



Ricerca di Sistema elettrico

Risultati dello studio per l'individuazione
e la validazione dei materiali da utilizzare
come getters per l'azoto sciolto in Litio

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RISULTATI DELLO STUDIO PER L'INDIVIDUAZIONE E LA VALIDAZIONE DEI MATERIALI DA UTILIZZARE COME GETTERS PER L'AZOTO SCIOLTO IN LITIO

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Responsabile del Progetto: A. Pizzuto, ENEA

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Sommario

This report deals with the topic of the selection of the best material(s) to employ as getter for Nitrogen solved in Lithium, i.e. to put inside the Hot Trap of a flowing Lithium plant in order to reduce as much as possible Nitrogen concentration in Lithium. To achieve this result, the getter, a solid and insoluble metallic material, must be able both to quickly adsorb Nitrogen from Lithium and to form with it a compound more stable than Lithium Nitride. The chemical nature of the getter, as well as its format and the operative parameters of its employment (temperature, contact time with Lithium,...) require to be deeply investigated in order to find out the best Hot Trap operative settings and get the higher purification efficiency.

This report introduces first the theoretical aspects of the gettering process, both the thermodynamic and the kinetic ones, then present a concise overview of the most significant experiments performed worldwide in this field by many different research groups since the last decades, particularly lingering on the getter attitude of Chromium, Vanadium, Tantalum, Iron and their alloys.

At the end of the discussion, an univocal choice of the best getter material is not possible, because the many tests performed by the many authors were often often not homogeneous (differing in temperature, Lithium flow rate, inventory and starting purity, time of the experiment...), therefore a direct comparison of all the results is not easily feasible. Anyway, it has been possible to narrow the selection of the potentially good getters, corresponding to pure Titanium and its alloys and maybe also Niobium, so to focus only on them a future, deeper comparative investigation. This experimental activity will start in the next months in ENEA laboratories.

1 Introduction

The purity level of liquid Lithium flowing inside a plant is one of the important aspects to take into account to minimize the corrosion exerted by the alkaline metal on the surfaces of the steels constituting the plant, this way assuring the desired plant efficiency and lifetime. Mostly, the presence of non metals like Carbon (C), Oxygen (O), Hydrogen (H) and particularly Nitrogen (N), in the form of anions solved by Lithium, greatly enhances the chemical corrosion mechanism affecting the steels [1-4]. Lithium purification is therefore mandatory and for this reason ENEA has been involved since the last decade in theoretical studies and experimental activities aimed both at defining the proper purification procedures and at quantifying the Nitrogen concentration in Lithium, as described in Rds/2013/125 [5], Rds/2013/126 [6], Rds/2013/209 [7] and [8]; these solutions have been implemented inside the Lifus 6 plant recently realized in the Brasimone Research Centre, where the target of a maximum Nitrogen concentration equal to 30 wppm had been set.

Despite the encouraging results achieved until now by ENEA in the purification of Lithium from Nitrogen [8], the full comprehension of the Nitrogen removal mechanisms, the possible need for an even purer Lithium (Nitrogen concentration ≤ 10 wppm, as for the future IFMIF-DONES facility) and the identification of the best experimental conditions for the process demand additional investigation and justify the activity summarized in this report, aimed at the selection of the most promising getter candidates for Nitrogen.

Nitrogen cannot in fact be reduced to concentration of the order of 10 wppm employing a simple precipitation method (Cold Trap), since even just above Lithium melting point its solubility is around 100 wppm: a different method is hence necessary. This method entails the use of a solid and insoluble metallic material, hereafter designed as 'getter', able to adsorb Nitrogen from Lithium thanks to the formation of a compound which can result more stable than Lithium Nitride. This process is to be performed inside a dedicated plant section, commonly called 'Nitrogen Hot Trap', since it requires temperature generally around 500-600°C, surely higher than the operative temperature of rest of the plant (this originates the term 'hot'). Conceptually, this trap is not but a large cylindrical tank, where Lithium is allowed to stay in contact with the selected getter, at high temperature and for a rather long time. The efficiency of the trap surely relies on the choice of the material employed as getter and in the definition of the best operative parameters (temperature, flow rate, purification time...) for such a material.

In the selection of the getter material, two main aspects require to be investigated: first, the thermodynamics of the process, which could preliminarily indicate if, on the basis of the stability of the N-getter system compared to the stability of the N-Li system, the chemical nature of the getter has the potential attitude to quantitatively remove Nitrogen; second, the kinetics of the process, which, considering all the steps through which Nitrogen diffuses across two phases in a different aggregation state, could indicate if the desired purity level can be achieved in a 'reasonable' time. Both the two aspects are anyway difficult to predict, in view of the complexity of the system: preliminary theoretical considerations can be useful essentially to exclude specific materials from being employed as getter for Nitrogen. However, to confirm the efficiency of the ones theoretically suitable for the application, a deep experimentation must be done, emulating as much as possible the real operative conditions, in terms of temperature, starting concentration, Lithium inventory, mass and format of the getter.

In the next sections is presented a summary of the main thermodynamic and kinetic aspects of the Nitrogen gettering process, together with a concise overview of the most significant experiments done since the last decades in this field. On the basis of all the data available, a narrow selection of the most suitable getters is proposed, deserving a future, deeper, comparative investigation.

2 Description of the activities and results

2.1 Thermodynamic considerations about the gettering process

The process through which Nitrogen is removed from liquid Lithium by an insoluble metallic getter can be viewed in its more simple and general way as a partition equilibrium between two phases in a different aggregation state: Nitrogen will move across the two phases, increasing its own activity in one of them and decreasing it in the other, until its chemical potentials in the two phases will match. Anyway, in considering this equilibrium, all the possible ways Nitrogen distributes inside each phase must be taken into account: in Lithium phase it could exist both as a solved N^{3-} anion and as Li_3N compound; similarly, in the getter phase, it could exist as a free element solved in the matrix of the solid metal or, depending on its concentration, could give rise to many segregated compounds with the general stoichiometry G_aN_b . At equilibrium condition, the ratio of the activities will correspond to a fixed value (at a given temperature), defined by the distribution coefficient, D_C , as below:

$$D_C = \frac{a_{N(G)}}{a_{N(Li)}} = e^{\frac{\mu^\circ(Li) - \mu^\circ(G)}{RT}} \quad (1)$$

where R is the gas constant, T is the absolute temperature, $\mu^\circ_{(x)}$ is the Nitrogen chemical potential in the x phase (Li =Lithium; G =Getter) in the standard state and $a_{N(x)}$ is the chemical activity of Nitrogen in the x phase. From the above equation, it is evident that the getter will be able to remove Nitrogen from Lithium if the difference $(\mu^\circ_{(Li)} - \mu^\circ_{(G)})$ is > 0 and the more this difference is positive.

A practical approach to calculate the thermodynamics of the system through measured or measurable quantities is based on the fact that free energy (as well as enthalpy and entropy) is a state function, not depending on the specific reaction path through which that state has been achieved. In this respect, it must be considered that, while Nitrogen activity in getter phase is related to ΔG°_f of G_aN_b ¹, in Lithium phase is related to ΔG°_f of Li_3N . Assuming that dilute solutions of N obey Henry's law in both the phases and that solubility (i.e. saturation concentration) at a given temperature is related to the solution energy (G_{soln}), it is possible to finally get an expression for the distribution coefficient (K) in terms of the equilibrium concentrations in the two phases ($C_{N(x)}$), as the one reported by Natesan [1]:

$$K = \frac{C_{N(G)}}{C_{N(Li)}} = \frac{C^\circ_{N(G)}}{C^\circ_{N(Li)}} e^{\frac{\Delta G^\circ_f(Li_3N) - \Delta G^\circ_f(GaN_b)}{RT}} \quad (2)$$

where $C^\circ_{N(x)}$ is the solubility of N in the x phase, $\Delta G^\circ_{f(Li_3N)}$ is the standard free energy of formation of Li_3N and $\Delta G^\circ_{f(GaN_b)}$ is the standard free energy of formation of the most stable compound N can form in the getter phase. The better getter would be therefore the one able to form a stable compound with Nitrogen (G_aN_b), since, the more negative is $\Delta G^\circ_{f(GaN_b)}$, the higher will result the value of K . Figure 1 left shows the $\Delta G^\circ_{f(GaN_b)}$ values of many transition metals nitrides versus absolute temperature, together with Li 's one: it is evident that only those metals whose curves are located below Li 's one could be selected as potential getter for Nitrogen.

Looking at equation (2), we see that K depends also on the solubilities of Nitrogen in the 2 phases, which both vary with temperature as well, according again to a logarithmic equation:

¹ Assuming G_aN_b is the unique or the largely most stable N -getter compound formed in the getter phase. If many compounds, with comparable stability, can form, they should all be considered in the (more complex) equation.

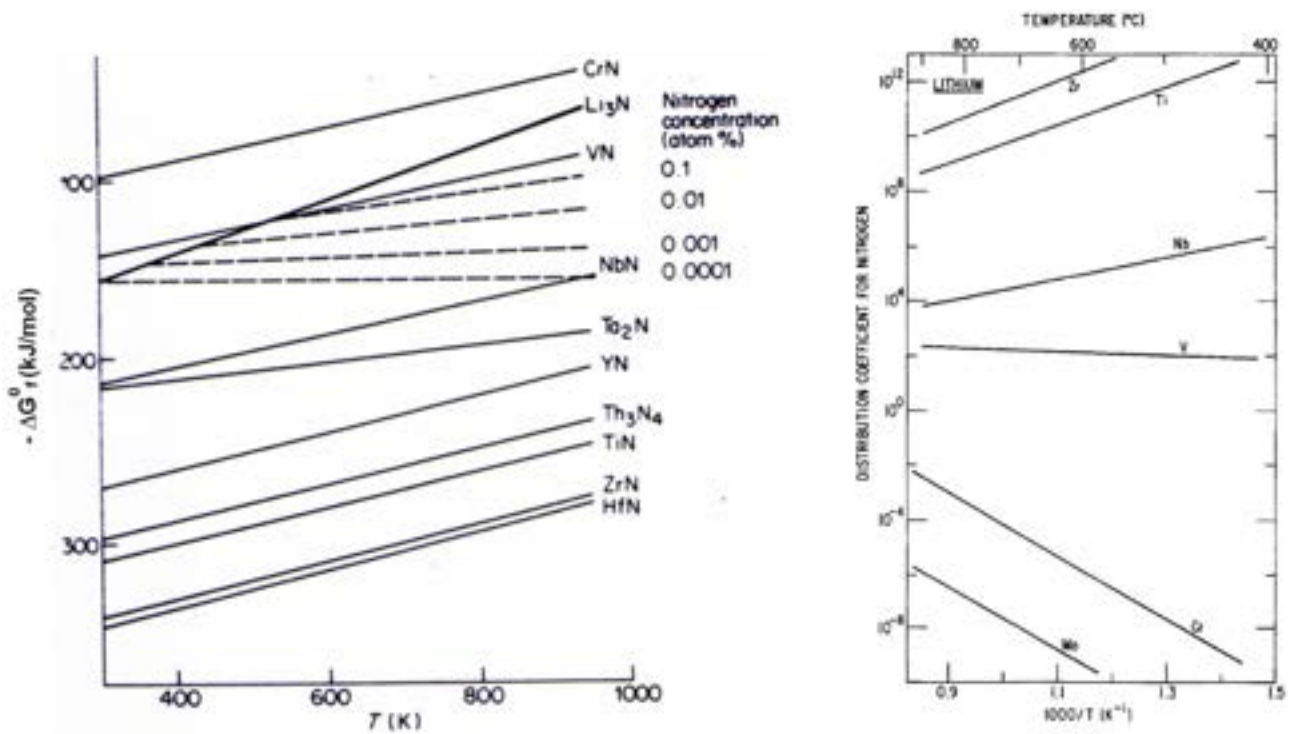


Figure 1: left: Ellingham diagram (free energy diagram for Nitrides); right: equilibrium distribution coefficients (K) for Nitrogen between Lithium and various transition metals

$$\ln(C_{N(x)}^{\circ}) = A + B/T \quad (3)$$

where A and B are known values [9], approximately constant with Temperature, respectively related to the partial molar entropy of the solution (S_{soln}) and to the partial molar enthalpy of the solution (H_{soln}) [10]. Introducing the dependences described by (3) in (2), is it possible to finally get a functional dependence of the distribution factor K on T:

$$\ln K = A_1 + B_1/T \quad (4)$$

The distribution coefficients calculated with (4) for Nitrogen between Lithium and many other metals are shown in Figure 1 right [1]: those characterized by $k \gg 1$ can be considered suitable for kidnapping Nitrogen from Lithium. Employing equation (2) and knowing the distribution coefficient (K), it is also possible to estimate the equilibrium concentration of Nitrogen in Lithium corresponding to the saturation concentration in the getter phase ($C_{N(G)}^{\circ}$), i.e. above which the formation of a stable G_aN_b compounds would be favoured. In fact, from (2) in this condition it follows:

$$\ln(C_{N(Li)}) = \ln(C_{N(G)}^{\circ}) - \ln(K) \quad (5)$$

These minimal concentration values were calculated by Natesan [1] (Figure 2). According to this figure, one would expect for instance that V_2N formation would not allow to reduce Nitrogen concentration in Lithium

below ~ 20-100 ppm (depending on the temperature), so it could not be good for the purpose of keeping it below 10 wppm: Nb, Ti and Zr nitrides instead would be able to fulfil this target.

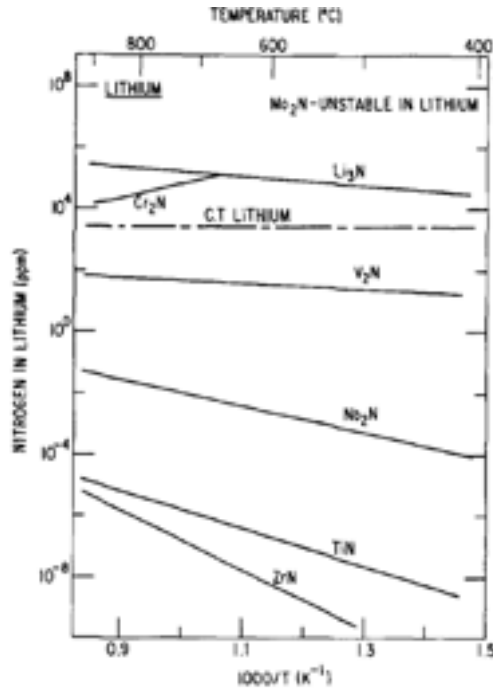


Figure 2: temperature dependence of the Nitrogen concentrations in Lithium at which some metal nitrides are stable [1]. Dashed line corresponds to Nitrogen concentration at cold trap temperature (200°C).

It must be anyway emphasized that the treatise here presented is very general and aimed only at defining the thermodynamic parameters of the process and providing a preliminary estimation of what should be expected with a specific getter material. Of course, real systems are more complex (for instance, ternary compounds may also form, like Li_9CrN_5 and Li_7VN_4), therefore a practical experimentation cannot be skipped in order to assess the real efficiency of a specific gettering solution.

2.2 Kinetic considerations about the gettering process

From the previous figures it can be seen that for those materials which have the potentiality to be selected as getter, the thermodynamic efficiency would be higher the lower is the temperature. Anyway, experimentation has shown that Nitrogen removal from Lithium increases with temperature: this fact points to the relevance of the kinetic aspect of the gettering process, being the overall rate of the process increased by the increase of temperature.

The mechanism of the gettering process is not easy to describe, because it is composed of many sequential steps. It can be schematically divided into the following steps:

- a. diffusion of Nitrogen inside the liquid Lithium phase, from its bulk to the interface with the solid getter;
- b. adsorption of Nitrogen at the solid surface of the getter;
- c. possible Nitrogen-getter interaction leading to the formation of more or less stable compounds (G_aN_b);

- d. diffusion of Nitrogen inside the getter, from the surface to the bulk, through a pure getter or a G_aN_b lattice.

To simplify the matter, we can observe that the overall rate of the process corresponds to the rate of the slower step, which is defined as *rds* (*rate determining step*).

In view of the relative rates of the previous steps, we can draw different hypothetical scenarios. If the diffusion of Nitrogen in the 2 phases was infinitely fast, as well as the adsorption/desorption at the interface, then the process would go on not differently from an homogeneous reaction, being the overall rate proportional to the concentration of Nitrogen in Lithium. Accordingly, if the gettering operation was performed in static condition (on a fixed Lithium mass), the concentration would decrease continuously and similarly the absolute rate, with a fixed rate constant. In this situation, the residual Nitrogen concentration in Lithium would decrease with a logarithmic trend (1st order kinetics). On the other hand, if Nitrogen concentration in Lithium was kept constant through a continuous replenishment (high dynamic conditions, for instance a very fast Lithium exchange at the getter surface), the amount of Nitrogen removed during time would be constant.

Actually this situation appears not realistic, a part from, perhaps, the very early stage of the process. In fact, once Nitrogen has been adsorbed at the getter surface, its diffusion inside the getter bulk can become the rate determining step, since diffusion inside a solid phase is generally a rather slow process. Since the rate with which Nitrogen passes from Lithium to the getter surface is higher than the rate with which it diffuses from this surface towards the interior of the getter, an accumulation of Nitrogen at the surface will occur, leading, in a more or less prolonged time, to its saturation.

In this condition, a purely diffusive regime is established, where the rate depends on the concentration gradient which goes with $t^{-0.5}$. Applying the two Fick's laws, it is possible to calculate the net flow of Nitrogen atoms at the interface per unit area and time (J , [$\text{mol m}^{-2} \text{s}^{-1}$]) as a function of the maximum concentration (saturation) at the surface of the getter (C_{sur}) and of the Diffusion Coefficient (D , [$\text{m}^2 \text{s}^{-1}$]) of Nitrogen inside the getter, as:

$$J = C_{\text{sur}} \sqrt{\frac{D}{\pi t}} \quad (6)$$

Through the previous expression, it is also possible to calculate the total amount of Nitrogen diffused inside the getter, $n(t)$, until a certain t value per unit area (A). Integrating equation (6) between 0 and t , we get in fact:

$$\frac{n(t)}{A} = \int_0^t J dt = 2 C_{\text{sur}} \sqrt{\frac{D t}{\pi}} \quad (7)$$

which shows that the total amount of diffused Nitrogen increases with $t^{0.5}$. Plotting therefore the diminution of Nitrogen in Lithium versus time, we'd observe, similarly, a quantity increasing with $t^{0.5}$.

Since the diffusion regime is the most likely expected after a faster initial stage of the process, it follows that the knowledge of the value of D can say much about the capability of that material to act as getter for Nitrogen. The experimentally evaluated values of D in some selected metals are reported in Table 1, together with the corresponding temperature range. Each of these values is characterized by a certain degree of uncertainty, resulting from many different experimentations reported in literature: anyway,

being the differences in the D values very large among the different metals (order of magnitudes), the uncertainties in the D value of a specific metal leave unchanged its relative position in the getter attitude scale. D values for many other metals and their dependences on temperature can be found in [11].

Table 1: approximate values of D for diffusion of Nitrogen in selected metals. The minimal D value is associated to the lower temperature, the maximum to the higher one. The range of D values includes also the experimental uncertainties

Metal	T [°C]	D [cm ² s ⁻¹]	Reference
Ti (hcp)	450-700	~ (10 ⁻¹⁷ ÷ 10 ⁻¹³)	[12]
Zr (hcp)	500-700	~ (3 x 10 ⁻¹⁷ ÷ 6 x 10 ⁻¹⁴)	[11]
V (bcc)	450-700	~ (5 x 10 ⁻¹³ ÷ 5 x 10 ⁻¹⁰)	[13]
Nb (bcc)	450-700	~ (2 x 10 ⁻¹³ ÷ 5 x 10 ⁻¹¹)	[13]
Ta (bcc)	450-700	~ (10 ⁻¹⁴ ÷ 10 ⁻¹¹)	[14]
Cr (bcc)	450-700	~ (10 ⁻¹³ ÷ 5 x 10 ⁻¹¹)	[11]
Fe (bcc)	200-700	~ (5 x 10 ⁻¹² ÷ 5 x 10 ⁻⁷)	[15,16]

From the table, it can be seen that Ti and Zr, which were shown by Figure 2 as the best getter candidates on the basis of the mere thermodynamics, are characterized unfortunately by the smallest diffusion coefficients. This is probably due to the hcp (*hexagonal close packed*) lattice structure they assume in their stable phase in that temperature range, which is more packed (higher atomic density) than the bcc (*body-centered cubic*) structure, peculiar instead to the other table metals. The value of Iron coefficient is the highest of all of them: even at 200°C (just above Lithium melting point), it results higher than Ti's and Zr's ones at 700°C; unfortunately, its compounds with Nitrogen are not thermodynamically stable.

Regarding the effective value of D, it must be anyway underlined that it could not be perfectly constant with time, since, as long as the diffusion process goes on and Nitrogen concentration inside the getter increases, a G_aN_b phase forms, in which the diffusion may differ (generally becoming slower) from that in pure G: this makes more difficult to estimate in advance the required time for the diffusion.

In conclusion of these theoretical sections, a specific material behaving 'very well' from both the viewpoint of the thermodynamics and the kinetics is not detected and probably a compromise between the two aspects would be found.

2.3 Other general considerations on the choice of the getter

A part from the previous aspects, also the following ones are to be considered in the selection of a proper getter:

- the format of the getter (for example: foil, pellets, sponge...). Getter of the same materials but with different formats are evidently characterized by different values of surface area per unit mass. A larger surface area is surely to be preferred from the kinetic point of view; anyway, a very finely dispersed getter could be more difficult to handle;
- the getter-Lithium phase diagram. Clearly, the getter must not suffer corrosion damages by liquid Lithium at the gettering temperatures. Its solubility in Lithium must be very limited and similarly the solubility of Lithium in it, so that liquid Lithium and solid getter remain as two separate phases;

- the cost of the getter. When passing from lab experimentation to large scale plants, the total amount of necessary getter can become huge. In this condition the total cost of the getter could become very high as well and put that getter outside the possibility of a real use.

2.4 *Gettering experiments in literature*

This section separately presents the main conclusions on the gettering behaviour of many of the aforementioned metals, as derived from the experimentation performed since the last decades by several research groups. It must be emphasized that this experimentation is far from being exhaustive, because the employed test conditions were often not homogeneous, therefore a direct comparison is not easily feasible. Anyway, some features of each getter can be highlighted and can indicate if and how its experimentation deserves to be continued (possibly improved) or not.

Most of the activity in this field was conducted in the framework of the EVEDA phase of the IFMIF project and focused on the employment of Titanium or Titanium alloys.

2.4.1 *Chromium*

The interaction of Chromium with Nitrogen dissolved in liquid Lithium has been subject of relevant investigation in the past, not only with the goal of employing it as getter, but mainly to characterize the corrosion resistance of many structural materials to Lithium, being Chromium an almost ubiquitous element in steels composition.

In the experiments it was shown that Chromium was somehow able to act as getter for Nitrogen, despite the low stability of its binary compounds with Nitrogen (Figures 1 and 2): this was ascribed to the formation of a ternary, more stable, compound (Li_9CrN_5). A recent thermodynamic analysis [17] calculated anyway that this compound could assure a good gettering efficiency only at rather high Nitrogen concentrations, while in case the target concentration was only 10 wppm, it would require very low temperatures, close to Lithium melting point, where on the other hand the kinetics of the process is expected to be depressed. At 550°C, starting from 115 wppm of Nitrogen and after 19 days of immersion, the lowest experimental concentration value achieved with Chromium was indeed 65 wppm [18], which is aligned with the theoretical prevision [17] at that temperature.

Chromium could hence be useful to reduce high Nitrogen concentrations in Lithium, but not down to 10 wppm, which is for instance IFMIF-DONES requirement. For this reason, it is believed that it doesn't deserve to be included among the best getters for Nitrogen.

2.4.2 *Vanadium*

Few experimental data are reported in literature on the possible employment of pure Vanadium as getter for Nitrogen. Also for V the formation of a ternary compound (Li_7VN_4) was proven [19], but it would be stable only above very high Nitrogen concentration (~ 23000 wppm at 750K), therefore it couldn't improve the gettering capability in our working range of concentration values.

For what concerns the binary nitride, VN, in theory the critical Nitrogen concentration for its formation should be well below 1 wppm. Anyway, when Vanadium plates ($1 \times 10 \times 50 \text{ mm}^3$) were immersed at 400-500-550°C in 12 or 25 g of liquid Lithium containing 100 wppm of Nitrogen [20], after about 1 month the maximum Nitrogen uptake was (indirectly) calculated as only ~ 5 wppm, translating into a residual concentration in Lithium ~ 95 wppm.

The reason of such a disappointing result was not really clarified; in any case, since in the same experiments [20] pure Titanium and V-Ti alloys plates demonstrated to capture more Nitrogen from Lithium, it is not

convenient to invest additional time on Vanadium, but it's preferable instead to focus on Titanium and its alloys (see next subsections).

2.4.3 Titanium

Titanium is surely the most investigated getter for Nitrogen, because of the high stability of the Ti-N compound (see Figures 1-2).

In view of this potentiality, Titanium was selected for instance as getter for Nitrogen in the dedicated Hot Trap of the ELS (Experimental Lithium System)[21]. In a first test, the Hot Trap (operated at 500°C in dynamic conditions and filled with a Titanium foil) was shown to remove 195 wppm of Nitrogen (from 230 to 35 wppm) from a Lithium inventory of 108 gal in 1 week operation; anyway, in the next tests, after replenishing Lithium with Nitrogen (to ~ 400-450 wppm), the same used getter was able to remove no Nitrogen more, after ~ 1 day of operation at 500-550°C. This fact confirmed the kinetic (diffusion) limit of the Titanium, whose gettering efficiency became smaller and smaller with time, approaching the final apparent value of 0. The employment of Titanium would ask therefore for a higher operative temperature, from one side, and from the other for the maximum possible external surface, so to work as long as possible far from the surface saturation condition.

The experimental investigations of the employed Titanium getters after their immersion in liquid Lithium at 500-600°C, confirmed in any case the penetration of Nitrogen, leading to the formation of various Ti-N phases. In one experiment performed at 500°C [22], the penetration seemed limited to about half of a micron (after 27 hours), with a concentration profile decreasing with the depth according to a diffusion model; in this experiment, the formation of the TiN phase was not observed. In a different experiment at 550°C [20], the author reported instead that Nitrogen was incorporated forming Titanium nitrides (mostly TiN, but with a minor presence of Ti₂N), rather than a solid solution of Nitrogen inside the metal: even after a long gettering time (more than a month), the penetration of Nitrogen appeared limited anyway to a layer ≤ 2 μm thick below the surface, highlighting the small mobility of the non metal inside the Titanium lattice. Finally, in an other experiment entailing 27 hours immersion in Lithium at 600°C [22], the intrusion of Nitrogen seemed to reach a maximum depth of 4 μm and led to a maximum Ti/N stoichiometric ratio ~ 1, corresponding to the TiN phase (Figure 3); Figure 4 reports O, N and Ti concentration profile (at%) along the getter depth, as evaluated by the EDS analysis of a specific portion of Figure 3. Tentatively putting together these different authors' results, it would seem that the maximum thickness of the penetration increased with temperature (from ~ half of a μm at 500°C, to ~ 2 μm at 550°C and then to ~ 4 μm at 600°C) and that was instead less affected by the duration of the gettering experiment. Therefore, once the penetration limit is reached, to overcome it would be more useful to raise the temperature instead of letting the getter in contact with Lithium for additional time.

Figure 3 shows nonetheless also the presence of some cracks; moreover, in a different experiment at 500°C, the author [22] found an apparent disappearance of Nitrogen at the end of the gettering experiment, in that the residual Nitrogen solved in Lithium plus the one quantified in Titanium according to its composition profile resulted less than the starting Nitrogen in Lithium, and justified this fact making the hypothesis that Lithium exerted a corrosion action on the Titanium-nitride formed at the solid surface, leading to the exfoliation of 1-10 μm size TiN corpuscles (not detectable by the chemical analysis [23]). This possible instability of Titanium in Lithium at high temperature will surely require additional investigation: if confirmed, it could pose a serious limit to the employment of Titanium as getter for Nitrogen.



Figure 3: TEM image (Focused Ion Beam) of the Ti thin foil cross section after 27 h immersion at 600°C.

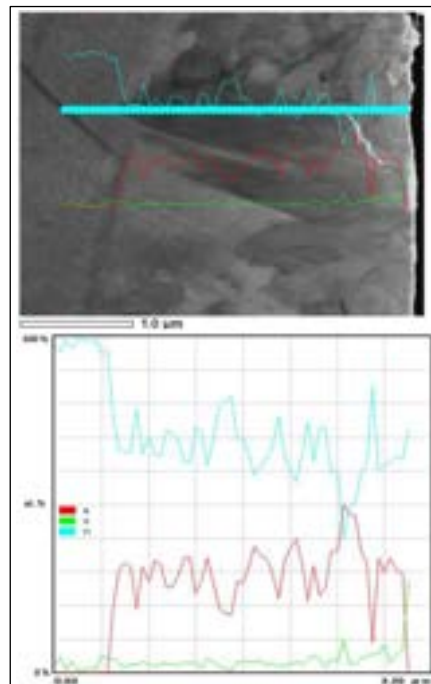


Figure 4: EDS concentration profiles of N, O and Ti along the depth of a portion of the Ti sheet immersed in Li at 600°C for 27 h.

The most interesting results achieved with a Titanium getter, in terms of Nitrogen reduction in Lithium, were obtained by ENEA in the framework of the IFMIF/EVEDA project [24-27]. In these experiments, in order to dispose of the maximum getter surface extension to delay the saturation condition, Titanium was selected not as dense material, but as a sponge (commercial *Sigma Aldrich* product - code 268526). The main features of this material, as declared by the supplier or resulting from ENEA characterization, are the

following: purity equal to 99.5%; format of chunks (2-12 mm); apparent density $\sim 0.54 \text{ g cm}^{-3}$, with a corresponding void factor of $\sim 88\%$ (due to both channels and pores inside each chunk and to the packing of the many chunks in the space); Specific Surface Area (SSA): $4.68 \text{ m}^2/\text{g}$ (this results, achieved with the Mercury Intrusion Porosimetry technique, is not exact, because many approximations are introduced in the calculation, anyway it can give at least an idea of the order of magnitude of the overall available surface, conservatively $\sim 1 \text{ m}^2/\text{g}$).

Approximately 8.2 Kg of this getter were put in contact with liquid Lithium inside the Hot Trap of the Lifus 6 plant [24, 28]; the trap was placed outside the main Lithium loop and operated in static condition on the whole Lithium inventory, $\sim 9.8 \text{ Kg}$. Considering the SSA value of the getter, the total getter surface available for the Nitrogen adsorption results hence of the order of 10^4 m^2 .

Let's look now at the experimental results got in 2015-2016. It must be underlined that a proper characterization of the Hot Trap efficiency was not possible, due to the small number of performed purification steps and also to the rather small measured concentration variations. Lifus 6 Lithium appeared in fact to be already a 'good quality' Lithium, being its starting Nitrogen concentration, before any purification activity, on the order of only some tens of wppm. Hot Trap purification operations were performed initially at 550°C , then at 600°C (with the same, already used, getter). Table 2 reports the values of Nitrogen concentration in Lithium resulting from the Hot Trap action at 550°C ; Table 3 refers instead to the operations at 600°C .

Table 2: summary of the Nitrogen concentration variations resulting from the Hot Trap action at 550°C .

Total purification time [hours]	Measured Nitrogen concentration [wppm]
0	$55.1 \pm 9.0 \text{ wppm}^2$
30	$25.6 \pm 2.7 \text{ wppm}$
411	$31.9 \pm 5.3 \text{ wppm}$

Table 3: summary of the Nitrogen concentration variations resulting from the Hot Trap action at 600°C .

First operation	
Purification time [hours]	Measured Nitrogen concentration [wppm]
0	$123.0 \pm 17.2 \text{ wppm}$
139	$35.7 \pm 6.3 \text{ wppm}$
Second operation	
Purification time [hours]	Measured Nitrogen concentration [wppm]
0	$56.0 \pm 5.9 \text{ wppm}$
169	$14.1 \pm 2.1 \text{ wppm}$

² The uncertainty reported in these values follows from both the random (each analysis has been generally executed twice) and the systematic errors (estimated as $\pm 2.5\%$) [23]

From the above tables it's clear that the Hot Trap played a positive role in reducing Nitrogen concentration in Lithium. After the first 30 hours of purification at 550°C, Nitrogen concentration was reduced in fact by 29.5 wppm, which, taking into account the Lithium mass, corresponds to the removal of ~ 300 mg of Nitrogen; it seems, on the other hand, that no additional reduction of Nitrogen was gained during the next purification step (381 hours more). In a first operation at 600°C, Nitrogen concentration was instead reduced from 123.0 wppm to 35.7 wppm in 139 hours, with a corresponding removal of about 890 mg of Nitrogen. It was therefore demonstrated that raising the operative temperature allowed the Hot Trap to rescue its gettering function, that seemed lost or momentarily shelved at the last stage of 550°C operations. In a following purification step at 600°C, Nitrogen concentration was instead reduced from a starting value of 56.0 wppm to 14.1 wppm in 169 hours (~ 1 week), with a corresponding mass removal of ~ 427 mg of Nitrogen. It is not this quantity anyway the most relevant result, but the final achieved concentration value. Such a low concentration value (14.1 wppm) in fact had never been reported in literature with Titanium getter and it is one of the lowest values ever achieved in all worldwide experimentations, even with different getters; moreover, it cannot be excluded that, allowing a longer purification time, it could have become even smaller.

The good achieved results may reasonably be ascribed to the large value of the getter SSA (~ 1 m²/g). If, for ease, we assume that all the adsorbed Nitrogen leads to the formation of TiN, characterized by a density of 5.22 g cm⁻³, and calculate the amount of Nitrogen kept as TiN in just 1 nm layer at the surface, the corresponding volume in such a layer would be (10⁴ cm²/g * 10⁻⁷ cm) = 10⁻³ cm³ per g of getter, i.e. 5.22 mg of TiN per g of getter, corresponding in turn to 1.18 mg of N per g of getter, a very high value (in the case of Lifus 6 results, only ~ 1.37 Kg of getter would have been enough to remove all that Nitrogen, far below the 8.2 Kg employed). On the other hand, 1 nm corresponds to the thickness of just few atomic layers and its filling would not become limiting from a kinetic point of view.

Therefore, if the existence of a thickness limit for the Nitrogen penetration inside Titanium (depending on temperature) has been evidenced, from a practical point of view this weakness can be overcome if the penetrated thickness is multiplied by a so large getter area to produce however a very large volume, capable of housing all the necessary Nitrogen (the advantage of a large surface area holds of course for any kind of getter, not only for Titanium).

In view of the good experimental results, it is believed that Titanium surely deserves to be included among the possible getter candidates for future fusion plants. Not necessarily the commercial product from *Sigma-Aldrich* should be considered: any other format or product could be good for the purpose, provided it's characterized by a very large surface area. Of course, the possibility of TiN being attacked by liquid Lithium at high temperature, which was hypothesized in one experimentation, should be investigated again and necessarily excluded in those operative conditions required for an efficient Nitrogen removal.

2.4.4 Vanadium-Titanium alloys

Metallic alloys could be interesting as gettering materials inasmuch they could present the advantages of all the constituting metals. One of the alloy element should be characterized by the possibility of establish a strong interaction with the captured Nitrogen, i.e. the formation of a thermodynamically stable compound; an other alloys element should instead assure an 'open' lattice structure, i.e. the good mobility of Nitrogen through the alloy. Having verified that Titanium satisfies the thermodynamic requirement of a strong interaction with Nitrogen, but it could be somehow limited by a rather small Nitrogen mobility inside its lattice, it was considered that a possible enhancement of its efficiency could be obtained by binding it with other metallic elements, characterized by larger diffusion coefficients. In this section V-Ti alloys are discussed, reporting the experiments performed by [18, 20].

V-5at.%Ti and V-10at.%Ti alloys plates (0.6x10x40 mm³), both realized by a plasma jet-melting furnace, were investigated by immersing them for 33 days in liquid Lithium, containing 55 wppm of Nitrogen, at

550°C. Weight and Nitrogen concentration inside the plates increased after the immersion, especially for V-10at.%Ti. In particular, analyzing in depth this alloy getter, the author concluded that Nitrogen was incorporated down to a depth of about 8 μm: in a first layer, 0.1 μm thick, Nitrogen existed mostly as TiN and VN; the second, lower layer, was characterized instead by the presence of a V-Ti-N solid solution. The improvement of this alloys respect to pure Titanium appears from the comparison with the results achieved by the same author in a twin experiment, in which the penetration of Nitrogen in a Ti plate appeared limited to 2 μm (already mentioned in the previous section, [20]).

The potentiality of the V-10at.%Ti getter was later investigated by directly measuring the real variation of Nitrogen concentration in Lithium. In this test, 3 getter plates (total surface ~ 24 cm²) were immersed for about 5 days at 550°C in 25 g of Lithium containing 370 wppm of Nitrogen. Lithium was analysed during the experiment for its Nitrogen concentration, which decreased according to the square root of the immersion time (Figure 5).

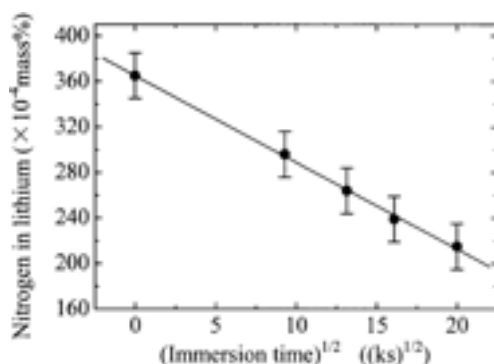


Figure 5: change of Nitrogen concentration in Lithium vs immersion time [20] employing V-10at%Ti as getter

Unfortunately the experiment was stopped after only 5 days; in case the Nitrogen decreasing trend remained constant ($-7.5 \text{ wppm (ks)}^{-0.5}$), the target of a residual 10 wppm concentration would have been achieved after 27 total days. Of course this simple extrapolation is not correct and only a future, additional experimentation focused on the low concentration ranges could confirm or not an efficiency like this.

On the basis of the analyzed experimentation, it is believed that V-Ti alloys deserve to be included among the possible getter candidates for Nitrogen.

2.4.5 Iron-Titanium alloys

The potentiality of Iron as alloying element for Titanium relies on the very high diffusion coefficient of Nitrogen inside it. If we look at Table 1 values, we see that diffusion coefficient in Iron is about 3 order of magnitude higher than in Vanadium at the same temperature and it's even 6-7 order of magnitude higher than in Titanium. Anyway, as already reported, Iron-Nitrogen compounds are not stable therefore a practical employment of pure Iron as getter cannot be feasible: Iron can hence find a useful application if bound with Titanium into alloys. Before going on with the results of the Fe-Ti alloys experimentation, let's look at the Fe-Ti phase diagram, shown in Figure 6.

According to the figure, the solubility of Titanium in Iron is poor. In the range of temperatures commonly considered for the gettering process (500-600°C), Titanium exists in a solid solution of α-Iron (bcc) only up to a content of ~ 1 at. %, while for higher Titanium contents part of this metal segregates leading to the formation of the Fe₂Ti intermetallic compound, characterized by an hexagonal (C14) structure (similar to

MgZn₂][29]. Since this compound is fragile, a maximum 10% atomic content of Titanium was considered in most of the Fe-Ti alloys employed in gettering experiments.

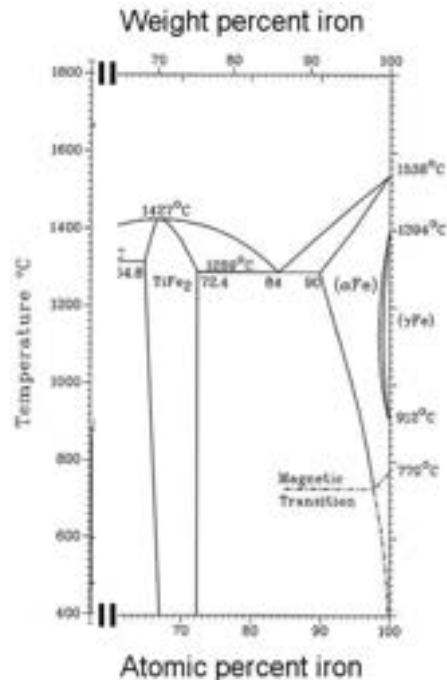


Figure 6: Iron-Titanium phase diagram [30]

First investigations of the gettering capability of the Fe-Ti alloys were performed in 2005 and 2006 [31-32]. In these studies 3 plates (total surface extension ~ 24 cm²) of Fe-5at.%Ti or Fe-10at.%Ti (both obtained by the plasma jet-melting furnace) were immersed in 25g of Lithium at 600°C; the starting concentration of Nitrogen in Lithium varied, depending on the specific test, from a maximum of ~ 300 to a minimum of ~ 90 wppm. Nitrogen concentration decrease was evident in all the tests, even if, after few days of immersion, it seemed in any case to stop, and a final value lower than 30 wppm was registered in no test. No clear difference in the efficiency of the 2 alloys was evidenced by the ensemble of all the experimental results.

The experimental investigation of the solid alloys after the tests indicated as TiN the main existence state of Nitrogen in these getter; in the Fe-10at.%Ti alloys, a decrease of the Fe₂Ti compound was in parallel noticed, therefore it was thought that in this alloys TiN originated mainly from Fe₂Ti. In the same alloy, TiN was prevalently localized in a 1-4 μm depth below the surface, while its presence in the 0-1 μm layer was less important. According to the author, the stop of the Nitrogen absorption observed after some days of immersion was not due hence to the coverage of the getter surface by Nitrogen, but to a possible diffusion barrier located some micron below the surface.

The Iron-Titanium alloys were largely investigated also by the Japanese partners of the IFMIF project, as reported in the related Deliverables [22, 30, 33-35]. In the 2009-2010 experiments [33], Fe-5at.%Ti and Fe-10at.%Ti alloys getters were fabricated with the centrifugal atomizer method as pebbles characterized by very small grain dimensions (from 10 to several hundreds microns) and by super-solutions of Titanium in α-Iron: in the fresh Fe-5at.%Ti the presence of Fe₂Ti was not revealed at all by SEM, while in Fe-10at.%Ti the largest fraction of Ti was however in the Fe₂Ti phase.

In the gettering experiments, 2.3 g of these alloy pebbles (selected by diameter in the range of 180-212 μm ; corresponding total external surface: 100 cm^2) were immersed at 600°C in 50 g of Lithium, whose starting Nitrogen concentration varied, from test to test, from ~ 300 to ~ 450 wppm. The outcome of these experiments indicated:

- a better gettering efficiency for the alloy containing more Ti (minimum achieved Nitrogen concentration: ~ 50 wppm, after ~ 330 hours);
- no significant difference in the rate of removal of Nitrogen between static and dynamic (rotating stirrer) conditions, indicating that it was not the diffusion of Nitrogen inside the alkaline metal the rate limiting factor;
- Fe_2Ti phase was the most responsible for the taking of Nitrogen (for the the alloy containing 10% of Ti it was estimated about 10 times higher than by the α -Iron solution), even if diffusion inside Fe_2Ti seemed to be slower than through the α -Iron solution: the best thing would be hence to dispose of a finely dispersion of the Fe_2Ti phase inside the Iron lattice;
- the getter surface was not saturated yet at the end of the test and the registered gettering rate decrease had been actually caused by the reduced Nitrogen concentration gradient at the Li-getter interface, more than by the diffusion rate inside the getter.

[22] focused instead on the application of Fe-5at.%Ti as getter for Nitrogen in a low concentration regime. In the reported experiment, 22g of Fe-5at.%Ti pebbles (diameter ~ 160 μm ; estimated total surface ~ 0.1 m^2) were immersed in 76 g of Lithium at 500°C. Nitrogen concentration was reduced from its starting value of 65 wppm to 25 wppm after 48 hours, then to 9.9 wppm after additional 96 hours of immersion (total time: 144 hours). This last result deserves attention, since for the first time a final Nitrogen concentration < 10 wppm was experimentally achieved, able to satisfy the strictest purity requirements. Probably, the good result could be ascribed to the higher getter extension (~ 0.1 m^2) than in the previously reported experiments (0.01 and 0.0024 m^2 respectively for [33] and [31-32]). Clearly, the starting concentration value was already rather low, therefore a simple extrapolation to higher concentration values it is not possible, in view of the risk of saturation and/or change of the prevalent kinetic step (rds) if much more Nitrogen had to be captured from the getter.

In any case, Fe-Ti alloys surely deserve to be considered among the getter candidates for Nitrogen.

3 Conclusions

At the end of this discussion, a sure, final decision about the best getter for Nitrogen solved in liquid Lithium cannot however be taken. The experimental conditions adopted for the different getters in the many tests performed by the many authors were in fact often non homogeneous, in that the operative temperature, the duration of the test, the total Lithium amount and its starting Nitrogen concentration (hence the total mass of Nitrogen to capture), the getter mass and its format (hence its external surface) varied from experiment to experiment: a direct comparison of all the achieved results is not possible.

As a general feature, it can anyway be asserted that the higher the getter surface extension (hence the Lithium-getter interface), the higher the efficiency of the getter, since more free sites will be available at the surface for the interaction with Nitrogen: this will enhance the rate of the adsorption process, as well as delay the reaching of a complete surface saturation condition. In this regard, the format of many small particles or of a sponge would be preferable to large dimensions getter, even bidimensional like foils.

Regarding the specific studies of the getters reported in this document, it can be concluded that pure Iron, pure Chromium and pure Vanadium do not appear interesting materials. The following materials showed instead a good behaviour:

A – Titanium: it was the only pure element material to demonstrate a sound gettering efficiency. The high thermodynamic stability of its compounds with Nitrogen makes it suitable for both high and low Nitrogen concentrations in Lithium, even if the slow Nitrogen diffusion inside its lattice could limit its employment to high temperature and very large surface extension. A warning was put anyway on the employment of Titanium in relation to a possible corrosion action exerted on it by liquid Lithium at high temperatures: future investigation is asked to confirm or belie this phenomenon, which could seriously limit the utilization of this material.

B – Vanadium-Titanium (10% at) alloy: in a comparative test, V-Ti alloys showed a more pronounced Nitrogen absorption than Titanium itself, particularly the alloy with 10 % at. of Titanium. Few experimental data are anyway available at the moment.

C – Iron-Titanium (10% at) alloy: in one experiment, one of these alloys was reported to lead to the lowest (in absolute) registered Nitrogen concentration (9.9 wppm). Respect to pure Titanium, this family of alloys has the advantage of a faster diffusion of Nitrogen inside the getter lattice (resembling Fe one), which makes more easily available for the interaction the Titanium atoms located deep inside the getter. The limits to their employment could be instead the need for a high concentration gradient at the Lithium-getter interface and the large required getter amount: for each 'active' Titanium in fact, 10 to 20 atoms of Iron are present in the alloy, determining a mass (and a volume) approximatively 10 to 20 times higher than pure Titanium one. On the whole, the Fe-10at.%Ti is expected to bring to better gettering results than Fe-5at.%Ti; anyway the specific fabrication procedure of the alloy could affect the efficiency of this kind of getters more than the exact composition value.

D – Niobium: this metal has never been tested until now as getter for Nitrogen, but, on the basis of the theoretical screening reported in sections 2.1 and 2.2 of this document, it could exhibit an interesting getter attitude, constituting a good compromise between the thermodynamics of the interaction with Nitrogen and the kinetics of the Nitrogen diffusion. Niobium could anyway suffer from a corrosive attack by liquid Lithium, being its estimated solubility in Lithium a little bigger than Titanium one [36-37] and being also susceptible to grain-boundary penetration by liquid Lithium, in case of a starting Oxygen concentration in the transition metal exceeding ~ 400 ppm [38]. For this reason, probably Niobium utilization should be limited to rather low temperatures ($\leq 500^\circ\text{C}$?). Also the cost of this metal (which is surely more expensive than Titanium) could be an issue, especially when conceived for a large scale plant.

The above getter candidates should be submitted to comparative tests, in which the experimental conditions should be the same for all of them. In a possible grid of tests, at least two different temperatures should be selected: a low one ($450\div 500^\circ\text{C}$) and a high one ($550\div 600^\circ\text{C}$). Similarly, two different starting values of Nitrogen concentration in Lithium should be investigated: a 'dirty' Lithium one ($500\div 1000$ wppm) and a 'clean' Lithium one ($100\div 200$ wppm). This way, it could be possible to point out which are the best operative settings for each candidate getter and which getter, at the end, could be considered the best one in absolute sense. Of course, if possible, it will be preferable to select the getter characterized by good efficiency even at low temperature, reducing the risk of the corrosive attack by Lithium and the general safety hazard associated to the employment of the alkaline metal.

This experimental activity is going to start in the next months in ENEA laboratories.

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5 Acronyms

a_x	Activity of the X element
A	Surface Area
C_x	Concentration of the X element
C_x°	Saturation concentration of the X element
C_{sur}	Concentration at the surface
D	Diffusion Coefficient
D_c	Activities Distribution Coefficient
DONES	Demo Oriented Neutron Source
ΔG	Free Energy Variation
ΔG_f°	Free Energy of Formation (in standard condition)
EVEDA	Engineering Validation and Engineering Design Activities
IFMIF	International Fusion Materials Irradiation Facility
J	Flow [$\text{mol m}^{-2} \text{s}^{-1}$]
K	Concentrations Distribution Coefficient
μ	Chemical potential
SSA	Specific Surface Area [$\text{m}^2 \text{g}^{-1}$]
T	Temperature
t	Time
wppm	Weight Parts per Million