

Towards a new approach for structural materials of Lead Fast Reactors

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Abstract. The development of Pb and LBE cooled fast reactors represents a unique challenge for the materials that are subject, at the same time, to degradation mechanisms related to neutron radiation damage, exposure to high temperatures and exposure to the HLM. The protection towards HLM corrosion, in the past, has been based on the control of the oxygen concentration in the HLM by keeping high oxygen concentration in the liquid metal and prevent the onset of severe oxidation. The proposed approach bases the protection of structural materials on oxides more stable than chromium oxide in HLM; namely alumina. Two paths are under study to perform stable oxides protections on LFR structures: 1) steel coating by anti-corrosion barriers, 2) alumina forming austenitics based on the optimization of composition to improve the resistance to irradiation swelling and maintain suitable mechanical properties.

Key Words: Lead Fast Reactor, HLM Corrosion, Alumina Coating, Alumina Forming Austenitics,

1. Introduction

Corrosion occurred as the greatest technological problem for Heavy Liquid Metal (HLM) cooled reactors since their first application, regarding the eutectic lead-bismuth alloy used for Alfa class Soviet submarines in the sixties. Several detrimental phenomena are involved to this regard, such as non-passivating oxidation, leading to thick oxide layers which are not stable and finally can cause dissolution of the steel constitutive elements. The most intensive and rapid corrosive process consists in the dissolution of the characteristic alloying elements of structural steels. The most sensitive element to this phenomenon is nickel but also chromium and iron show a certain solubility in heavy metals melts. The dissolutive process, if not counter-acted, proceeds at the speed of 200 microns/year at a temperature of 550 °C for a typical austenitic steel exposed to liquid lead at concentration of 10⁻⁷ wt% oxygen [1].

Various counter-measures can be taken to control the corrosion phenomena, following the approach currently adopted in other technological fields and based on different approaches, acting for example on: material selection, design (low temperature operations by design, decrease velocity of fluids to reduce erosion/corrosion..) and environmental control (low temperature, active control of the concentration of oxygen, inhibitors,...). The research work carried out during the last decade has shown that all the conventional steels for nuclear applications suffer to various extent of corrosion issues in HLM, so that their use may pose operational problems. The environmental control, namely the operation under controlled oxygen content and low temperature conditions in order to keep low oxidation levels (Active Oxygen Control, AOC), has proven to be effective in handling corrosion issues by promoting the formation of a self-healing oxide film on the steels surface, so reducing steel corrosion and coolant contamination.

AOC was the solution devised by the Russian designers in the sixties and proved to be very practical and efficient at the operational temperature of Alfa class submarines, although that might lead to safety problems. It consisted in dissolving oxygen in the liquid metal and keeping its concentration lower than the value at which lead oxide precipitates but high enough to allow the formation of the protective oxides, mainly chromia (Cr_2O_3), on the stainless steel surface. To this purpose, by means of suitable control systems, a high dissolved oxygen content, between 10^{-5} wt% and 10^{-6} wt%, was kept within the heavy liquid metal. Figure 1 illustrates in a simplified manner, by using the Ellingham diagram, the concentration limits for the oxygen dissolved for a LFR reactor following the approach adopted for Alfa class submarines.

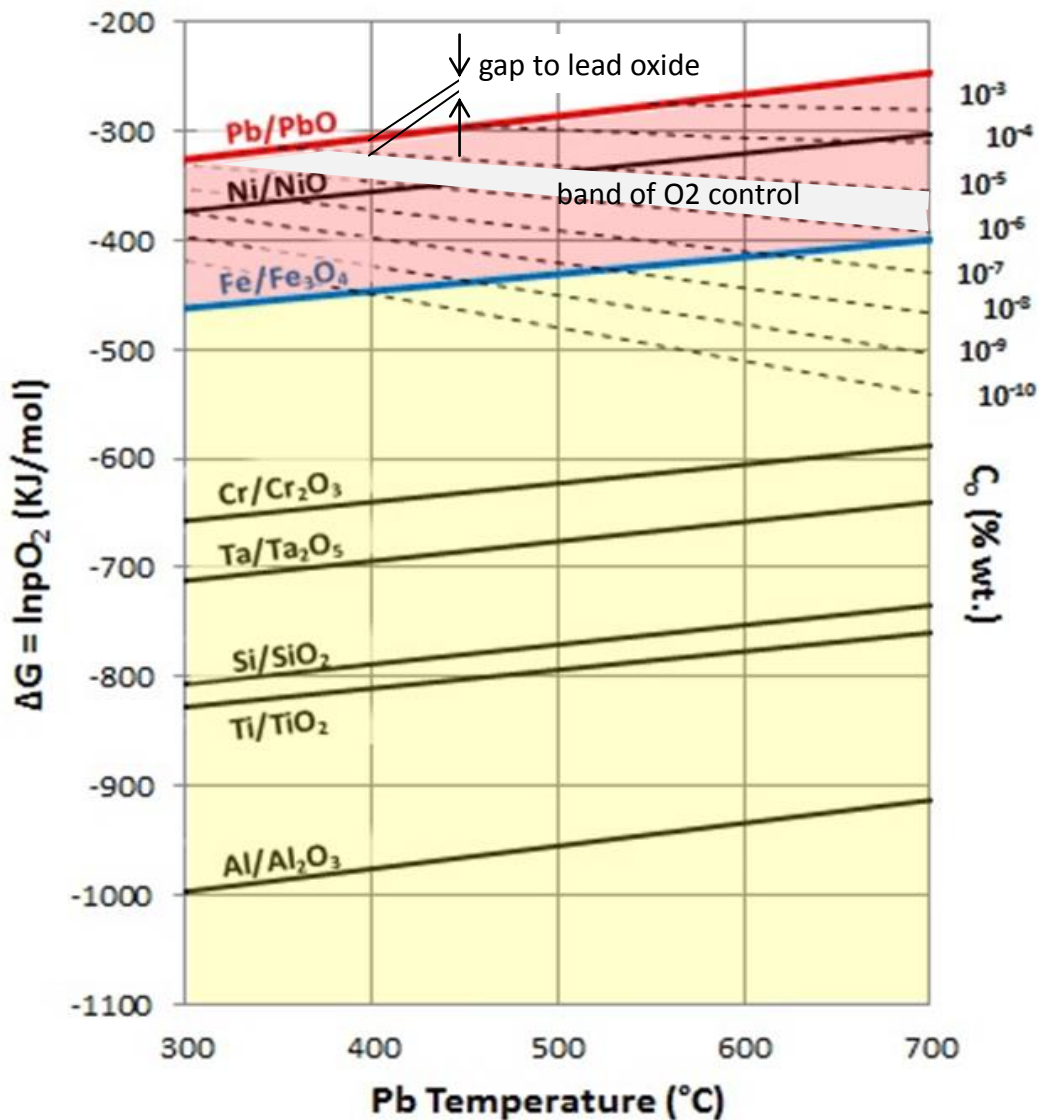


FIG. 1 Ellingham diagram

Note that the lower level of oxygen content allows, at any operational temperature, the formation of magnetite, the less stable oxide among those constituting the protective layer while the upper level is set to prevent the formation of lead oxide.

Chromia layers have been reported to provide adequate corrosion resistance up to about 500 °C, after which dissolution attack quickly occurs [2]. In general it has been shown that above

temperatures of around 450~500°C, depending on the steel considered and on the working conditions, the operation under controlled oxygen content is not effective and the chromium rich oxide layer that forms on the steel surface and acts as physical barrier to further oxidation in most environments, is not effective in passivating the steel in HLM containing oxygen at high temperature.

2. Materials issue for LFR

In order to simplify the control process that, for a thermodynamic machine of significant dimensions, also requires a continuous measurement of the thermal stratification, it could be appropriate to maintain oxygen level next to the upper limit to prevent metal dissolution everywhere. As the upper limit of oxygen corresponds to the formation of lead oxides, at the lower temperature (400°C), a careful control of the oxygen content is necessary since the gap in oxygen concentration to form lead oxide, that is a solid product, is very narrow, as can be seen in figure 1. The PbO solid formations could clog the coolant channels. The two conflicting needs to ensure the formation of the layer of chromium and iron oxides and to prevent the formation of lead oxides, require a sophisticated control through very fine adjustment of the system chemistry.

For a reactor pool volume larger than 100 cubic meters, it appears difficult to maintain accurate control of the coolant chemistry in order to keep the oxygen concentration in the limits allowed to inhibit corrosion. For this reason some recent lead reactor designs intend to assign corrosion control rather than to the AOC, to the operation of the reactor at high temperatures and low oxygen concentrations and the careful selection of materials, either by special alloys or by surface treatment. Both approaches are viable and, if successful, will enable significant cost savings to the chemical control, as well as increased safety.

The current design of the lead cooled ALFRED reactor foresees operating the reactor with an oxygen content well below the saturation limit at the coldest temperature of the coolant (400°C), so eliminating the risk of lead oxide precipitation in the whole system. This option exposes the steels to severe corrosion/dissolution issues in the regions of the reactor operated at temperatures above 450 °C. The core structures are at temperatures higher than the above mentioned limits at which oxidation is not protective and the corrosion processes cannot be neglected; due to the small thickness of the cladding tubes, additional protection measures must be put in place. A long term strategy foresees the development of a new class of materials resistant to the oxidation in HLM environment and able to withstand the neutron radiation damage up to elevated doses. As an example, self-passivating alumina forming austenitic steels have shown good performances compared to the conventional steels. Additionally, technological advancement are expected to enable the fuel cladding of LFR concepts to operate from the present lower temperatures (550°C) to the higher future temperatures (800°C). Materials capable of higher temperature exposure will be needed to support the 800°C systems and these materials are likely to differ from those envisaged at present.

In the short term the most viable solution to face corrosion is the use of the special steels developed so far for fuel claddings of fast reactors and assign the protection from the HLM corrosion to protective surface treatments. The base material is kept to retain the mechanical properties aimed to guarantee the integrity of core components against stresses and irradiation. The surface material/treatment is added to complement the base material with the corrosion resistance. This approach has the obvious advantage of using materials with known properties already codified in the regulations [3]. In this context, in Italy at ENEA were

promoted studies to identify suitable protective coatings for a Lead cooled reactor as well as new metal alloys that do not need any coating.

The main target of the research was the development of a suitable material for fuel claddings of Lead Fast Reactor, which represent the main challenge for this kind of reactors. As second priority also the clear identification of structural materials for main vessel and reactor internals represent an important technological challenge.

The reference material was the 15-15 Ti steel used in sodium cooled fast reactors, due to its already proven good performance in high temperature, fast neutrons environment. The 15-15 Ti steel, like other austenitic steels, is prone to liquid metal corrosion in HLM, although less intense than 316L steel [1]. The corrosion protection coating of such steel appeared the most practical solution to preserve the thermomechanical and swelling performance and to enhance its corrosion resistance.

Looking at the Ellingham diagram in Figure 1, one can immediately observe that the more stable protective oxide for lead environment is aluminum oxide Al_2O_3 given the lowest value of free energy of formation. Therefore the first objective was the development of ceramic alumina coatings and metallic alumina forming coatings. Parallel to this research, which will be presented in a dedicated paragraph, it was also started the development of an advanced austenitic steel that should be possibly used for the fuel claddings without any need for coating.

From the fabrication point of view of coatings for the cladding, it is required that, to avoid unwanted reactions in the MOX fuel contained within the pins, the coating deposition process happen without any temperature increase. This prescription is also necessary since the swelling resistant austenitic steels of the class 15-15 Ti are metastable by design. As a consequence any thermal modification of the material which could hinder its swelling properties must be avoided. The qualification of this approach consisted of corrosion test, scratch tests and thermal shocks to verify adherence, mechanical tests and thermal cycling tests to verify compliance with the substrate even in plastic condition and finally heavy ions irradiations to simulate the behavior under fast neutrons flux.

The long term strategy aims to devise a completely new fuel cladding material that besides corrosion resistance, should be able to face creep, swelling and loss of toughness which are typical in the harsh LFR core operating conditions. This achievement will require a long lasting development as well as qualification process. An intermediate step will be the development of a new material for LFR steam generator and internals. This material will associate the required chemical compatibility and mechanical properties, outside the intense neutron flux. In case of successful performance, a second qualification phase will be performed to extensively verify its resistance under fast neutron flux, up to 100 dpa neutron damage. At present several studies are ongoing to identify candidate materials which could be adapted to address additional performances. Recent material researches by Oak Ridge [4] and KTH [1] have focused on the corrosion and creep performance of Alumina Forming Austenitic (AFA) steels. These materials are candidate for the long term strategy process as better described in the dedicated paragraph.

3. Short term strategy based on surface protection

The candidate coatings/surface treatments can be divided in two classes, inert coatings and self-passivating coatings. In the first case the protection is provided by a compound with high stability in the environment, in the second case the protection is provided by the precipitation of a stable compound by reaction with environment that passivates the surface.

Beyond the corrosion resistance in HLM, the coating must have good adhesion and mechanical compatibility with the substrate, low swelling property and a stable microstructure under neutron irradiation up to elevated dpa doses. Surface finish is critical in order to achieve the optimum performance in terms of endurance under thermal cycling and the surfaces to be coated need to be mirror finished (RMS roughness <10 nm) by grinding and polishing. The self-healing i.e. ability to regenerate by reaction with the environment in case of scratch or surface failure is a further requirement. As stated above, the acceptable deposition processes must be carried out at low temperature in order to avoid the degradation of the steel performance under neutron irradiation. This constraint ruled out the adoption of diffusion coating techniques that imply the exposure to high temperatures for times of the order of hours to allow for the formation of the diffusion layer over the steel surface. Low temperature pack cementation FeCrAl depositions have been tried but with poor results in terms of thickness of the diffused layer and surface roughness.

3.1. Passivating coatings

Among the overlay metallic coatings, FeAl and FeCrAl showed excellent behavior in oxygen rich liquid lead and after 5000 hours of exposure in the CHEOPEIII loop, FeAl specimens showed an outstanding performance with no corrosion nor erosion attacks (figure 3).

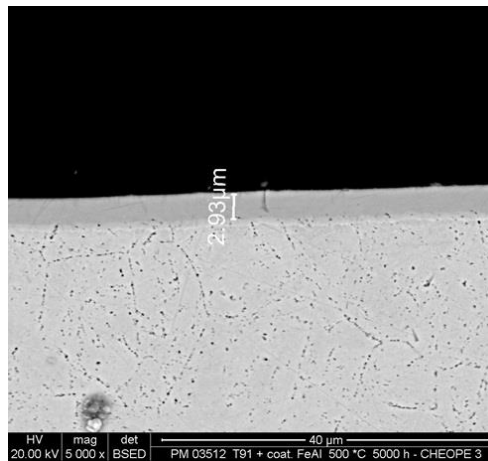


FIG.3. FeCr coating on T91 Steel after 5000 hours exposition in flowing Pb at 500°C

Coatings have been produced via several techniques, namely magnetron sputtering, High Velocity Oxy Fuel and Arc-PVD. Long-term tests in flowing lead and thermal creep experiments in molten lead are under way. Nevertheless, apart from corrosion and thermal creep, other issues still need to be addressed. Among them, the most important is the resistance to the displacement damage induced by irradiation, swelling, irradiation creep, stability of the microstructure, changes in mechanical properties and adhesion strength.

To protect the core structural materials, the coatings must retain their properties after neutron irradiation up to a dose of 100 dpa that is the dose at which the fuel element is replaced for reprocessing. The 100 dpa dose is typically obtained after 3 years irradiation in the core of the Alfred reactor. In order to assess the performance of FeCrAl coatings under neutron irradiation, an ion irradiation campaign has been started at the National Laboratories of the Istituto Nazionale di Fisica Nucleare (INFN).

3.2. Ceramic coatings

The experimental campaign on ceramic coatings was mainly focused on the deposition of Al_2O_3 coatings by detonation gun spraying and by pulsed laser deposition. Detonation gun spraying is a thermal spray coating process, which gives overlay coatings with an extremely good adhesive strength, low porosity and coating surface with compressive residual stresses.

A schematic view of the deposition apparatus is shown in figure 4. The combustion of a gas mixture in the detonation chamber generates high pressure shock waves, which then propagate through the gas stream. The hot gases produced travel through the barrel at a high speed and high temperature, carrying the material to be deposited in a plastic state with velocity of 1200m/sec. The particles impact on the surface form a splat and the coating results from several splats.

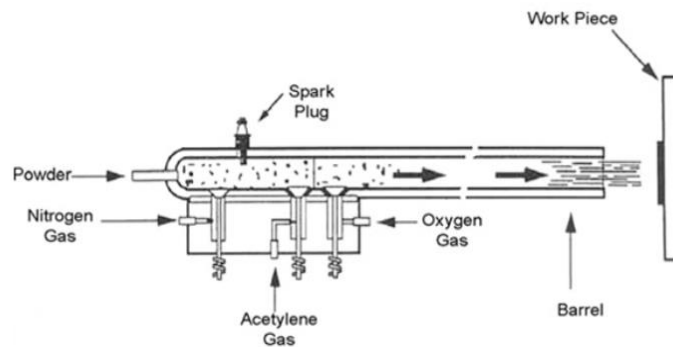


FIG.4. Schematic view of detonation spray device

The temperature and speed of the particles lead to dense and adherent layers. Due to the size of the splats and the residual stresses generated during the cooling down, the minimum applicable protective layer is around 10 micrometers. Although the powders reach temperatures up to 4000°C the deposition does not affect the substrate temperature which remains cold. The residual stresses can be reduced by interposing a buffer layer on the substrate.

The technique is mainly suitable for reactor vessel where the precision control of the layer thickness is not required and large surface have to be treated. A typical sample is shown in figure 5 as obtained by 30 micrometers powder size and using an aluminum intermediate layer.

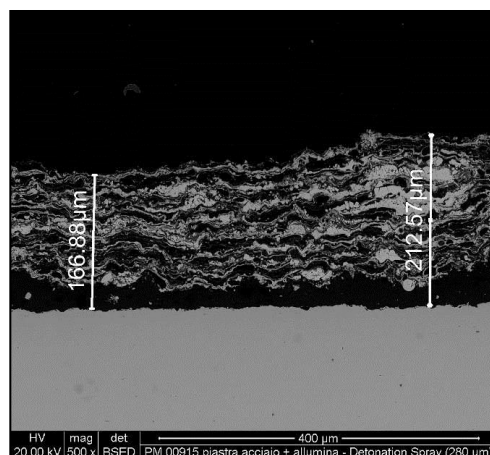


FIG.5. Alumina coated steel by detonation spray

The pulsed laser deposition technique (PLD) uses high power UV laser pulses to melt, evaporate and ionize material from the surface of a target of the material to be deposited so producing a plasma plume that expands at high speed to re-condense on a substrate where the coating growth occurs. The Al₂O₃ coatings produced by PLD developed by the Istituto Italiano di Tecnologia in collaboration with ENEA at present represent the most promising option to protect the core components by the corrosion. Pulsed Laser Deposition (PLD) is a relatively simple technology that allows to grow thin films of a wide range of materials. The deposition conditions can be adjusted to obtain different coating microstructures, varying from fully dense and compact to columnar and porous [5]. Fully dense and compact Al₂O₃ coating grown by PLD on various steels have been obtained by tailoring the deposition process to attain an advanced nanocomposite which consists of a homogeneous dispersion of ultra-fine nanocrystalline domains (6±4 nm) in an amorphous alumina matrix (figure 6), with the desired properties [6]. Some of the main features of this material are an ensemble of metal-like mechanical properties ($E=195\pm9$ GPa, $\nu=0,29\pm0,02$) and enhanced plastic behavior, full compactness and strong adhesion, as well as a relatively high hardness ($H=10$ GPa) [6]. Another interesting feature of this material is the coefficient of thermal expansion, which is higher than for polycrystalline aluminum oxide. Specifically, reference values for sapphire are around 8×10^{-6} °C⁻¹, whereas a mean value of 12.66×10^{-6} °C⁻¹ between 300 K and 1073 K was measured for PLD-grown Al₂O₃ [7]. This value is close to the reference value for 1515Ti steels (i.e. in the range of 12×10^{-6} °C⁻¹ at room temperature [8]), providing benefits with respect to thermal cycling issues. As a matter of fact, the coating-substrate interface has been observed to perform well under repeated thermal cycles between 350°C and 600°C, with no loss of adhesion nor surface cracking [9].

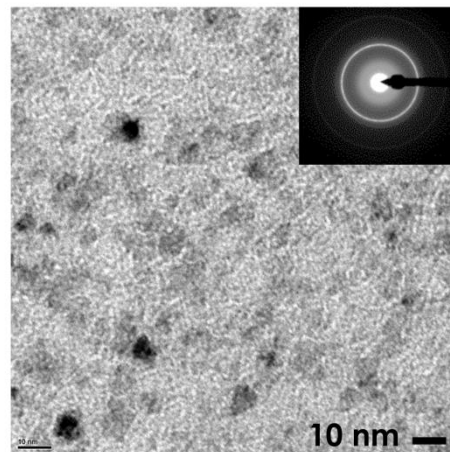


FIG.6. Microstructure of the Al₂O₃ coatings showing homogeneous dispersion of randomly oriented crystalline Al₂O₃ nanodomains in an amorphous Al₂O₃ matrix as shown in the electron diffraction patterns on the right.

The Al₂O₃ barriers have been tested for protecting the steels up to 600°C in stagnant HLMS with outstanding results [10]. Long-term (>10.000 hours) tests in flowing Lead and thermal creep experiments on coated 15-15 Ti tubes and plates are in progress. The performance of the Al₂O₃ nanocomposites under irradiation have been investigated via heavy ion irradiations during several experiments at various doses up to 150 dpa at 600 °C. Irradiations have been carried out at the JANNUS-Saclay platform on bilayers FeCrAlY buffer +Top Al₂O₃ on 15-15 Ti and the doses were 20, 40, and 150 dpa. The energy of the ions has been chosen in order to obtain the maximum displacement damage at the interface with the substrate. Such high levels of radiation damage approach or even exceed the damage exposures foreseen for the ALFRED reactor. In figure 7 a,b,c,d it is shown the evolution of the microstructure increasing

the irradiation dose. These images indicate that increasing the irradiation dose, a fully nanocrystalline structure is developed followed by grain growth. The average grain size increases from 6 ± 4 nm to 101 ± 56 nm at 20 dpa, 153 ± 62 nm at 40 dpa and 293 ± 85 nm at 150 dpa. The crystalline phases present in the irradiated nanoceramic are γ -Al₂O₃ up to 40 dpa, and both γ -Al₂O₃ and α -Al₂O₃ at 150 dpa. The presence of these different phases doesn't seem to affect the performance of the coatings, since no surface cracks are observed, regardless of the dpa levels reached. Some voids are present at the interface with the steel substrate and in the FeCrAlY bonding layer. It is important to outline that the irradiation did not induce any loss of adhesion or delamination effects at the thin film-substrate[11]. The ion irradiations are part of a broader irradiation project which includes irradiation experiments on coated steel samples by fast neutron flux up to high doses.

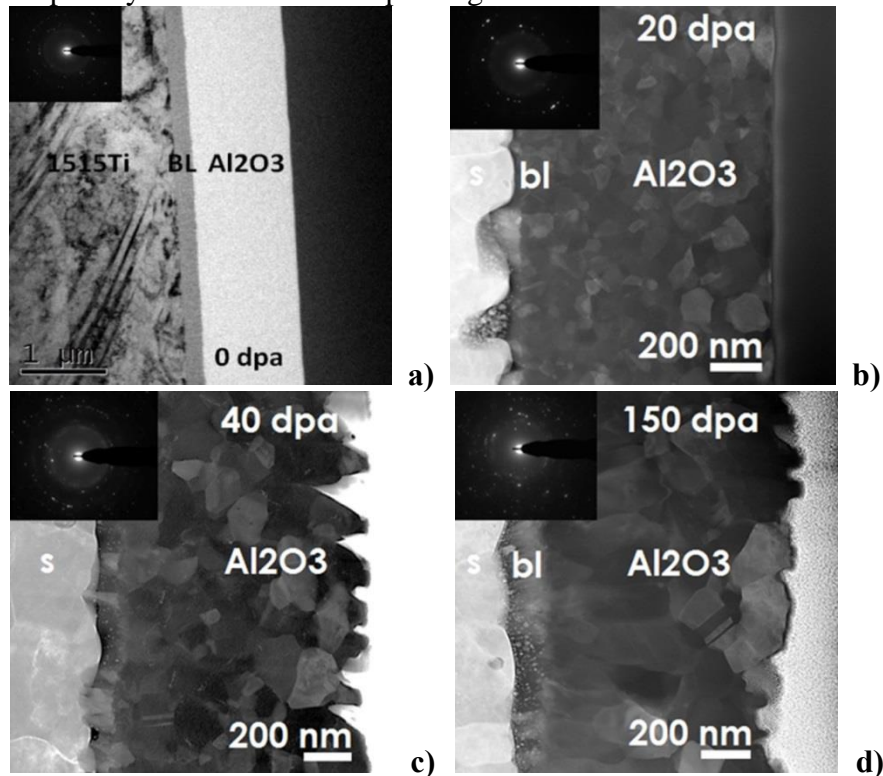


FIG.7. TEM micrographs (diffraction pattern in the insets) showing the microstructural evolution during ion irradiation: as-deposited (a) after 20 dpa (b), 40 dpa (c) and 150 dpa (d) at 600 °C

4. Long term strategy based on corrosion resistant advanced steels

Alumina forming austenitic (AFA) Steels were recently developed and commercialized by the American company Carpenter Technology Corporation with the aim to increase the corrosion/oxidation resistance and the creep strength at high temperature [12]. These steels, that contain a maximum 25% Ni exhibit a creep strength comparable with some superalloys containing a much higher amount of Nickel and a superior oxidation resistance due to the formation of a Alumina-Chromia rich layer. Some AFA alloys were tested in Liquid Pb environment, as reported previously [1], showing an outstanding corrosion resistance.

The steels of the 15-15 Ti class are the result of an R&D activity lasted various decades by fine-tuning the composition of the alloy; for many aspects, the development/tuning of a new class of swelling resistant alumina forming alloys is a challenging task. On the other hand the large mole of data produced, the methodology developed and the use of computational thermodynamics tools can be used for an accelerated development. The mitigation of the

swelling in austenitic steels has been achieved by an alloy design which account for the following factors as demonstrated by the improvements obtained during the last decades:

- optimization of the solid solution concentration of alloying and impurity elements that form “point defect-impurity” complexes acting as vacancies-interstitial recombination centres and/or change the diffusion characteristics of the point defects,
- introduction of a certain degree of phase instability leading to secondary “in-service” precipitation of coherent carbides, highly effective in stabilizing the dislocations microstructure and promoting recombination at the particle-matrix interface,
- optimization of the density of dislocations acting as point defect sinks and retarding the onset of swelling.

The factors that control swelling are clearly interrelated and influence each other.

The Carpenter AFA steel for several aspects is very similar to A286 alloy, for which neutron irradiation data are available and some guidance considerations can be inferred by its behaviour. A286 is a 25% Ni superalloy hardened by 2%Ti-0.2%Al and γ' precipitation after ageing treatment with excellent swelling and creep resistance during neutron irradiation. A286 as well as other γ' hardened alloys have been studied in the US National Alloy Development Program for application of gamma prime hardened alloys in liquid metal fast breeder reactors. The following Table 1 reports the composition of the two alloys.

	C	Mn	Si	P	S	Cr	Ni	Mo	Cu	V	Ti	Al	B	W	Nb	Fe
A286	0,04	1,27	0,53	0,015	0,002	14,04	25,8	1,3	0,08	0,26	2,17	0,19	0,004	0	0	Bal
OC-A[10]	0,05	2	0,14	0,22	0,011	14,06	25,02	2	0,51	0,05	0,05	3,03	0,01	0,96	1,02	Bal

Table 1. The composition of the A286 and OC-A alloys (Wt.%)

The following figure 8 reports a thermodynamic calculation performed with MatCalc Code by using the Ni-alloys database. Based on the simulation it is possible to do some preliminary considerations.

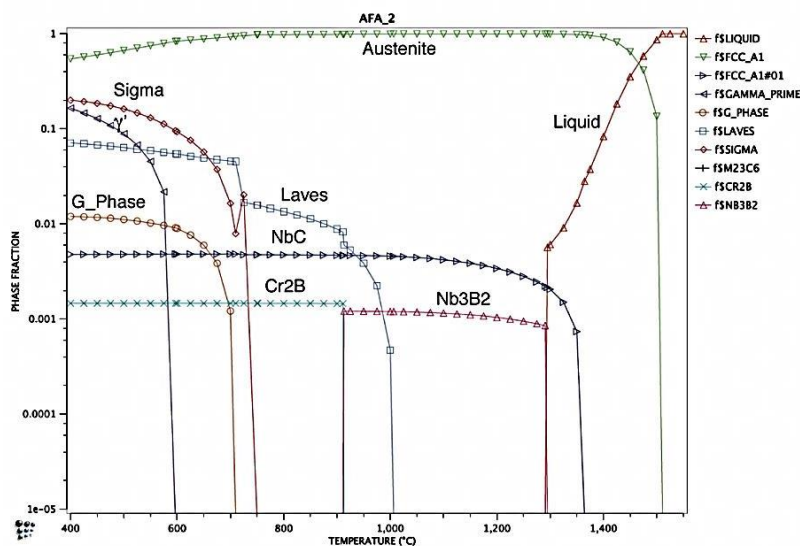


FIG.8. Stepped Thermodynamic calculation for the 25Ni-2Mn-0.14Si-14Cr-2Mo-0.96W-3Al-1.02Nb-0.05 C-0.0096B (Alloy OC-A in [10]).

Fig.8 shows that G-phase is foreseen to form under thermal condition and may be enhanced by irradiation. G-phase is known to be associated with high swelling due to the high growth-rate of the voids attached to the Si-rich particles [13]. The silicon content in OC-A is pretty low and should be kept as it is, given its beneficial effect on swelling. Since the development

of G-phase by thermal ageing in alloy OC-A is due to niobium, it is needed to eliminate or strongly reduce the Nb content and substitute it with Titanium. Gamma prime Ni₃(Ti,Al) tends to transform into eta phase (Ni₃Ti) after long term thermal aging and irradiation enhances the tendency for transformation in A286 [14]: the very low Ti to Al ratio in the OC-A steel should prevent this issue. Some embrittlement phenomena at low irradiation temperature were reported for A286 steel caused by γ' precipitation and nucleation of dislocation loops [15]. These issues could equally affect the performance of the AFA under irradiation and the microstructural and compositional characteristics needed to reduce the low temperature interstitial loops nucleation rate should be identified and corrected. All these items are under investigation by the thermodynamic point of view, the analysis of the literature data and heavy ions irradiations.

The development of AFA steels as fuel cladding materials requires several compositional changes and qualification under neutron irradiation. The lesson learned during the development of superalloys for fast reactors applications and the use of thermodynamic simulations and ion irradiations can be used for an accelerated development. On the contrary commercial AFA steels, can be used for all parts that are outside the reactor core, such as heat exchangers and internals, due to their high creep strength and oxidation resistance in Liquid Pb environment.

Acknowledgements

The authors acknowledge the support from the Italian Ministry of Economic Development through the AdP MiSE-ENEA. This work contributes to Sub-Program SP3 of the Joint Program on Nuclear Materials of the European Energy Research Alliance. The French network of irradiation facilities EMIR is acknowledged for granted access to the JANNUS platform of CEA-Saclay.

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