were performed on the 14Cr ODS GETMAT alloy at ENEA and on GESA treated and 15-15Ti samples at KIT. The two experimental devices were different. Both campaigns were carried out at 500°C, in Oxygen saturated Pb with comparable loads and fretting frequencies. However, while the FRETHME machine at KIT was able to test several specimens in one run, the ENEA GIORDI device could perform fretting experiments only on one sample. Figures WP2.8 and WP2.9 depict some of the interesting outcomes of the two test series.

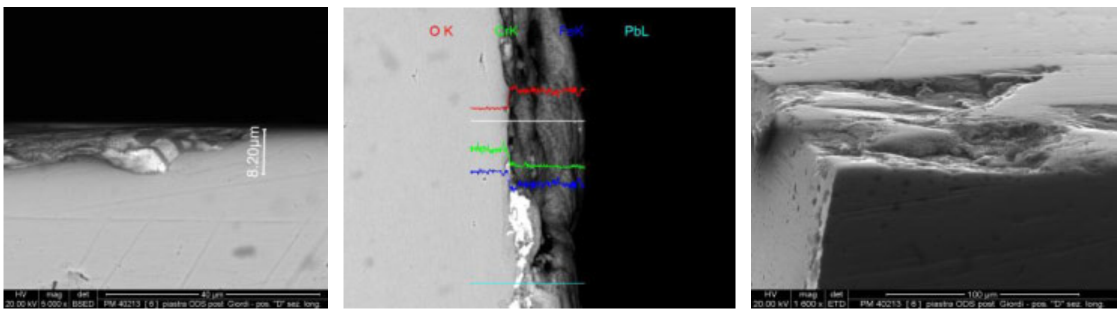


Figure WP2.8: Fretted 14 ODS in ENEA GIORDI: depth, linescan and 3d SEM imaging of the sample.

In summary the fretting tests have shown the following:

- Favorable load-amplitude combinations can minimize the fretting severity. The fretting penetration and the wear rate decrease with increasing applied load (after the turning point) and with a decreasing sliding amplitude. As displayed by the published fretting maps there are load-amplitude combinations (e.g. load > 75 N and amplitude < 15 µm) that favors the evolution of the fretting contact towards the stick regime. These load-amplitude combinations are expected to lower the specific wear coefficient (and consequently the wear and penetration rate) by up to one order of magnitude compared to e.g. the combination 50N and 15 µm, which correspond to the lowest measured penetration rates. Such conditions can be established with an adequate assembly strategy.
- Suitable material improvement and corrosion counter-measures improve the wear resistance and mitigate the corrosion enhancement of fretting.
- A reduction of the penetration rate up to about 50 % already after 150 h was achieved by using pre-oxidized components. Likewise, for the specific case of austenitic steels, the use of Ni-enriched Pb reduces the fretting corrosion severity.
- Additionally, surface treatment and Al-alloying (e.g. GESA-T91) improves remarkably the wear and the corrosion resistance property also at high temperature.
- Fretting is a self-mitigating process. Fretting becomes less and less severe with the time. After 930 h, the penetration rate is up to 70% lower than during the run-in period.
- The beginning of the fretting process is delayed with respect to the reactor start-up. In e.g. the reactor core, the fretting process starts when, due to the irradiation damage and thermal-mechanical loads, the components (e.g. springs, supports or spacer grids) aimed to fasten the fuel cladding tubes (or other parts) lose their mechanical and dimensional properties. Therefore, the fretting process affect the e.g. fuel cladding tube for less than 25000 h.
- Possible design solution like the installation of spacer grids and supports in the non-active area of the fuel (lower temperature and irradiation) can delay the beginning of the fretting process further.

It is possible to claim with a certain confidence that, the listed aspects will allow the achievement of a penetration rate complying with the operating requirement (0.002 µm/h), which is about one order of magnitude lower than the one measured experimentally (in the best cases from 0.039 to 0.063 µm/h for the three materials).

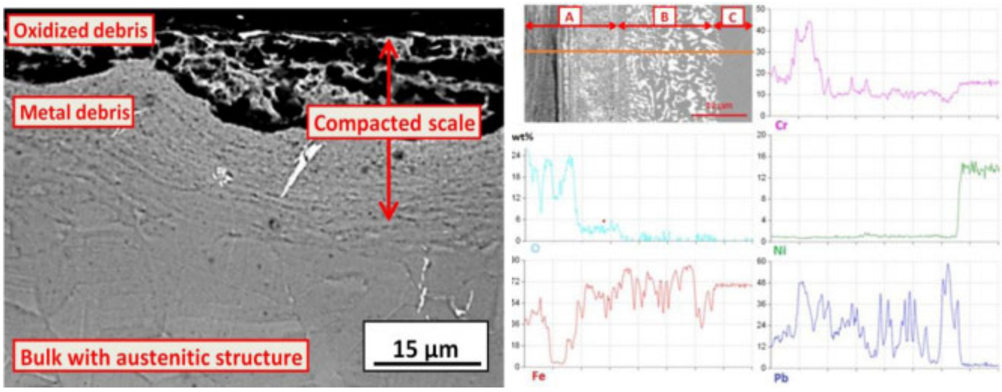


Figure WP2.9: 15-15Ti stainless steel fretted sample at KIT (Courtesy M. Del Giacco, KIT)

Finally the characterization of pre-corroded steels in liquid flowing Pb at ENEA rig LECOR at 500°C, the project partner RSE performed small punch tests. Following results have been obtained:

- No effects of long time exposure on tensile properties, neither when determined by small punch tensile for both 12 and 14 Cr ODS, nor when determined by uniaxial test as observed for 12 Cr ODS.

- Limited effect of exposure on creep rupture time reduction: 14 Cr ODS estimated by small punch creep show some tests with reduced rupture time but data can be considered into conventional creep scatter-band of reference not corroded material data, 12 Cr ODS estimated by uniaxial creep tests shows a trend of slightly more pronounced reduction for rupture times but data are always into the scatter of reference not corroded material. The observed creep resistance reduction can be related to the presence of penetrating oxidation on specimen surface enhancing localized stress concentration.

- Significant effect of exposure on material embrittlement observed for both ODS steels: a relevant increase of transition temperature estimated by small punch tensile for 14 Cr ODS and a relevant reduction of impact energy for 12 Cr ODS, the latest confirmed by a less relevant (than 14 Cr) but anyway evident increase of small punch tensile transition temperature.

LCF tests were conducted at RT and at 750 °C on 14 Cr-ODS steel specimens in simulated Gas cooled Fast Reactor (GFR) environment, already quoted. Figure WP2.10 show some selected results.

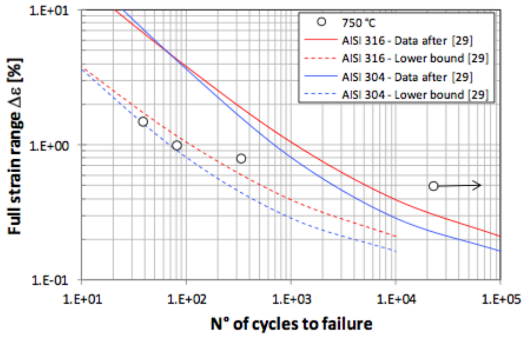


Figure WP2.10: LCF tests performed in impure He and compared to austenitic steels (Courtesy A. Gessi, ENEA).

Based on the results of such tests, the following main observations can be drawn:

- The general behavior of the 14 Cr-ODS steel during HT-LCF tests conducted in simulated GFR environment does not show significant differences as compared to other rather similar materials;
- The increase in test temperature from RT to 750 °C appeared to produce a fatigue life reduction by a factor as large as 25.
- the HT-LCF data for 14 Cr- ODS steel tested in simulated GFR environment appear to lay near the lower bound of the typical scatter-band observed for 304 and 316 SS, when tested in air at the same temperature;
- this could be an indication of an actual lower fatigue strength of 14 Cr-ODS steel as compared to 304 and 316 SS in HT-LCF regime, or of a detrimental effect of simulated GFR test environment as compared to air or both. It is however worth noting that the number of

tests which were conducted is by far too low for above observations to be regarded as conclusive and/or soundly based.

Finally Slow strain rate tests have been performed on the ODS alloys in SCW. The results are reported in figure WP2.11.

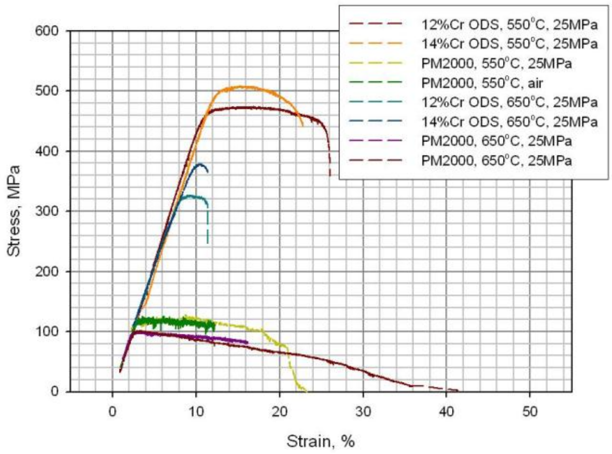


Figure WP2.11: SSRT curves of ODS at different temperatures (Courtesy VTT).

The results indicate that the ODS steels are less susceptible to SCC than austenitic stainless steels or Ni-base alloys. However, ODS steels have been tested very little in SCW and more data is required in order to make any final conclusions.

WP3 Irradiation behaviour of structural material

The objective of WP3 was to enlarge the database concerning the effect of irradiation on the relevant properties (i.e. needed for the design, such as dimensional and mechanical properties) of materials selected for core/target components of future GENIV reactors and ADS. The materials of interest were primarily FM steels, in particular 9Cr steels such as T91 (9Cr1MoVNb) and EM10 (9Cr1Mo) which are reference materials for different nuclear applications, however data on Fe-Cr based ODS alloys and austenitic steels were obtained as well. These data were generated as part of Post Irradiation Experiments (PIE) performed after irradiation campaigns conducted in various radiation environments, in some cases with the investigated materials irradiated in contact with aggressive coolants. This work package was organised in six different tasks, each task being devoted to the PIE of a particular irradiation experiment (with the noteworthy exception of task 3.5 which included both the design and implementation of a new irradiation campaign called LEXURII, followed by the corresponding PIE). The main results obtained within WP3 are presented hereafter and were discussed according to the following topics:

- Behaviour of 9Cr martensitic steels, in terms of dimensional stability and evolution of mechanical properties, after high dose irradiation in a fission environment: this issue is of particular interest for the core components of future GENIV reactors such as the hexagonal duct (wrapper tube) of the fuel assembly. As part of WP3, data up to a maximum dose of 155 dpa were obtained (Boitix9 irradiation in Phénix reactor).
- Effect of irradiation in a spallation environment on the mechanical properties of martensitic steels and ODS alloys: core and target components of ADS will be irradiated with high energy protons and spallation neutrons. In such environment, the combined effects of displacement damage and transmutation-induced impurities, in particular large quantities of

helium, are of special concern. Via mechanical tests and microstructural investigation performed after irradiation in the SINQ target, the behaviours of FM steels and ODS alloys were assessed following irradiation in a spallation environment to a maximum dose of about 20 dpa and an accumulated helium content of about 1800 appm.

- Combined effect of irradiation and contact with the coolant: possible synergistic effect of irradiation and embrittlement due to the coolant is a major issue, in particular for systems cooled by heavy liquid metals, and published results were so far extremely scarce. This topic was addressed via the PIE of IBIS (HFR reactor), ASTIR (BR2 reactor) and LEXURII (BOR60) which included tests performed in the liquid metal (liquid Pb-Bi or Pb) on specimens irradiated in contact with the coolant. Furthermore, the PIE of the MEGAPIE target provided unique data regarding the combined effect of irradiation in a spallation environment, in contact with flowing liquid Pb-Bi and with cyclic thermal/mechanical loading on the properties of T91 steel.

Effect of high dose irradiation in a fast spectrum on mechanical properties and dimensional stability of a 9Cr martensitic steel.

9Cr martensitic steels are known for the excellent radiation resistance (i.e. negligible void swelling, little hardening and moderate degradation of impact properties) when irradiated in a fast spectrum at temperatures above approximately 380°-400°C up to doses of at least 100 dpa. However significantly higher values for the end-of-life neutron exposure of in-core components such as the hexagonal duct, up to about 150 or higher, are envisaged in the case of future GEnIV reactors. Moreover, it has been argued that the excellent swelling resistance of FM steels is mainly due to a long incubation period (transient regime) and that FM steels should eventually swell at a steady state swelling rate which could be as high as 0.2%/dpa. Therefore, a comprehensive PIE programme was conducted using an hexagonal duct made of 9Cr-1Mo steel (EM10 grade) irradiated in Phénix reactor up to a maximum dose of 155 dpa (Boitix9 experiment). Tensile and impact specimens were machined at different locations along the duct, corresponding to irradiation temperatures ranging from about 385°C to 530°C and doses in the range 43-155 dpa. Mechanical tests confirmed the very good radiation resistance of EM10 steel for these specific irradiation conditions. As shown in Fig. WP3.1, irradiation induced hardening for all tested specimens, including after irradiation to 155 dpa, was found to be relatively small. Likewise, a moderate degradation of impact properties after irradiation was observed.

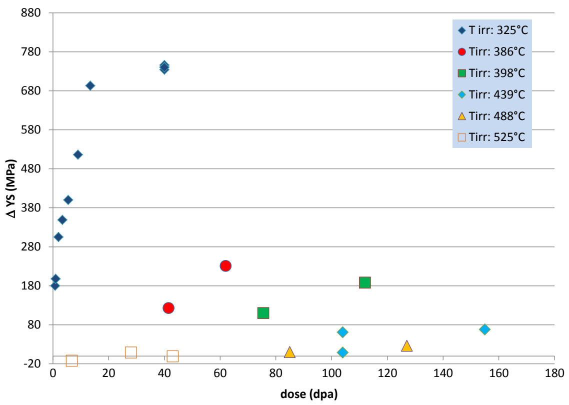
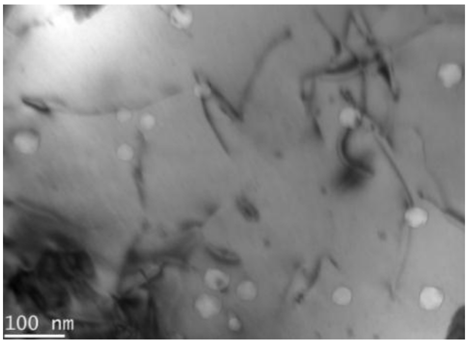
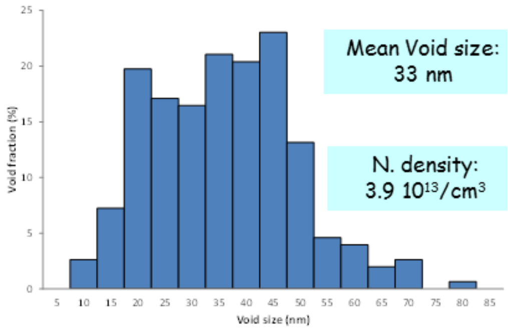


Figure WP3.1- Radiation-induced hardening as a function of dose for the same heat of 9Cr-1Mo (EM10) steel irradiated at different temperatures. Data obtained as part of WP3 (Boitix9 wrapper) are indicated by a circle (courtesy J. Henry, CEA).

As in other metallic alloys, the irradiation temperature has a drastic impact on the evolution of the mechanical properties. For instance at 325°C, in addition to formation of α' precipitates, irradiation induced the formation of a high density of small dislocation loops, possibly decorated with solute atoms, which are the main source of the high hardening observed after irradiation (see WP4, T4.2). In the temperature range of interest for the Boitix 9 wrapper, the dislocation microstructure was found by TEM investigations to be very different and consistent with the measured hardening: at 398°C (112 dpa), a low density of large dislocation loops was observed, while at 439°C (155 dpa), hardly any loop could be detected and the dislocation density was found to be similar to that measured prior irradiation.

In addition to the effect of irradiation on mechanical properties, dimensional stability is also of prime importance. Macroscopic density measurement showed that void swelling was very small. This conclusion was confirmed by TEM investigations of the void microstructures, as shown in Fig. WP3.2 in the case of the maximum irradiation dose. This low void volume fraction and the fact that voids were found to be heterogeneously distributed within the matrix are indications that EM10 steel was still in the incubation stage of void swelling even after irradiation to 155 dpa, which demonstrates the very high swelling resistance of this type of steel.





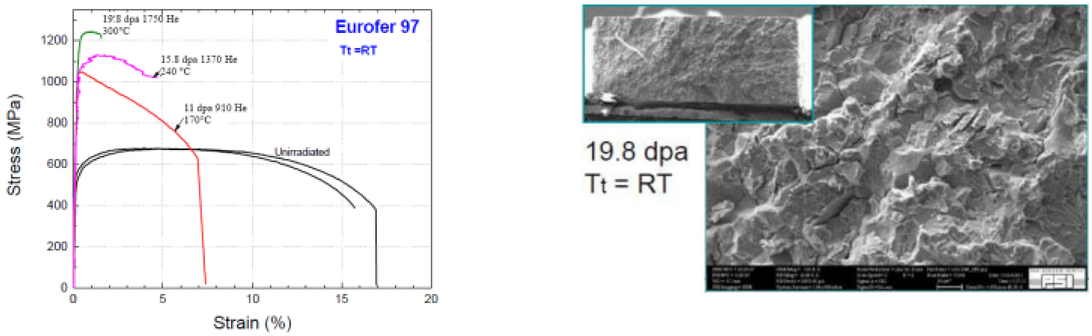
a) b)

Figure WP3.2- Void microstructure in 9Cr-1Mo (EM10) steel irradiated to 155 dpa at 439°C. a) TEM micrograph ; b) void size distribution. The void volume fraction was measured to be 0.1%. (courtesy J. Henry, CEA)

Effect of irradiation in a spallation environment on the mechanical properties of martensitic steels and ODS alloys

FM steels and ODS alloys are candidate materials for the structures of spallation targets and core components of ADS. In order to evaluate the evolution of mechanical properties in the relevant radiation environment, tensile and impact specimens of various FM steels and ODS alloys were inserted in the solid spallation target SINQ (PSI) and irradiated with high energy protons and spallation neutrons at temperatures in the range [150-350°C].

For all investigated FM steels, the same tensile behavior was observed. An example is shown in Fig. WP3.3 in the case of a 9Cr martensitic steel.



a) b)

Figure WP3.3- a) Engineering tensile curves for a 9Cr martensitic steel (Eurofer grade) irradiated in the SINQ spallation target. For each curve, the dose, accumulated He content (in appm) and average irradiation temperatures are indicated; b) SEM fracture surface examinations of the sample irradiated to 19.8 dpa, 1750 appm He (courtesy Y. Dai, PSI).

The specimens irradiated at the highest doses and helium contents displayed very high hardening and a fully brittle, predominantly intergranular fracture mode, as clearly demonstrated by the tensile curve and SEM fracture surface observations for the specimen irradiated to 19.8 dpa and 1750 appm He. This behavior, which is not observed for FM steels irradiated in fission conditions at the same temperature and to similar damage dose, is the result of i) matrix hardening due to the formation of small dislocation loops and a very high density of tiny helium bubbles (~ 3 10^{16} cm^{-3} according to TEM measurements) and ii) strong decrease of grain boundary cohesion due to the presence of helium (as shown by ab initio calculations). Likewise, measurements of impact properties after irradiation of FM steels in the SINQ target show a degree of embrittlement significantly greater than that expected after irradiation in fission conditions.

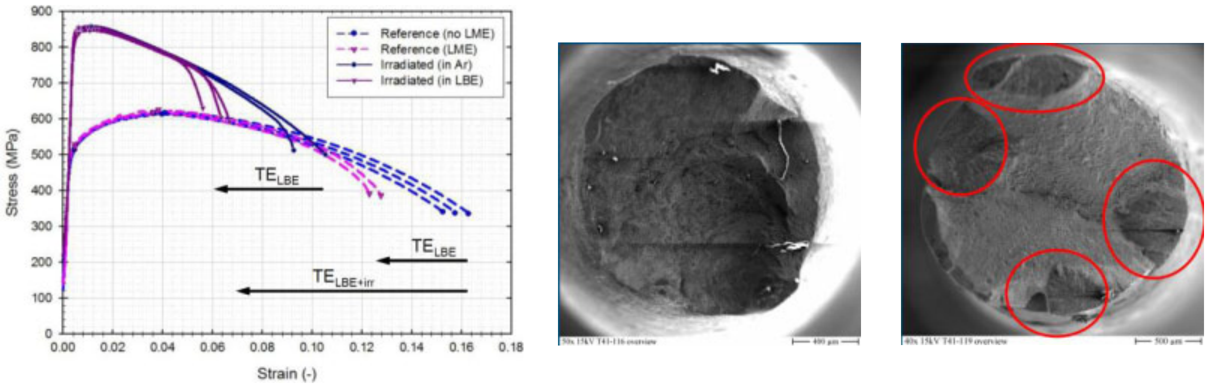
By contrast, ODS alloys displayed a better mechanical behavior after irradiation in spallation conditions. For example, 14Cr ODS (MA957 grade) irradiated in SINQ retained significant ductility when tensile tested at room temperature, whereas, as mentioned above, FM steels subjected to identical irradiation conditions exhibited a brittle intergranular fracture mode. The high density of nanoclusters in ODS alloys are believed to act as effective sinks for point defects as well as trapping sites for helium, thereby decreasing the irradiation induced hardening and protecting grain boundaries from helium embrittlement effect.

Combined effects of Heavy Liquid Metals (HLM) and irradiation

As several advanced nuclear systems will use a HLM (liquid Lead or Lead Bismuth Eutectic) as coolant, the issue of possible combined effects due to irradiation and HLM is of great importance and has little been addressed in the past. This issue was therefore the focus of several tasks of WP3, in particular via the PIE of ASTIR, LEXURII and MEGAPIE. As part of ASTIR experiment, the behavior of T91 steel in lead Bismuth Eutectic (LBE) was investigated. Liquid Metal Embrittlement (LME) was observed in the case of T91 in the as-received condition. For instance, when tensile tested in LBE, the total elongation was reduced as compared to a test in an inert atmosphere (i.e. fracture occured earlier), while the shapes of the tensile curves in both environments up to the rupture strain were very similar (see Fig. WP3.4). However, the necessary condition for LME occurrence is an intimate contact between the steel and the liquid metal. This was achieved in the as-received condition for instance by exposing the samples before tensile testing to LBE with low oxygen concentration, which resulted in a partial dissolution of the protective oxide layer typically found on the steel surface.

The effects of test temperature and strain rate on LME were studied in the case of unirradiated specimens and it was found that the reduction of the total elongation has a non-monotonic dependence on both parameters: LME occurs only in a particular range of temperature and strain rate. For a strain rate of about 5 10^{-5} s^{-1}, the minimum of the ductility trough occurs at about 350°C.

In order to investigate the effect of irradiation on the LME sensitivity of T91, tensile tests were carried out both in inert atmosphere (Ar) and in LBE on T91 specimens irradiated to 2.6 dpa as part of ASTIR irradiation experiments. The tests were performed at 300°C, which was the average irradiation temperature.



a) b) c)

Figure WP3.4- a) Engineering stress-strain curves for T91 in as-received and irradiated (2.6 dpa at 300°C) conditions, tensile tested in Argon and in LBE at 300°C. b) and c): SEM fracture surface examinations of irradiated samples tested a) in Ar, showing a fully ductile, dimpled type fracture surface, b) in LBE, showing several brittle areas corresponding to the brittle propagation of cracks initiated at the specimen surface (courtesy S. Gavrilov, SCK-CEN).

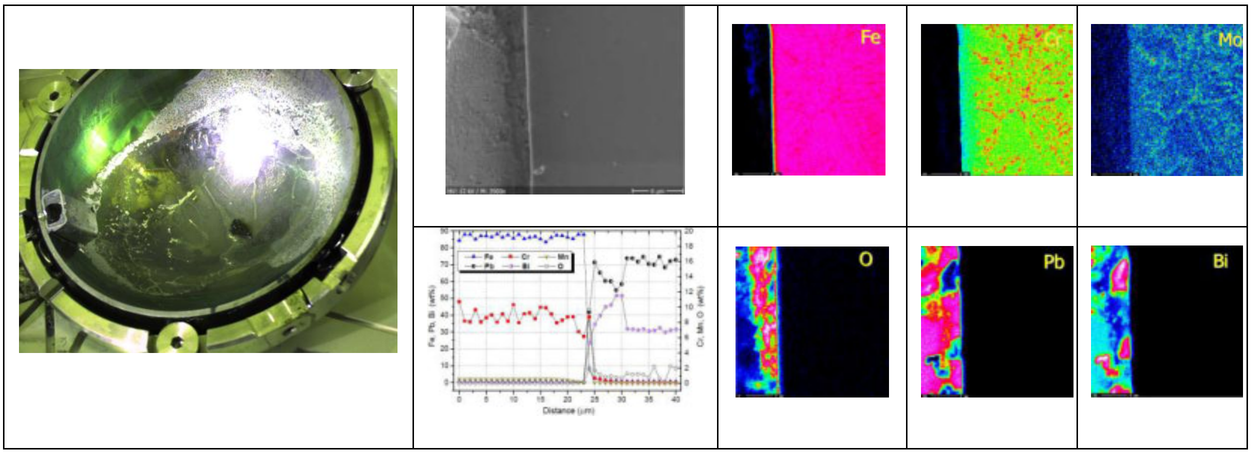
As shown in Fig. WP3.4, a strong LME effect was observed for irradiated specimens tested in LBE. When tested in LBE, multiple cracks initiation occurred at the surface followed by brittle propagation (see Fig. WP3.4b). The average reduction of total elongation due to LME (about 63% reduction compared to tests in Ar atmosphere) was proportionally larger than the average reduction measured in the case of unirradiated samples. Furthermore, ASTIR specimens had not been irradiated in contact with LBE. While for unirradiated specimens, pre-exposure to the liquid metal was found to be a necessary condition of LME occurrence, this was not the case for irradiated specimens. Thus irradiation was found to enhance the LME sensitivity of T91. This conclusion was also supported by the results of PIE performed on T91 specimens irradiated in contact with LBE to about 6 dpa at 350°C (LEXUR II experiment). Also in this case, a drastic reduction of total elongation was observed for irradiated specimens tested in LBE at 350°C compared to values measured in inert atmosphere.

By contrast, for all investigated testing conditions, including tests on specimens irradiated to 2.6 and 6 dpa (as part of ASTIR and LEXURII experiments), austenitic stainless steels (316L and 15-15 Ti grades) did not show any evidence of LME due to LBE.

In addition to tests performed on irradiated specimens, WP3 included as well the PIE of the MEGAPIE target, which was a unique opportunity to investigate the behavior of a real component whose structural materials had been irradiated in contact with flowing LBE and subjected to cyclic thermal/mechanical loadings. It must be emphasized that target dismantling using a large dedicated bandsaw, LME out-melting with a specially designed oven, raw cutting of the PIE structural material samples, cleaning of the raw cut samples (in order to remove alpha contamination), fine-cutting via EDM of a large number of samples which were then packed and sent to partner laboratories, represented a huge amount of work successfully carried out by PSI in its hot laboratories.

Prior to target dismantling, gamma-mapping of target tip was performed and the results were used for evaluating the proton fluence distribution and irradiation doses on the various target components. In addition, gamma analysis of LBE samples taken from different positions in the target showed good agreement between experimentally determined and calculated activities, which indicates that predictions by nuclear codes can be reliably used for safety assessments.

As target dismantling and specimen cutting took much longer than originally planned, only a limited PIE programme could be carried out in the timeframe of the GETMAT project. However, interesting results were obtained. Tensile tests (in air) were performed on specimens cut from the hemispherical beam entrance window made of T91, which was the most irradiated component (the maximum dose was calculated to be about 6 dpa). As expected, significant hardening was measured, however the total elongation of the sample with highest dose was found to be still above 10%, indicating a fully ductile behaviour. These results were in line with available data for T91 irradiated in spallation conditions in the same temperature range (close to 300°C).

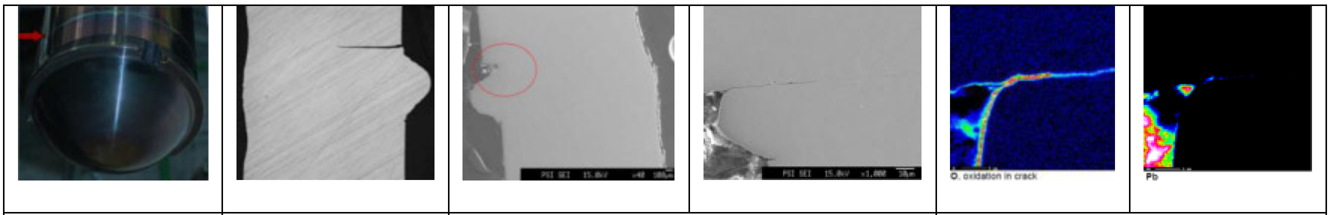


a) b)

Figure WP3.5- a) Inner surface of the T91 beam entrance window after LBE outmelting. b) cross section of a specimen cut from the beam window and Electron Probe Microanalysis (EPMA) mapping (courtesy Y. Dai, PSI).

No severe LBE corrosion nor erosion phenomena were observed on the T91 and 316L components, as shown for instance in Fig. WP3.5 in the case of a T91 specimen extracted from the beam window. These findings are consistent with the results of the investigations performed on coupons irradiated in contact with stagnant LBE (LEXURII PIE) which did not reveal any corrosion damage.

However, SEM and EPMA investigations performed by PSI and CEA have revealed the presence of a deep crack in the Lower liquid Metal Container made of T91 steel, located close to the Electron Beam (EB) weld line (Fig. WP3.6). Such a deep crack was not present before irradiation, but was perhaps nucleated during welding. Cyclic mechanical loading (due to proton beam trips) may have induced rapid crack propagation, since it has been shown that fatigue crack propagation is accelerated in LBE as compared to crack growth rates in inert environment.



a)	b)	c)	d)

Figure WP3.6- a) Lower Liquid Metal container (before irradiation) with location of EB weld line indicated by a red arrow. b) Optical Microscopy micrograph showing a deep crack (on the inner surface of the container) close to the weld line. c) SEM micrographs (different sample) and d) EPMA chemical maps (courtesy Y. Dai, PSI and J. Henry, CEA).

WP4 - Multiscale modelling and model experiments on irradiated Fe and Fe-Cr alloys

The objective of the WP was to develop physical models to describe the behaviour of iron-chromium alloys when subjected to thermal ageing and irradiation. These alloys were chosen as model materials for high-chromium ferritic/martensitic steels. The processes of interest were nanostructural and microchemical changes that occur under irradiation or ageing and the corresponding changes in mechanical properties, mainly hardening, which in turn causes embrittlement. The approach used was computer-based multiscale modelling (from ab initio to dislocation dynamics), integrated by the performance of specific modelling-oriented experiments. The latter provided detailed data on which models can be contrasted. They also suggested, in combination with fundamental atomistic calculations, physical mechanisms that the nanostructure and microchemical models, as well as mechanical models, needed to incorporate. The link with other work-packages is ensured first of all by the comprehension of the mechanisms that govern the changes induced by irradiation in iron-alloys with varying content of chromium, which allows experimental results on steels performed in the rest of the project, especially WP3, to be better rationalised. Moreoever, the characterisation of technological properties, such as fracture toughness, in model alloys was included in some tasks.

The ultimate objective of the WP was to understand which nanostructural features are the main cause of radiation- hardening and embrittlement and to develop a physical model capable of rationalising the experimental data available on this issue. This goal was indeed reached.

The work-package was **organised in three main tasks**, the highlights of which are given in what follows.

Modelling of fundamental properties of Fe and Fe-Cr alloys

The main objective here was the development and application of advanced cohesive models for Fe-Cr alloys, capable of describing their thermodynamic properties and the properties of point-defects (vacancies, self-interstitials), as well as extended-defects (dislocations, grain boundaries), possibly including magnetic properties. The development of these cohesive models, then applied for atomistic calculations, required extensive density functional theory (DFT), or first principle (ab initio), calculations. The Highlights of the task are:

- First principles calculations have provided new insights into several areas of interest for the basic properties of ferritic alloys; specifically, elements commonly found in nuclear steels, not only Cr but also other solutes in Fe-Cr, have been studied. The main findings of these calculations can be so summarized:

- The magnetic coupling has been extensively studied in Fe-Cr, revealing how magnetism influences some of the properties of the alloy, especially the mixing enthalpy, determining the change of its sign from negative to positive in the low Cr region, thereby leading to a modified phase diagram as compared to the standard one. These effects, however, are suitable to be described also by classical potentials that do not explicitly include spins. The strong vibrational entropy of Fe-Cr has been verified: it plays an instrumental role for the shape of the Fe-Cr phase diagram, by lowering the temperature at which the misicibility gap closes. Non-configurational entropy should therefore be included in models that describe thermodynamics from an atomistic standpoint.
- For the grain boundary and surface segregation of Cr in Fe, new insights have been gained, namely, Cr appears to have a tendency to segregate at grain boundaries, while it would tend to stay away from free surfaces, especially voids.
- The study of the interaction with point defects with all possible solute atoms in iron revealed that the elements that are found experimentally to form aggregates and to segregate at dislocations or grain boundaries (namely Cr, but also Ni, Mn, Si, P, and Cu) are found to migrate together with point defects, both vacancies and, in a few cases, interstitials. This proves to be a very effective physical mechanism to explain the experimental evidence.

- The development and validation of the interatomic potentials has rendered a new, improved interatomic model for Fe-Cr in very good agreement with experiments and first principles calculations (see above). This potential has been validated and put to use in dislocation studies, defect diffusion, solute-defect interactions, etc. In particular, Metropolis Monte Carlo studies revealed how Cr is expected to re-distribute at steady-state under thermal ageing and, as a tendency, under irradiation. Fig. WP4.1 illustrates graphically the results of these studies.

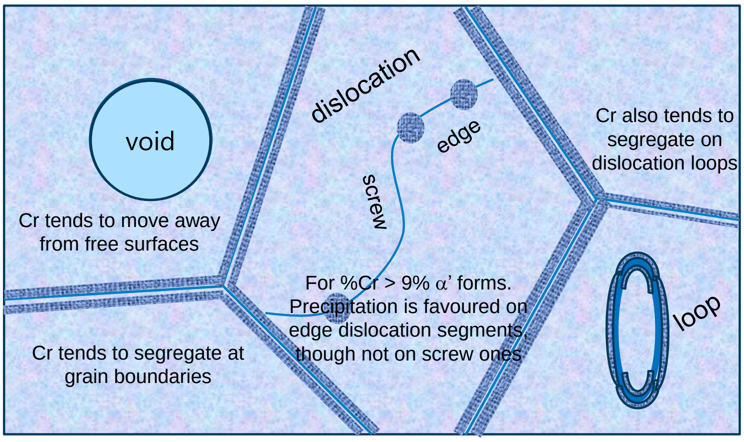


Figure WP4.1 – Graphical summary of how Cr atoms tend to redistribute under ageing at steady-state due to thermodynamics driving forces. Results are broadly in agreement with experiments (courtesy L. Malerba, SCK-CEN).

Modelling of radiation effects in Fe and Fe-Cr alloys

The main objective here was the development of microchemical and nanostructure evolution models, as well as (radiation) hardening models, for Fe-Cr alloys subjected to thermal ageing and irradiation, using as input the results of applying *ab initio* and/or empirical cohesive models from the task described before. The former category includes several types of kinetic Monte Carlo (KMC) models, each of them with its advantages and limitations, as well as cluster dynamics (or rate theory) models.

The latter consistes basically of the coupling of molecular dynamics (MD) with dislocation dynamics (DD): it is the first time that this coupling is successfully applied to rationalise quantitatively the results of mechanical tests, starting from fully physical considerations. The Highlights of the task are:

- Atomistic KMC (AKMC) techniques have reached maturity as methodology for the simulation of thermal ageing processes (phase separation, segregation, …) driven by diffusion of solute atoms, although some specific difficulties still exist with the diffusion of interstitial species like carbon. The importance of including (semi-empirically) magnetic and vibrational effects emerges from both DFT calculations and experimental studies, and should be considered as one of the main improvements to be introduced in the technique in the long run.
- For AKMC simulations of irradiation processes, important progresses have been made to include multiple vacancies and single-interstitials: the effect of Cr on point defect diffusion properties is explicitly accounted for and simulations of resistivity recovery experiments including effect of Cr and C have been successfully performed. Also the calculations of the L_{ij} phenomenological coefficients (Onsager matrix), for use in radiation-induced segregation models, has been performed and processes such as phase separation and segregation can be now successfully simulated starting from fully physical considerations (see example of AKMC results in Fig. WP4.2). However, the modelling of SIA clusters in an AKMC framework remains challenging. More generally, the correct inclusion in the model of extended defects that imply an intense strain field, such as dislocations and grain boundaries, remains challenging.
- Object KMC (OKMC) and Cluster Dynamics (or Rate Theory) models have been developed and a direct comparison with experiments has been made to validate them. The key difficulty is the determination of parameters for concentrated alloys. A new method, cellular OKMC, has been developed, with promising results. As an alternative, a "grey-alloy" model in which the presence of Cr and C atoms on the nanostructural evolution under irradiation is taken into account by modifying defect stability and mobility parameters, without explicitly introducing solute atoms, has been also developed. The latter allowed good agreement with experimental trends to be obtained, but cannot model precipitation (see results in Fig. WP4.3). The former, on the other hand, allows precipitation to be described and in the long run should be able to treat also the other defects, in a similar way to the "grey-alloy" model, providing also more local information, but for the moment these features are not included in the code. The rate theory models that have been developed clearly required specific fitting to experimental data

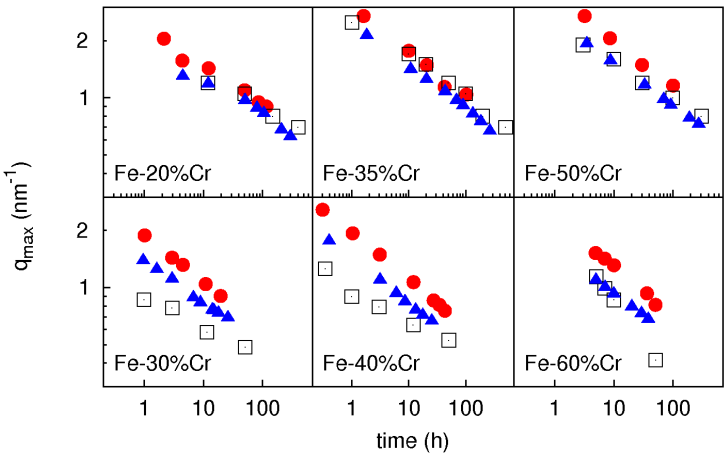


Figure WP4.2 – Direct comparison between SANS experiments (empty squares, □) and AKMC simulations of thermal ageing and phase separation in Fe-Cr alloys, with (blue triangles ▲) and without (red circles ●) magnetic correction (courtesy F. Soisson, CEA).

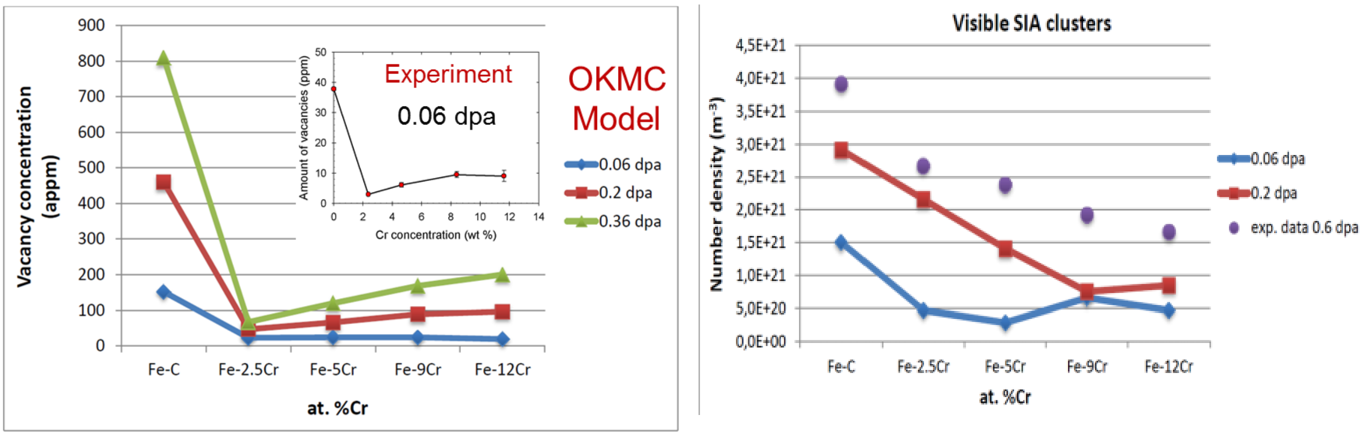


Figure WP4.3 – Comparison between PAS (left) and TEM (right) experiments on Fe-Cr alloys neutron irradiated at 300°C and the results of the "grey alloy" OKMC model: computing time issues did not allow 0.6 dpa to be reached, but agreement on trends is good (courtesy M. Chiapetto).

- Extensive MD simulations have been performed in order to assess the friction stress due to solution strengthening in Fe-Cr alloys of different concentrations, as well as to evaluate the critical stress for unpinning of both edge and screw dislocations at defects, including Cr-decorated loops and Cr-rich precipitates, which are assumed to be the main cause of irradiation hardening in Fe-Cr alloys. The studies have been performed for different defect and box sizes, different temperatures and also different strain rates. They have clearly shown that dislocation loops decorated by Cr atoms (and/or other solute atoms), as a consequence of segregation, are stronger obstacles than undecorated loops and are, therefore, candidates to be the main origin of radiation hardening in these (and other) Fe alloys (see Fig. WP4.4). A method to extract from MD data the parameters for DD simulations has been developed: this is based on the deduction of the 'friction stress inside the defect', which depends only on the type of defect, as well as the activation energy of unpinning.

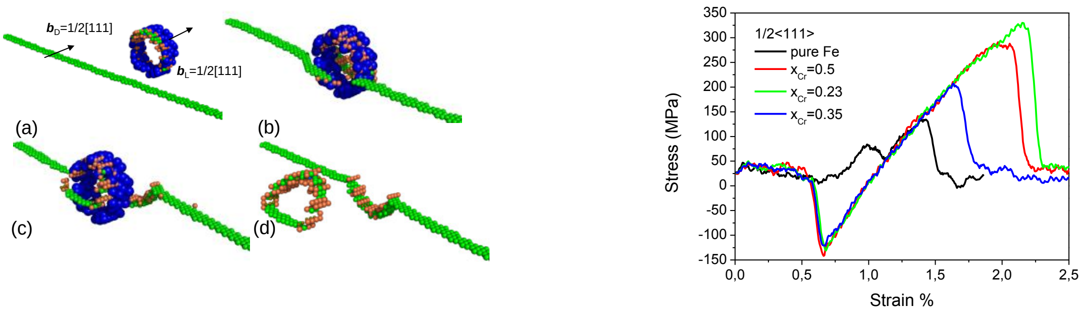
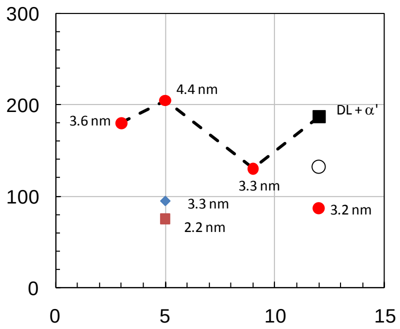


Fig. WP4.4 – Left: Mechanism of interaction of an edge dislocation with a Cr enriched loop as simulated by molecular dynamics; Right: Effect of Cr enrichment on stress-strain curve during interaction of Cr-decorated loop with dislocation (courtesy D. Terentyev, SCK-CEN).

- The MD results of strength of irradiation defects (loops, Cr decorated loops, Cr precipitates, …) have been incorporated into a DD model, allowing simulations of radiation strengthening in Fe-Cr alloys as a function of Cr content: it is the first time that a fully physical model manages to account in a quantitative way for the radiation-hardening measured experimentally (see Fig.

WP4.5). Key is the recognition that screw dislocations that interact with loops having collinear Burgers vector may form helical turns, thereby producing hardening effects 10 time larger than the Orowan mechanism. The main sources of hardening are the Cr-NiSiP clusters, if assumed to be small loops with solute atoms segregated on them.



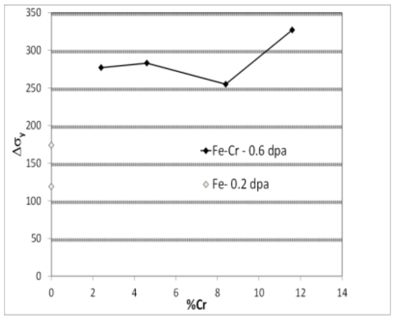


Fig. WP4.5 – Comparison of radiation hardening as predicted by the physical DD model and as measured – no fitting or calibration has been performed (courtesy G. Monnet, EDF).

Modelling-oriented experiments in Fe and FeCr alloys

The main objective here was to perform modelling-oriented experiments, in which selected reference materials (Fe-Cr model alloys) were characterised after neutron and ion irradiation, from the nanostructural, microchemical and mechanical point of view, using a combination of experimental techniques. Highlights of task:

Two main sets of experimental results have been obtained within this task:

1. Characterization of neutron irradiated Fe-Cr alloys
2. Characterization of ion irradiated Fe-Cr alloys

In both cases the goal was to combine different experimental characterization techniques in order to get a complete picture of the nanostructural and microchemical changes occurred in the alloys as functions of Cr content and irradiation dose, as well as, partly, temperature. The materials considered were alloys containing nominally 2.5, 5, 9 and 12 %Cr.

<u>Neutron irradiated Fe-Cr alloys (0.06, 0.6 and 1.5 dpa, at 300°C)</u>

Positron Annihilation Spectroscopy (PAS)

Mainly single vacancies are present in FeCr alloys compared to pure Fe: very little vacancy clustering occurs when Cr is present (data for 0.06 dpa).

Higher amount of vacancies is observed at higher dose, but nonetheless the presence of Cr reduces the amount of vacancies drastically.

Small Angle Neutron Scattering (SANS)

The irradiation-induced clusters detected are Cr-rich α'-phase particles for Fe-12%Cr, while in the other alloys they must contain impurity elements, such as C, Ni, P or Si.

There is a pronounced fluence dependence of the concentration and size of α'-phase particles below 0.6 dpa and a saturation-like behaviour beyond.

For lower Cr levels, pronounced fluence dependence below 0.6 dpa was also found

Atom Probe Tomography (APT)

α' clusters were only present in the supersaturated alloys (Fe-9%Cr and Fe-12%Cr)

CrSiPNi enriched clusters were observed in all alloys with the same number density: they are suspected to be small loops on which solute atoms segregated (Fig. WP4.6).

Segregation of Si, P and Cr atoms on dislocation lines and grain boundaries was observed.

Zones free of clusters were found around the GBs, suggesting that solute segregation should be the mechanism leading to the formation of the CrSiPNi clusters: they would not form where other sinks attract the solute atoms.

Transmission Electron Microscopy (TEM)

Dislocation loops are inhomogeneously distributed for 5, 9 and 12 Cr with dislocation loops preferentially located close to grain boundaries and dislocation lines; they are homogeneously distributed for 2.5%Cr: there is an effect of Cr content on the distribution of dislocation loops. This effect might be indirect: it is known that 2.5 %Cr alloy is ferritic and has C in the matrix, while the other alloys are ferritic/martensitic and C has segregated at grain boundaries. C in the matrix would act as trap for dislocation loops that, otherwise, would be free to migrate to sinks (dislocations and grain boundaries).

<u>Ion irradiated Fe-Cr alloys (to 1 and 5 dpa, at 100, 300 and 420 °C)</u>

PAS (Coincidence Doppler Broadening and Positron Annihilation Lifetime Spectroscopy):

Vacancy-type defects were produced by the ion irradiation. The amount and size decrease when %Cr increases and also, when the irradiation T increases. Larger voids and more vacancies were observed at the lower dose.

X-ray Magnetic Circular Dichroism (XMCD):

Ion irradiation produces a redistribution of Cr atoms, deduced from the magnetic moment, which can be an indication of Cr clustering also observed by APT.

APT:

NiSiPCr rich clusters where observed after ion irradiation (Cr is the main element)

No α'precipitates are observed in this case, differently from neutron irradiation.

Extended X-Ray Absorption Fine Structure and X-Ray Diffraction (EXAFS):

This technique indicates the presence of vacancy and interstitial type defects.

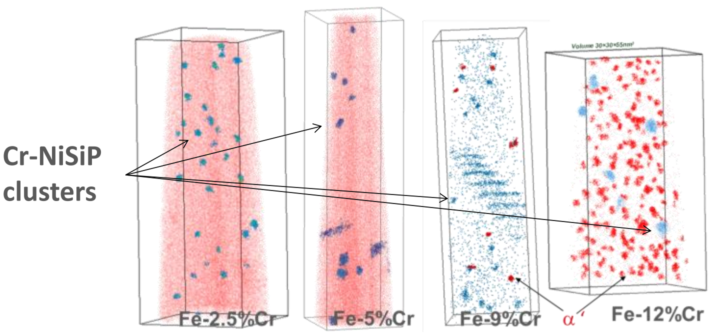
TEM:

Homogeneous distributions of interstitial dislocation loops are found at all conditions.

Indications of denuded zones close to grain boundaries in Fe12Cr.

At 1 dpa: there is no significant effect of Cr content (from 9 to 12) on size or density of defects. Differences are expected in the proportion of ½⟨111⟩ loops to ⟨100⟩ loops.

At 5 dpa: higher density in 9Cr, while larger size in 12Cr.



Alloy	α' clusters (nm)	α' clusters N_v (m^{-3})	NiSiPCr-enriched clusters (nm)	NiSiPCr-enriched clusters N_v (m^{-3})
Fe-2.5%Cr	-	-	1.8 ± 0.2	(1.3 ± 0.5)·10^{23}
Fe-5%Cr	-	-	2.2 ± 0.2	(1.3 ± 0.5)·10^{23}
Fe-9%Cr	1.1 ± 0.2	(2.1 ± 0.2)·10^{23}	1.65 ± 0.2	(2.4 ± 0.5)10^{23}
Fe-12%Cr	1.1 ± 0.2	(5.0 ± 0.5)·10^{24}	1.6 ± 0.2	(1.1 ± 0.7)·10^{23}

Figure WP4.6: Atom Probe Tomography (APT) on neutron irradiated specimens

4.1.4 Potential impact, exploitation of results and dissemination

It was foreseen that the potential impact of the GETMAT is to advance in the area of materials technology for transmutation and Generation IV reactors. Priority R&D topics were identified and accordingly success indicators defined. Moreover, it was stated that GETMAT would also impact the share of activities among different EU countries and laboratories accounting for their specific area of excellence and by optimising the utilisation of relevant installations.
The potential impact of GETMAT results, as declared at the beginning of the project (and possibly also beyond) is hereafter discussed by evaluating the success indicators as defined in the description of work.

For what concerns the exploitation of results, the project evolution and final outcome can be considered as an essential step towards the emplacement of the Joint Program for Nuclear Materials (JPNM), within the framework of the European Energy Research Alliance. For instance, GETMAT results have been essential in identifying research themes for the JPNM which were translated in Sub-programmes and Pilot Projects.
Moreover, GETMAT has invited a number of external partners to participate to the user group. The user group was composed by steel maker industries such as OCAS, and CENTROINOX, the nuclear industry as AREVA and ANSALDO, by research associations as IFE (Norway) and Non-European R&D labs as LANL (USA).
In the section “exploitation of results” a detailed analysis on how the user group has appreciated the GETMAT results will be described.

Finally, GETMAT has done an important work on dissemination of results as well as on education and training. Both aspects were part of the success indicators and will be described in this section. Moreover, in the section 4.3 “Use and dissemination of foreground” further details will be given.

4.1.4.1 List of success indicators (KPIs)

In order to assess the potential impact of the GETMAT project an analysis of the “Success Indicators” defined at the beginning of the project is hereafter done. The “Success Indicators”, have been identified with the objectives to measure through specific milestones and deliverables the achievement of the declared objectives. The impact of these achievements is then discussed.
Due to technical problems that have been encountered during the project life and that have been solved by e.g. re-addressing activities, it was necessary to adapt the “Success Indicators” accordingly to the updated activities. The identified “Success Indicators” can also be understood as Key Performance Indicators (KPIs) since the “KPIs are commonly used to evaluate the success of e.g. an organization / program / project or the success of a particular activity”.
The list of KPIs (or Success Indicators) for the GETMAT Project, as reported in the description of work, can be classified within two categories. The first category is related to the scientific advancement, while the second category is related to the project output in terms of dissemination and education and training activities.

Hereafter the list of KPIs is given:

<u>GETMAT KPIs (or “Success Indicators”)</u> <u>category I</u>: scientific advancement

1. Distribution to the laboratories of the ODS alloys manufactured in Europe and Japan. Since the fabrication of the ODS alloys is considered a challenging task, due to the amount of material to be produced, the achievement of this milestone will be a successful accomplishment and will give the opportunity to the laboratories to perform the planned experiment within a not too tight schedule.

2. Demonstration of the high temperature performance of the three ODS alloys and their weldability. The accomplishment of this demonstration will be an important task for the definition of the future R&D needs within the area of ODS alloys development for nuclear applications. In addition the compatibility tests of the ODS alloys with different coolants, will complete this framework.
3. The full manufacturing process of "smart coatings" for fuel claddings with optimised chemical composition and standardised qualification process will be available and can be therefore considered in the future consideration concerning materials selection related to the issue of alternative corrosion protection method.
4. The results of the PIE of the samples irradiated in the BOITIX9 and SUPERNOVA experiments (Phénix), MEGAPIE/SINQ, ASTIR (BR2), IBIS/SUMO (HFR) and BOR60 will be collected in several deliverables. A comprehensive representation of these data and the ability to transmit the performance assessment of irradiated materials in presence of coolant to the designers will be an important success indicator.
5. The successful completion of the model parameterisation phase and the timely start of a second phase of model development based on the use of available experimental results will be also used to measure the performance of the project on the modelling side.

GETMAT KPIs (or success indicators) category II: dissemination and education & training
1. Organisation of Workshop / Training Courses / Schools
2. Number of PhDs and Post-Doc students: The reference in the description of work was is 5% of the total budget of the project. This corresponds to a number of PhD/Post-Doc students in the order of 6-10
3. Number and type of publications

4.1.4.2 Discussion and assessment of the KPIs (or Success Indicators) and their potential impact

GETMAT KPIs (or "Success Indicators") category I: scientific advancement

1. Distribution to the laboratories of the ODS alloys manufactured in Europe and Japan.

Achievement: Within the GETMAT project ~ 65 kg of ODS were produced and distributed to the GETMAT partners as agreed in the description of work:

- ~ 30 kg 14 Cr ODS
- ~ 20 kg 12 Cr ODS
- ~15 kg 9 Cr ODS

- The distribution of ODS to the laboratories has been achieved with some delay
  - ODS manufacturing at large scale in Europe had technical problems at KIT and SCK-CEN
  - GETMAT management achieved to get ODS from Japan

Potential impact:
- It has been seen that the large scale production of ODS steel needs further development in Europe.

- All GETMAT partners were working on the same batch of materials, therefore results were comparable. Moreover, the ODS has been distributed such as to optimise the use of facilities and installations in EU.

2. Demonstration of the high temperature performance of the three ODS alloys and their weldability and compatibility tests of the ODS alloys with different coolants

Achievement:

- Tests at high temperature have been performed up to 750°C
    - first mapping of ODS performance has been achieved
    - consistency of the mechanical test results coming from different project partners has been obtained, that is an indication of the expertise of the participants
- Investigated welding techniques
    - Diffusion Bonding, Friction Stir Welding and Explosive Welding can be applied on ODS. However, careful identification of welding/joining parameters is a prerequisite
- Compatibility with coolants checked
    - Dependencies from coolant chemistry and temperature have been identified. Operational window of the ODS steels could be seen, where as expected the 14Cr ODS showed the highest resistance to oxidation.

Potential Impact:

- Results obtained have made evident the necessity to deepen the microstructural behaviour of ODS when subject to high stresses and to correlate fracture mechanism with microstructure. This will be done within JPNM and related projects.
- It has been suggested that the GETMAT-ODS might be suitable for clad material of advanced nuclear reactors. The first results are promising, however it is necessary to continue R&D to address anisotropic effects and assess ODS performance in relevant neutron irradiation fields. Moreover, the performances of GETMAT-ODS were not so satisfactory to recommend this material for structural components of reactor systems.

3. The full manufacturing process of "smart coatings" for fuel claddings with optimised chemical composition and standardised qualification process

Achievement

- The thickness of the coating prior to GESA treatment must be < 30µm.
- The surface alloyed layer (after "GESA" treatment) has following required composition: >8wt% Al, between 9 and 14 wt% Cr and the balance is Fe.
- The GESA treatment parameters are fixed regarding electron energy (120keV), pulse duration (40 to 60 µs) and power density (about 2MW/cm²). More than 2 pulses are needed (one with full duration, the other with half duration) to homogenize the final surface
- Short term oxidation experiments in gas Atmosphere is suitable to assess the capability to form thin Al-rich oxide scales in liquid Pb alloys. However the required Al content in gas is at least 2wt% high compared to Pb.

Remaining open issues:

- Deposition process for the coating is still not hundred per cent satisfying. Selected deposition process results in too high oxygen content.
- Can rare earth elements further reduce the required Al content required for thin slowly growing Al-rich oxide formation?
- Process stability and repeatability using the needed GESA IV is still not optimal.

- Qualification process is not working to that extend as expected – an elemental analysis based process (e.g. large area EDX) should be considered and investigated

Potential Impact

- Corrosion protection barrier can be further developed for LFR systems
- Among the two combinations of techniques investigated, i.e. HVOF+Laser treatment and LPPS+GESA treatment, this second one seems to be more promising. However LPPS might be replaced with VPS.
- A qualification process of corrosion protection barrier for nuclear application needs to be defined.
- Relevant activities on corrosion protection barrier for Generation IV and transmutation systems have been included in JPNM and related projects.

4. The results of the PIE of the samples irradiated in the BOITIX9 and SUPERNOVA experiments (Phénix), MEGAPIE/SINQ, ASTIR (BR2), IBIS/SUMO (HFR) and BOR60.

Achievements

- PIE results have been generated on conventional steels as 9-12 F/M and austenitic steels in different environments.

Potential Impact

- The long-term use of 9-12 F/M steel in high temperature and neutron irradiation fields (i.e. for Generation IV and transmutation reactors) is restricted to a temperature range of 350-550°C due to irradiation embrittlement (in the low temperature range) and poor thermal-creep and stress-to-rupture resistance in the high temperature range
- Liquid metal (especially Pb-Bi eutectic and Pb) assisted degradation of 9-12 F/M steel limit the use of this materials for LFR and ADS. Similar degradations have not been observed on austenitic steels.
- In proton-neutron mixed spectrum the high He production rate per dpa might contribute to the hardening and embrittlement of 9-12 F/M steel. These data are relevant for the neutron spallation target development, which is an essential component of ADS systems

***5.** The successful completion of the model parameterisation phase and the timely start of a second phase of model development based on the use of available experimental results*

Achievements

- Fe-Cr phase diagram assessment and role of vibrational entropy
- Definition of an improved interatomic potential for Fe-Cr which has proven to be in very good agreement with experiments.
- Theoretical explanation and experimental verification of irradiation hardening and embrittlement in Fe-Cr alloys
- Identification of differences in the field of ion vs. neutron irradiations

Potential Impacts

- The solubility of Cr in Fe and formation of phases (as given in the phase diagram in the low Cr concentration range) needs to be revised.
- Explanation of irradiation hardening and embrittlement of Fe-Cr alloys can be extended to F/M steels. However, further steps in the theoretical approaches are still needed

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- The solubility of Cr in Fe and formation of phases (as given in the phase diagram in the low Cr concentration range) needs to be revised.
- Explanation of irradiation hardening and embrittlement of Fe-Cr alloys can be extended to F/M steels. However, further steps in the theoretical approaches are still needed
- Modelling activities relevant for Generation IV and transmutation systems have been included in JPNM and related projects.
- Even if ion irradiations are relatively easy experiments to be performed. It has been demonstrated that results from ion irradiation have to be handled carefully when used as surrogate of neutron irradiation.

Category II of GETMAT KPIs: dissemination

1. Organisation of Workshop / Training Courses / Schools

Within the GETMAT project it has been agreed to organize two Schools and two workshops. The scientific output of these training activities is here measured in terms of number of attendees at the Schools/Workshop, number of lectures and poster presented.
It should be here also noticed that all GETMAT training activities, but the final international workshop have been organized in cooperation with other EC funded projects and / or international organisations as e.g. IAEA, USA laboratories etc. This approach of organizing training sessions within GETMAT has allowed the improvement of those components of the project that are directly related to the European Research Area and Networking of Excellence.

The training activities organised are:

- First International School on MATerials for nuclear REactors (MATRE-1). 18-23 October 2009 - Auberge de la Ferme - Rochehaut-sur-Semois, Belgium. This school has been organised together with the EC supported PERFORM60 project
- First International Workshop on Dispersion Strengthened Steels for Advanced Nuclear Applications (DIANA-I). 4-8 April, 2011 – Centre CNRS Paul Langevin, Aussois, France. This Workshop has been co-sponsored by: IAEA, LANL (USA), LLNL (USA), ORNL (USA) , OCAS/Arcelor-Mittal (Belgium), SCK•CEN (Belgium) CEA (France), KIT (Germany) PSI (Switzerland) and JRC-IE (EU). Selected paper of the DIANA – I workshop have been published on Journal of Nuclear Materials.

- Joint GETMAT/MATTER Summer school - MUNECO: Materials UNder Extreme Conditions. This school has been organised together with the EC supported MATTER project
- Final International GETMAT Workshop

Name of Training Activitiy	No. Lectures	No. Posters	No. Attendees
MATRE - 1	18	24	62
DIANA – I	36	16	84
MUNECO	25	32	54
Final Int. Workshop	30	20	75

2. Number of PhDs and Post-Doc students: 30

3. Number and type of publications:
    - Conference / Workshop contributions: 4
    - Journal Papers: 68
    - Others (Newsletter etc.): 4

4.1.5.3 Exploitation of results: the GETMAT User Group

All participants to the GETMAT user Group were asked to fill in a questionnaire. The questions and the answers are hereafter summarised. It should be noted that not all members of the user group have answered.

- **Question 1: Could you exploit the results produced within GETMAT within your institution. Do you have / will have possibilities to disseminate the knowledge acquired within GETMAT?**

    ✓ Answer from steel industry: The GETMAT project definitively helped to guide and enrich the research done by the steel industry. The knowledge obtained within GETMAT is disseminated inside our organisation and towards strategic partners.

    ✓ Answer from nuclear industry: Unfortunately there was no possibility to access any document produced in GETMAT

    ✓ Answer from research organisations (EU and non-EU): The results produced within GETMAT were useful in the development of instruments for use in liquid metals and supercritical water. There are various possibilities to disseminate the knowledge acquired within GETMAT (User-Group lecture, Conferences, ..).

- **Question 2: Could you identify limits and potentialities of the investigated GETMAT materials, i.e. 9Cr – F/M steels and ODS steels?**

  - ✓ Answer from steel industry: Yes, both for 9Cr-F/M as well as ODS steels the knowledge has has been develop but the industry became also aware of important limitations, mainly for ODS steels.

  - ✓ Answer from nuclear industry: 9Cr – F/M is still used in both ELFR and ALFRED reactor cooled by Lead.
    - o The operating temperature has been selected above the Ductile to Brittle Transition threshold under irradiation.
    - o The lead velocity has been limited lower 2 m/s to limit erosion issue
    - o The oxygen controlled environment has been adopted to limit corrosion

    However the R&D should not be limited to search material suitable for fluent molten Lead but also the influence of the Lead chemistry shall be exploited. We believe that appropriate structural material in conjunction with Lead chemistry (i.e. corrosion inhibitors) have more potentialities to solve the material issues.

  - ✓ Answer from research organisations (EU and non-EU): ODS steels clearly have potential, but there is still a long way to go (improvement in fabrication routes, avoiding strong anisotropies, and the behaviour under irradiation is not sufficiently explored).

- **Question 3: Did you consider the interaction with the GETMAT community through the technical meetings as effective?**

  - ✓ Answer from steel industry: Yes, and there was also the possibility to extent the research network through the GETMAT project.

  - ✓ Answer from nuclear industry: Designers should be maintained informed about the GETMAT results not only with technical meeting but with appropriate documentation for implementing them in the design.

  - ✓ Answer from research organisations (EU and non-EU): Yes, the technical meetings were very well organized (and intense) and offered sufficient possibilities for discussions (during the meeting, as well as outside of the meeting).

- **Question 4: Do you have any suggestion on how User Groups can be implemented in EC supported R&D future projects on materials for nuclear applications?**

  - ✓ Answer from steel industry: Interaction is mainly related to the technical meetings. It could be beneficial to include maybe one or two additional workshops on sidely related themes like economic impact etc.

  - ✓ Answer from nuclear industry: Improve the two way communication with dedicated workshops and provide summary and full detailed results.

  - ✓ Answer from research organisations (EU and non-EU): (1) The case of our institute (IFE) is special because Norway cannot directly receive EC support (as Norway is not in EURATOM). However, we could (technically) contribute through instrumentation and materials testing under irradiation and we have unique capabilities for doing this. If such possibilities are recognized from the start in future projects, subcontracting could be considered. (2) I enjoyed my participation in this user group but it was difficult because of my location out of the US. If the user group has members outside of Europe, it may help participation if some meetings were held as side meetings in conjunction with other related international meetings as long as other members of the user group are also attending this meeting. Otherwise, I found this a very useful user group.

As can be seen from the collected answers, the relation with the User Group occurred only through the technical meetings. This might be the reason why the GETMAT results have been considered more of impact for the steel maker industry and the other research laboratories and less for the nuclear industry.
Moreover, as further suggestion to organise dedicated meetings with the user group to address topics not strictly related to the project might be taken into account for the future. Finally, all members of the user group could exploit the GETMAT results for further develop in their own field.

<u>Exloitation of results of WP 4</u>

The WP4 of GETMAT, besides determining important advances in the understanding of the fundamental mechanisms that drive nanostructural and microchemical evolution under irradiation in F/M alloys and are ultimately responsible for their radiation hardening and embrittlement, has strongly contributed to create a community of researchers in Europe now used to collaborating very closely and exchanging data. The results will be exploited within this community to advance further along a path that is by now largely consensual, and consolidated in the workplan of the JPNM of the EERA. The identification of segregation on extended defects of solute atoms transported by point-defects as key mechanism leading to the appearance of embrittling "phases" in F/M alloys has triggered the research now started in the framework of the MatISSE project, which aims at developing models for multicomponent alloys, having a path to follow in this direction. Importantly, this mechanism has been proven to be common to reactor pressure vessel steels used in current reactors, thereby providing a framework for further advances in that area, too. The success in using dislocation dynamics to rationalise on physical basis radiation hardening, moreover, sets a precedent that can be now followed for other systems. Also the methodological advances are important and will be usable worldwide. Finally, the experimental programme will remain an example to be followed in terms of correlated and complementary use of experimental techniques to fully characterise irradiated materials, pointing out also caveats when using ions to emulate neutron irradiation.

4.1.6 Project public website

The project website has been developed.

Internet address: http://nuklear-server.nuklear.kit.edu/getmat/index.php

The website is continuously up-dated both at the public part and at the member area part. Within the public part all presentations made during the training activities are accessible.

Moreover, the communication between beneficiaries has been always very constructive and good. This communication normally occurred through e-mail and phone calls. Moreover, technical meetings at WP and task levels have been organised on regular basis to discuss technical details of problems and outcomes.

The GETMAT project has also a good communication with other projects as e.g. the MATTER, SEARCH, MAXIMA and with the EERA Joint Program Nuclear Materials. Moreover, during the reporting period GETMAT has contributed to the identification of R&D gaps in the ODS area relevant for the JPNM and the proposal preparation of the MatISSE project.

Through the GETMAT User Group, parties (industries, non-EU research associations) that are external to the project are informed about relevant advancement of the project.

Finally, the GETMAT project has given an essential contribution to the Strategic Research and Innovation Agenda of the SNETP, to the EERA Joint Program on Nuclear Materials and to the EC road-mapping exercise on materials for the SET-Plan.

4.1 Use and dissemination of foreground

Detailed lists of dissemination measures, including any scientific publications relating to foreground, are contained in the Final Report submitted via the Participant Portal.
Section A (Public) contains a list of 76 scientific publications and another list of 174 dissemination activities, such as organisation of conferences, posters, exhibitions.
Section B should specify the exploitable foreground and provide plans for exploitation. These tables in the Participant Portal are empty, because the GETMAT project has not produced patents, trademarks, registered designs, etc.

4.2 Report on societal implications

A General Information *(completed automatically when **Grant Agreement number** is entered.*

Grant Agreement Number:	212175
Title of Project:	GEn IV and Transmutation MATerials
Name and Title of Coordinator:	Dr. Concetta Fazio, Karlsruher Institut fuer Technologie

B Ethics

1. Did your project undergo an Ethics Review (and/or Screening)? • If Yes: have you described the progress of compliance with the relevant Ethics Review/Screening Requirements in the frame of the periodic/final project reports? Special Reminder: the progress of compliance with the Ethics Review/Screening Requirements should be described in the Period/Final Project Reports under the Section 3.2.2 *'Work Progress and Achievements'*	***No***
2. Please indicate whether your project involved any of the following issues (tick box) :	
RESEARCH ON HUMANS	
• Did the project involve children?	No
• Did the project involve patients?	No
• Did the project involve persons not able to give consent?	No
• Did the project involve adult healthy volunteers?	No
• Did the project involve Human genetic material?	No
• Did the project involve Human biological samples?	No
• Did the project involve Human data collection?	No
RESEARCH ON HUMAN EMBRYO/FOETUS	
• Did the project involve Human Embryos?	No
• Did the project involve Human Foetal Tissue / Cells?	No
• Did the project involve Human Embryonic Stem Cells (hESCs)?	No
• Did the project on human Embryonic Stem Cells involve cells in culture?	No
• Did the project on human Embryonic Stem Cells involve the derivation of cells from Embryos?	No
PRIVACY	
• Did the project involve processing of genetic information or personal data (eg. health, sexual lifestyle, ethnicity, political opinion, religious or philosophical conviction)?	No
• Did the project involve tracking the location or observation of people?	No
RESEARCH ON ANIMALS	
• Did the project involve research on animals?	No
• Were those animals transgenic small laboratory animals?	No
• Were those animals transgenic farm animals?	No
• Were those animals cloned farm animals?	No
• Were those animals non-human primates?	No
RESEARCH INVOLVING DEVELOPING COUNTRIES	
• Did the project involve the use of local resources (genetic, animal, plant etc)?	No
• Was the project of benefit to local community (capacity building, access to healthcare, education etc)?	No
DUAL USE	
• Research having direct military use	No
• Research having the potential for terrorist abuse	No

C Workforce Statistics

3. Workforce statistics for the project: Please indicate in the table below the number of people who worked on the project (on a headcount basis).

Type of Position	Number of Women	Number of Men
Scientific Coordinator	1	0
Work package leaders	2	4
Experienced researchers (i.e. PhD holders)	28	85
PhD Students	16	14
Other	6	16

4. How many additional researchers (in companies and universities) were recruited specifically for this project?	**3**
Of which, indicate the number of men:	3

D Gender Aspects

5. Did you carry out specific Gender Equality Actions under the project?	○ X	Yes No

6. Which of the following actions did you carry out and how effective were they?

	Not at all effective				Very effective
❑ Design and implement an equal opportunity policy	○	○	○	○	○
❑ Set targets to achieve a gender balance in the workforce	○	○	○	○	○
❑ Organise conferences and workshops on gender	○	○	○	○	○
❑ Actions to improve work-life balance	○	○	○	○	○

○ Other:

7. Was there a gender dimension associated with the research content – i.e. wherever people were the focus of the research as, for example, consumers, users, patients or in trials, was the issue of gender considered and addressed?

○ Yes- please specify

X No

E Synergies with Science Education

8. Did your project involve working with students and/or school pupils (e.g. open days, participation in science festivals and events, prizes/competitions or joint projects)?

X Yes- please specify

- Regular visits to the University of high-school students / One day a year poster presentation of research at the University;
- PhD students of CNR;
- Open seminars for students and industry;
- student's project work;
- Annual open days at laboratory, although these were not specific to current project. One of the project scientist also did school outreach (on other funding)

○ No

9. Did the project generate any science education material (e.g. kits, websites, explanatory booklets, DVDs)?

X Yes- please specify: Pictures for lectures on materal

○ No

F Interdisciplinarity

10. Which disciplines (see list below) are involved in your project?

X Main discipline[1]: Physical Science

X Associated discipline[1]: Chemical Science

X Associated discipline[1]: Other Engineering Science

G Engaging with Civil society and policy makers

11a Did your project engage with societal actors beyond the research community? *(if 'No', go to Question 14)*	○ X	Yes No

11b If yes, did you engage with citizens (citizens' panels / juries) or organised civil society (NGOs, patients' groups etc.)?

○ No

○ Yes- in determining what research should be performed

○ Yes - in implementing the research

○ Yes, in communicating /disseminating / using the results of the project

[1] Insert number from list below (Frascati Manual).

11c In doing so, did your project involve actors whose role is mainly to organise the dialogue with citizens and organised civil society (e.g. professional mediator; communication company, science museums)?	○ ○	Yes No

12. Did you engage with government / public bodies or policy makers (including international organisations)

- ○ No
- ○ Yes- in framing the research agenda
- ○ Yes - in implementing the research agenda
- ○ Yes, in communicating /disseminating / using the results of the project

13a Will the project generate outputs (expertise or scientific advice) which could be used by policy makers?

- ○ Yes – as a **primary** objective (please indicate areas below- multiple answers possible)
- ○ Yes – as a **secondary** objective (please indicate areas below - multiple answer possible)
- ○ No

13b If Yes, in which fields?

Agriculture Audiovisual and Media Budget Competition Consumers Culture Customs Development Economic and Monetary Affairs Education, Training, Youth Employment and Social Affairs		Energy Enlargement Enterprise Environment External Relations External Trade Fisheries and Maritime Affairs Food Safety Foreign and Security Policy Fraud Humanitarian aid		Human rights Information Society Institutional affairs Internal Market Justice, freedom and security Public Health Regional Policy Research and Innovation Space Taxation Transport	

13c If Yes, at which level?

- ○ Local / regional levels
- ○ National level
- ○ European level
- ○ International level

H Use and dissemination

14. How many Articles were published/accepted for publication in peer-reviewed journals?	**76**
To how many of these is open access[2] provided?	**12**
How many of these are published in open access journals?	**12**
How many of these are published in open repositories?	**0**
To how many of these is open access not provided?	**64**
Please check all applicable reasons for not providing open access:	
❑ publisher's licensing agreement would not permit publishing in a repository ❑ no suitable repository available ❑ no suitable open access journal available ❑ no funds available to publish in an open access journal ❑ lack of time and resources ❑ lack of information on open access ❑ other[3]: ……………	

15. How many new patent applications ('priority filings') have been made? *("Technologically unique": multiple applications for the same invention in different jurisdictions should be counted as just one application of grant).*	**0**

16. Indicate how many of the following Intellectual Property Rights were applied for (give number in each box).	Trademark	**0**
	Registered design	**0**
	Other	**0**

17. How many spin-off companies were created / are planned as a direct result of the project?	**0**
Indicate the approximate number of additional jobs in these companies:	**0**

18. Please indicate whether your project has a potential impact on employment, in comparison with the situation before your project:

❑	Increase in employment, or	❑	In small & medium-sized enterprises
❑	Safeguard employment, or	❑	In large companies
❑	Decrease in employment,	X	None of the above / not relevant to the project
X	Difficult to estimate / not possible to quantify		

19. For your project partnership please estimate the employment effect resulting directly from your participation in Full Time Equivalent (*FTE = one person working fulltime for a year*) jobs:	*Indicate figure:* *Difficult to estimate*

[2] Open Access is defined as free of charge access for anyone via Internet.

[3] For instance: classification for security project.

Difficult to estimate / not possible to quantify	X

I Media and Communication to the general public

20. As part of the project, were any of the beneficiaries professionals in communication or media relations?

○ Yes X No

21. As part of the project, have any beneficiaries received professional media / communication training / advice to improve communication with the general public?

○ Yes X No

22 Which of the following have been used to communicate information about your project to the general public, or have resulted from your project?

❑	Press Release	X	Coverage in specialist press
❑	Media briefing	X	Coverage in general (non-specialist) press
❑	TV coverage / report	❑	Coverage in national press
❑	Radio coverage / report	❑	Coverage in international press
❑	Brochures /posters / flyers	X	Website for the general public / internet
❑	DVD /Film /Multimedia	X	Event targeting general public (festival, conference, exhibition, science café)

23 In which languages are the information products for the general public produced?

❑	Language of the coordinator	X	English
❑	Other language(s)		

2. FINAL REPORT ON THE DISTRIBUTION OF THE EUROPEAN UNION FINANCIAL CONTRIBUTION

The final report on the distribution of the European Union financial contribution can only be prepared after the final payment of the European Union.
In the GETMAT periodic reports, the distribution of the payments received in the reporting period has been shown and commented.