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# Review of Tritium Retention in First Wall Materials

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## **ABSTRACT**

The hydrogen retention properties of first wall materials used in existing tokamaks is briefly reviewed. Estimates are made of the hydrogen retention for the ITER first wall, for which vanadium-based alloys are a candidate material.

## **1. INTRODUCTION**

For a fusion reactor, the extent of tritium loss from the fuel cycle through the build-up of the tritium inventory in first wall components will depend upon material selection. The first wall of early fusion experiments was typically little more than the stainless steel vacuum vessel, while in later machines the effort to eliminate high-Z plasma impurities resulted in a first wall covered with low-Z material, typically graphite. This step in the evolution of the first wall resulted in completely different hydrogen retention behaviour.

Actual fusion reactors will require first wall materials which can cope with the expected high neutron and heat fluxes. The incorporation of these new materials will again alter the hydrogen retention character of the vessel. Low activation vanadium alloys may result in enhanced tritium retention since vanadium has a high hydrogen solubility, absorbs hydrogen exothermically from the gas phase, and has a very high diffusion coefficient for hydrogen.

This paper reviews the tritium retention properties of materials previously used in tokamaks as well as addressing those materials being considered for first wall applications in D-T burning machines. The first two sections deal with stainless steel and incinel and carbon. These materials are the main constituents of present day tokamaks. Beryllium, a material which has seen some application in tokamaks, is briefly discussed. The final section deals with vanadium since vanadium-based alloys are candidate materials for the next generation of tokamak.

## **2. STAINLESS STEEL AND INCONEL**

These alloys tend to be used for ultrahigh vacuum applications because of their chemically stable surfaces which lead to good outgassing properties. With regards to hydrogen they are endothermic occluders, that is, hydrogen atoms in solution in the metal have a higher potential energy than when bound together as

molecules in the gas phase. This tends to result in fairly low bulk concentrations; typical maximum concentrations for a stainless steel tokamak would be  $<1$  H atom for every  $10^4$  metal atoms [1].

To reach these concentrations in an endothermic metal relies upon wall pumping, that is the rapid uptake of hydrogen by a metal when the hydrogen arrives as ions or atoms. In this chemical form hydrogen sees almost no adsorption barrier at the surface. However, to be released atoms must overcome a barrier for recombination into molecules [2].

The wall pumping effect tends to be transient: during a discharge atoms are consumed by the walls but when the discharge ends the hydrogen is released back into the vacuum vessel [3].

The above wall pumping and release effect has been described by computer codes such as PERI [4] and DIFFUSE [5] which solve the diffusion equation with the quadratic boundary condition for recombination. The DIFFUSE code also considers the effects of traps which may be present due to bombardment by energetic particles. The weakness of these codes, however, is that the results depend on the value of the rate coefficients used. The recombination coefficient  $K_r$ , tends to be a quantity which is extremely sensitive to the exact state of the surface; increasing surface coverage by impurities tends to decrease  $K_r$  in general [2, 6-8].

Finally, because hydrogen atoms are fairly mobile in these metals, especially at high temperature ( $T \geq 250^\circ\text{C}$ ), it is possible that significant quantities of hydrogen can permeate through material. This is an important issue for tritium systems where tritium can escape the containment provided by the vacuum vessel into the external atmosphere or coolant. The hydrogen diffusivities and solubilities for stainless steel and inconel are shown in Fig. 1 and Fig. 2 respectively, compared to other first wall materials.

### **3. GRAPHITE**

Graphite has a very different retention behaviour compared to metals. The hydrogen is relatively immobile, the diffusivity becoming significant only at high temperatures ( $>1000\text{K}$ ). The concentrations of hydrogen in graphite, for hydrogen fluences relevant to tokamaks, are very much higher than for stainless steel,

approaching  $\sim 0.5$  H/C. This is not due to hydrogen in solution but instead it is the result of trapping [9].

Measurements of the diffusion of hydrogen in graphite vary over a wide range, but generally show a strong temperature dependence (fig. 1) [10]. One reason for the spread is certainly that of the different types of graphite, with different porosity and grain sizes. Another reason for the spread is that because of trapping, the transport of hydrogen is not simply a diffusion problem; the "apparent" diffusion coefficient will depend on concentration.

Graphite in tokamaks leads to a smaller dynamic retention capacity as compared to metal walled tokamaks [11]. A graphite first wall will pump when the graphite has been depleted of hydrogen, say by helium glow discharge cleaning, but it quickly fills to saturation when exposed to a hydrogen flux, and then it ceases to pump [12]. Furthermore, the outgassing after the discharge is slow, so that the wall pump does not "regenerate" itself.

It should be noted, however, that when graphite is at elevated temperatures, or if the graphite is chemically active (by boron or beryllium) it is possible to get the transient wall pumping effect. This is because the higher temperature, and reduced potential energy barrier resulting from Be or B doping, speed up the hydrogen outgassing.

During discharges, the main mechanism for hydrogen release from the graphite is sputtering. This leads to density control problems when increasing the energy content of the plasma by injecting power; this leads to higher conducted power loads to the walls which then release trapped hydrogen into the plasma [13]. The hydrogen inventory in a carbon lined tokamak which is available for release by sputtering can be many times higher than the hydrogen content of a typical plasma.

Finally, the most important mechanism for the long-term hydrogen retention in graphite is codeposition [14]. This refers to the process where hydrogen and carbon atoms incident on a substrate (which need not be a carbon substrate) grow to form a H-saturated carbon film. This process is seen to occur in tokamaks where heat and particle loads cause net erosion of carbon in areas in contact with the plasma and net deposition in neighbouring areas. The important feature of codeposition is that there is no limit to the film thickness, so that hydrogen can

get buried in films many microns thick: much too deep to be released by sputtering. Among the materials considered for first wall applications, the codeposition process is peculiar to carbon (and carbon based materials). The tritium inventory which could build up in codeposited films is a major drawback of using graphite in a fusion reactor.

There are many carbon based materials such as carbon/carbon-fibre composites and boron doped graphites which have in general the same tritium retention behaviour as pure graphites and hence will not be discussed separately here.

#### **4. BERYLLIUM**

Experimental data on hydrogen retention for beryllium is not as extensive as for graphite. Measurements of diffusivity [15] are hampered by relatively impervious beryllium oxide, but never-the-less indicate a small diffusion coefficient as compared to transition metals (fig. 1). Measurements of H solubility are scarce, and although the temperature dependence varies between investigations, the magnitude of the solubility appears to be small (fig. 2).

It is not clear whether beryllium can be treated like a metal, in the way described in the early section on stainless steel. Because the diffusivity and permeability are so low, the effects of trapping may play a more important roll in determining the retention.

Annealing of samples implanted with H indicate two distinct trapping levels, both much weaker than for graphite [16,17].

In the JET tokamak, when beryllium was introduced as a first wall material, the wall pumping during discharges increased dramatically [11]. A substantial fraction of the gas pumped out during the discharge was released between discharges. In this sense, the behaviour was reminiscent of an all metal (inconel or stainless steel) tokamak.

This does not mean, however, that the processes involved in hydrogen retention in beryllium are the same as those in stainless steel. Firstly, the walls of JET can hardly be called beryllium. Post-mortem analysis of tiles show a mixture of roughly equal amounts of Be in C covering all surfaces. The C/Be material also has very large D concentrations in samples removed from the torus, i.e.



resembling the case for carbon more than metals [18]. Secondly, if the carbon plus beryllium has much smaller trapping energies than pure carbon, hydrogen would be released in significant quantities between tokamak discharges, as is the case in an all-metal tokamak.

## **5. MOLYBDENUM AND TUNGSTEN**

Molybdenum and Tungsten have been considered for first wall application on the basis of their high melting point and resistance to sputtering [19]. They are mainly being considered as divertor target plate material.

The main disadvantage is the high atomic number; only a small amount of metal atoms in the main plasma lead to unacceptable levels of radiated power. Early use of these materials in tokamaks suffered from this, nevertheless these materials are being reconsidered for first wall applications. The challenge will be to limit the local plasma temperature to a few eV to suppress sputtering. It is also necessary to limit the concentrations of ions of intermediate Z (e.g. O,C) which strike the target since these sputter tungsten much more efficiently than deuterium does.

The data on diffusivity and solubility of hydrogen in Mo and W (fig. 1 & 2) is more extensive than for Be, but there is still a wide variation in the results from different studies [10]. This is attributed to the different crystal and grain structure of samples between investigations. Since the diffusion within grains is relatively slow, grain boundaries can form short-circuit paths for H transport.

Although the H solubility in these metals is very low, large amounts of hydrogen can be loaded into the near surface region by implantation. This is due to ion induced damage which creates deep traps for the hydrogen.

## **6. VANADIUM-BASED ALLOY**

Vanadium based alloys are being considered for ITER, in particular the alloy V-5G-5Ti, because of their superior radiation resistance, thermal conductivity and good mechanical propertire.

## 6.1 Hydrogen absorption properties

Vanadium belongs to the class of metals which dissolve hydrogen exothermically. Consequently large quantities of hydrogen can be absorbed in the metal when exposed to modest external gas pressures (e.g. for vanadium at 150°C exposed to H<sub>2</sub> at 1.33 Pa has an equilibrium concentration of ~ 0.02 H/V [20]). Furthermore, if the temperature is sufficiently low (e.g. <180°C for H in V) a metal hydride phase can form, a process which must be avoided since metal hydrides are extremely brittle, and their formation is associated with a large volume increase.

Addition of small amounts of alloying elements can greatly affect the equilibrium hydrogen concentration for a given pressure and temperature [21,22]. Pure vanadium is a strong exothermic occluder of hydrogen. The addition of chromium tends to make the alloy less exothermic while the addition of titanium has the opposite effect. The hydrogen phase diagram for the alloy V-5Cr-5Ti therefore may be assumed to be similar to the H-V system.

Among all metal hydride systems, hydrogen-vanadium phase diagrams show the largest isotope dependence [23]. The maximum temperature at which metal hydride can form is about 180°C for H-V while being only ~130°C for D-V and ~100°C for T-V (fig. 3). In any case this sets a minimum temperature at which the material can be used: about 200°C.

Because hydrogen transport parameters can in general be very sensitive to temperature, the likely operating temperature of a vanadium first wall will be considered. Vanadium has the unusual property that the vanadium oxide V<sub>2</sub>O<sub>5</sub> has a much lower melting point ( $T_m = 667^\circ\text{C}$ ) than vanadium ( $T_m = 1900^\circ\text{C}$ ), and thus is susceptible to corrosion by oxygen above 667°C. The use of lithium as a coolant restricts the operating temperature to above 180°C (lithium melting point) and below ~500°C (where corrosion due to minute amounts of N dissolved in Li becomes a problem). Finally, the effects of radiation on mechanical properties would suggest an operating temperature between 500-700°C, in order to avoid embrittlement due to radiation hardening and helium. Consideration of all these factors puts the operation temperature of the vanadium wall at ~300-500°C.

The hydrogen solubility of vanadium is shown in fig. 2. The bulk diffusion coefficient for H in V is among the highest for any metal (fig. 1);  $\sim 10^{-8}$  m<sup>2</sup>/s in

the range 300-500°C [24]. This means that hydrogen would penetrate a 1 cm thickness of vanadium in  $\sim 10^4$  seconds.

Hydrogen transport can also be limited by barriers for dissociation and recombination of H<sub>2</sub> molecules at surfaces. The barriers are due to impurity atoms on the surface and hence are not a fundamental constant of the metal [2]. Vanadium is actually one of the more thoroughly studied metals in this respect [25]. The recombination rate coefficient, has been measured as a function of the surface coverage of sulfur atoms (fig. 4). It was observed that relative to other metals studied, the surface barrier for recombination was insensitive to the sulfur coverage.

Because of vanadium's high diffusion coefficient and inherently low recombination coefficient, release of implanted hydrogen ions is limited by recombination instead of diffusion. As such, energetic particles can be treated as if they arrive at the surface (i.e. the implantation depth may be ignored).

## **6.2 Effect of Hydrogen on Mechanical Properties**

A tokamak with vanadium walls has yet to be operated. To get a first approximation of the H retention expected for ITER, calculations will be made in the following section, based on measured hydrogen transport parameters for vanadium. The above information on the vanadium wall is important in two aspects: the quantity of fuel which will be lost to the wall and the degradation of vanadium's mechanical properties for sufficiently large hydrogen concentrations.

Several studies exist in the literature showing the effect of hydrogen on the mechanical properties of vanadium-based alloys [26-29]. Hydrogen concentrations of 0.1 at. % have been shown to increase the ductile to brittle transition temperature in V-5Cr-5Ti and other vanadium based alloys. Fortunately, this DBTT is still well below the minimum expected operating temperature, 200°C. Although hydride formation is not expected at temperatures above 180°C, stress in the material can result in hydride formation at higher temperatures. The hydrogen concentration can be as much as  $10^4$  times greater than that given by the solubility as a result of stresses [38]. As a result, hydrogen tends to diffuse to those areas under dilational stress (Gorsky effect) [30] which can promote crack propagation.

The local hydrogen concentration depends on the local temperature, i.e. hydrogen will tend to diffuse to and accumulate in regions where the temperature is the lowest. In order to be able to estimate the concentration gradients set up by temperature gradients, it is necessary to determine the value of the "Heat of Transport". This has only been investigated more fully for pure vanadium as well as niobium and tantalum hydrogen systems.

The vanadium alloys under discussion tend to have excellent resistance to radiation induced swelling [31]. However, the influence of hydrogen on swelling of neutron irradiated samples has not yet been established.

### 6.3 Calculation of Tritium Retention

To calculate the T inventory, we need first to know the hydrogen (deuterium, tritium) fluxes to the walls. The first wall of ITER will be exposed to mainly two sources of hydrogen:

- During glow discharge cleaning (GDC) the hydrogen gas pressure is usually held at approximately 1 Pa. GDC is carried out for several days at a time. The ion flux to the wall is on the order of  $10^{18}/\text{m}^2\text{s}$ . The energy of the ions impinging the wall is 200-300 eV.
- For actual tokamak discharges, the main particle flux will be directed at the divertor, which for ITER will not be constructed of vanadium. There will, however, be a substantial flux of charge-exchange neutrals to the vanadium walls, especially near the divertor. These fluxes are estimated to be as high as  $10^{21}\text{H}/\text{m}^2$  [32].

In considering the hydrogen retention due to these particle fluxes, it will be assumed that the back surface (unexposed to any H flux) of the vanadium is impermeable to hydrogen. This might actually be the case if ceramic coatings are used to electrically insulate the vanadium from liquid lithium coolant. In keeping with this, no calculation is presented for a tritium permeation rate through the first wall.

The concentration of hydrogen which will result from exposure to molecules during GDC is given by the hydrogen solubility. For

$P = 1\text{Pa}$ , this gives  $C(300^\circ\text{C}) = 9 \times 10^{25}\text{H}/\text{m}^3$  and  $C(500^\circ\text{C}) = 2 \times 10^{25}\text{H}/\text{m}^3$ , i.e. 250 - 1200 at ppm [33]. Since the duration of GDC (days) will be longer than the

characteristic filling time for 0.01m walls, either by diffusion or adsorption, the above equilibrium values will be attained.

Although the ion flux during GDC is smaller than the molecule flux, the ions have a high probability of being absorbed. (Note that there will also be a flux of neutral H atoms, comparable in magnitude to the ion flux, also with a high sticking probability). The equilibrium concentration due to the ions and atoms only is deduced by balancing the incoming flux,  $\phi_{H^+}$ , with the outgoing recombining flux.

$$\phi_{H^+} = K_r C^2 \quad (1)$$

where  $K_r$  is the recombination coefficient which describes the rate of release from the surface in terms of the bulk H concentration  $C$  [2].

Using the values of  $K_r$  deduced by Yamawaki et al. [25], and an ion flux of  $10^{18}$  H/m<sup>2</sup>s gives  $C(300^\circ\text{C}) = 4 \times 10^{25}$  H/m<sup>3</sup> and  $C(500^\circ\text{C}) = 3 \times 10^{24}$  H/m<sup>3</sup>. The filling time associated with ultimate concentrations are 100hrs and 10hrs respectively indicating that bulk hydrogen concentrations of 50-600 at. ppm will be reached if GDC continues for a few days, which is comparable to the contribution from the neutral gas molecules.

During a tokamak discharge the wall may be thought of as semi-infinite in depth, since the time to diffuse a 0.01m thickness of vanadium ( $\sim 10^4$ sec) is longer than the expected ITER pulse length ( $\sim 10^3$  sec). In this case we can apply the model for wall pumping [3] which considers simultaneous diffusion and recombination. The critical parameter in this model is the ratio of the diffusion coefficient to recombination coefficient  $\frac{D}{K_r}$ . Using the values of  $D$  and  $K_r$  from laboratory measurements [25] gives values of  $D/K_r$  of  $2 \times 10^{25}$  m<sup>-2</sup> and  $2 \times 10^{23}$  m<sup>-2</sup> for 300°C and 500°C respectively. This indicates an enormous capacity for particles to be pumped by the wall. Typical values of  $\frac{D}{K_r}$ ,  $3 \times 10^{18} - 9 \times 10^{20}$  m<sup>-2</sup> measured for inconel [3] and for beryllium about  $10^{20}$  m<sup>-2</sup> [34], are substantially smaller.

The pumping parameter can be interpreted physically as the incident fluence which the surface intercepts before the outgoing flux due to recombination becomes significant compared to the incident flux. The wall pumping time constant,  $\tau_p$ , is defined as

$$\tau_p = \frac{D}{K_r} \cdot \frac{1}{\phi_{in}} \quad (2)$$

where  $\phi_{in}$  is the incident flux. For the flux of  $10^{21} \text{H/m}^2 \text{s}$ , this gives  $\tau_p (300^\circ\text{C}) = 2 \times 10^4 \text{ sec}$  and  $\tau_p (500^\circ\text{C}) = 200 \text{ sec}$ . This means that except for surfaces with  $T > 400^\circ\text{C}$  and receiving fluxes  $> 10^{20} \text{H/m}^2 \text{s}$ , most of the H atoms striking the wall will not be released from the wall during the discharge.

Regarding plasma operation, wall pumping is an advantageous feature. It permits strong gas puffing, which can be used for gas-target divertor concepts. It also means there should be relatively easy density control. A density control problem may arise however if the wall heats up substantially over the course of the discharge; the walls will tend to expel hydrogen since steady state hydrogen concentration for a given flux is inversely proportional to temperature.

The maximum hydrogen concentration that will arise is determined from eqn. (1) for the case where  $\tau_p$  is less than the pulse length, giving  $C_{\max} (500^\circ\text{C}) = 6 \times 10^{24} \text{m}^{-3}$ .

The maximum concentration at  $300^\circ\text{C}$  will be given approximately the fluence over the diffusion depth since  $\tau_p$  is greater than the pulse length. At the maximum concentration,  $C(300^\circ\text{C}) = 3 \times 10^{26} \text{m}^{-3}$  (~4500 ppm), hydrogen can start to have deleterious effects on mechanical properties.

It is difficult to predict the inventory in the ITER wall since the distribution of the atomic flux is not known. A rough estimate can be made using the following assumptions: pulse length  $\Delta t_a = 10^3$  seconds, a uniform flux  $\phi_{in} = 10^{19} \text{H/m}^2 \text{s}$ , wall thickness  $x = 0.01 \text{m}$ , area =  $1000 \text{m}^2$ , average time between pulses  $\Delta t_b = 5 \times 10^3$  seconds. After many pulses, the concentration of hydrogen will reach a steady average level  $\bar{c}$  so that the influx during each discharge is balanced by release between discharges

$$\phi_{in} \cdot \Delta t_a = K_r \bar{c}^2 \cdot \Delta t_b \quad (3)$$

The average concentration which develop for these conditions are ~850 at ppm at  $300^\circ\text{C}$  and 70 at ppm at  $500^\circ\text{C}$ . For a 50/50 DT mixtures this amounts to 1.5kg and 120g of tritium respectively.

This inventory is however recoverable in principle. If release of the hydrogen in the wall is limited by recombination, then the time needed to reduce the concentration to 10 at. ppm (i.e. 8g of tritium for the above parameters) would be 35 days for 300°C and 45hrs for 500°C. It should be possible to speed up the tritium release by exchange with hydrogen or deuterium [35].

In all the above calculations the values of diffusivity, solubility and the recombination coefficient used apply to unirradiated material. A full set of parameters for neutron damaged material are not yet available, but will be needed since it is not clear what effect irradiation will have.

It may be possible to reduce the amount of hydrogen absorbed in the vanadium if a sufficiently thick permeation barrier is on the front surface. This approach is being considered by the TPX group in their design of the Ti alloy vacuum vessel [36]. A beryllium coating is being considered for ITER since it has low Z as well as low permeability. Permeation barriers are a major technological challenge. The plasma will tend to erode the coating. Too thick a coating will be susceptible to cracking or detachment, while too thin a coating may actually worsen the tritium uptake by acting as a barrier to recombination more than as a barrier to inward diffusion. Furthermore since Be tends to form brittle intermetallic compounds with most metals, an intermediate bonding layer may be needed.

## CONCLUSIONS

The hydrogen retention character of a tokamak strongly depends on the first wall materials used. The neutron fluxes expected in an ITER type tokamak requires the use of new first wall materials, vanadium based alloys being a prime candidate. These alloys will have hydrogen absorption properties similar to pure vanadium, and large concentrations of hydrogen (deuterium, tritium) can be expected to build up in the first wall.

Estimates of the tritium retention for a tokamak the size of ITER with a vanadium first wall in the temperature range 300°C to 500°C lead to the following conclusions:

- 1) Hydrogen concentrations will approach a level (0.1 at %) where mechanical properties are affected. These problems may be aggravated by enhanced hydrogen accumulation in areas under stress (e.g. weld joints). The situation is better for higher temperatures.

- 2) Large tritium inventories can be expected: ~1.5Kg for 300°C walls and 120g for 500°C walls. This inventory, however, is fairly mobile and the majority can be recovered by allowing the walls to outgas. It may be possible to shield the vanadium from incident tritium atoms, but this presents a major engineering challenge.
- 3) In general, first wall components have a finite capacity to pump hydrogen out of the plasma. In previous tokamak experiments this transient pumping effect has lasted up to ~1-10 seconds. It might seem then that such a short effect would have little impact on a machine with a 1000 second pulse length. Estimates of the wall pumping capacity for a vanadium first wall, however, suggest that a wall pumping effect in ITER will last hundreds of seconds.

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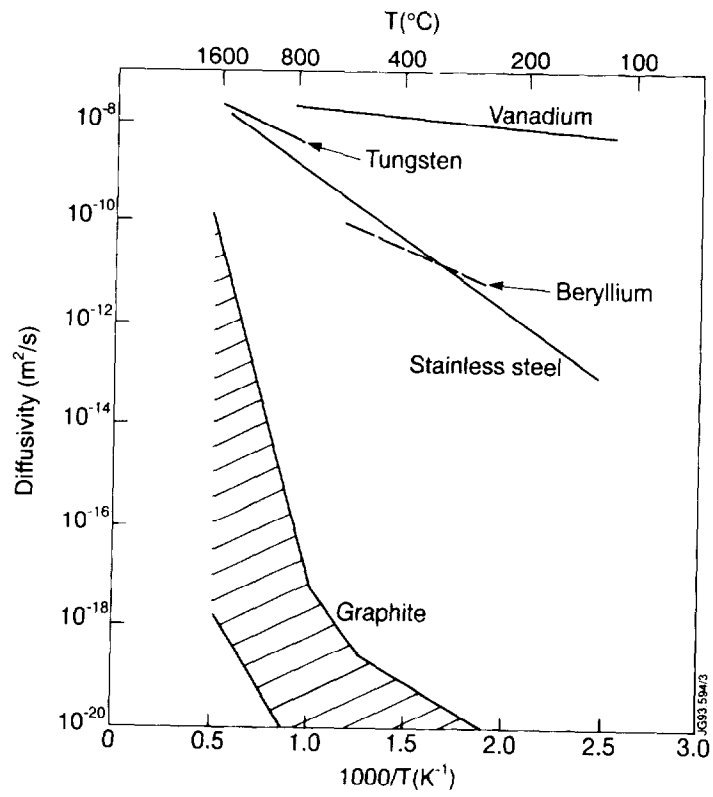


Fig.1. Diffusion coefficients for hydrogen in various first wall materials [37].

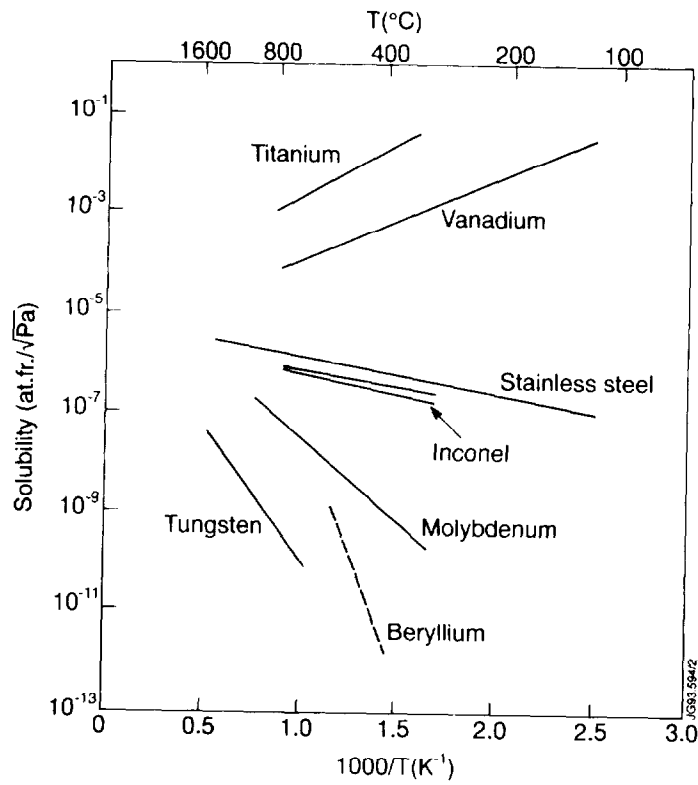


Fig.2. Hydrogen solubilities of various first wall materials [37].

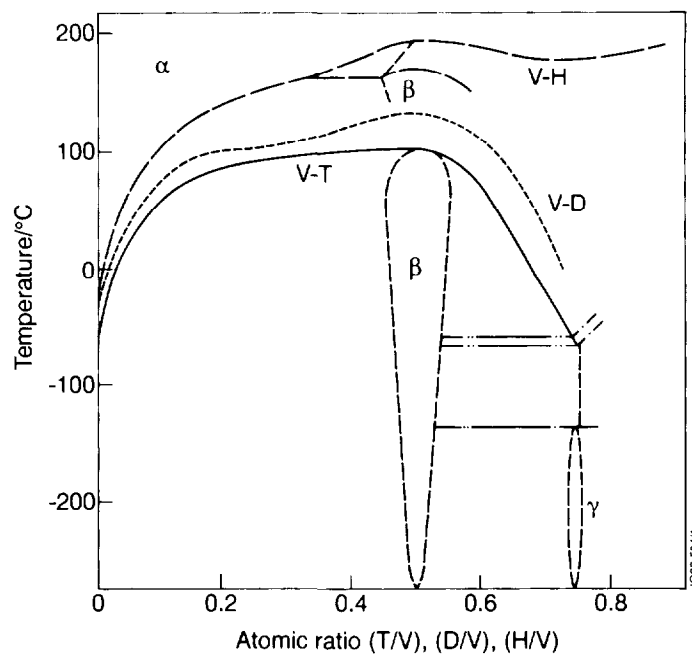


Fig.3. Phase diagram for V-H, V-D and V-T systems [23].

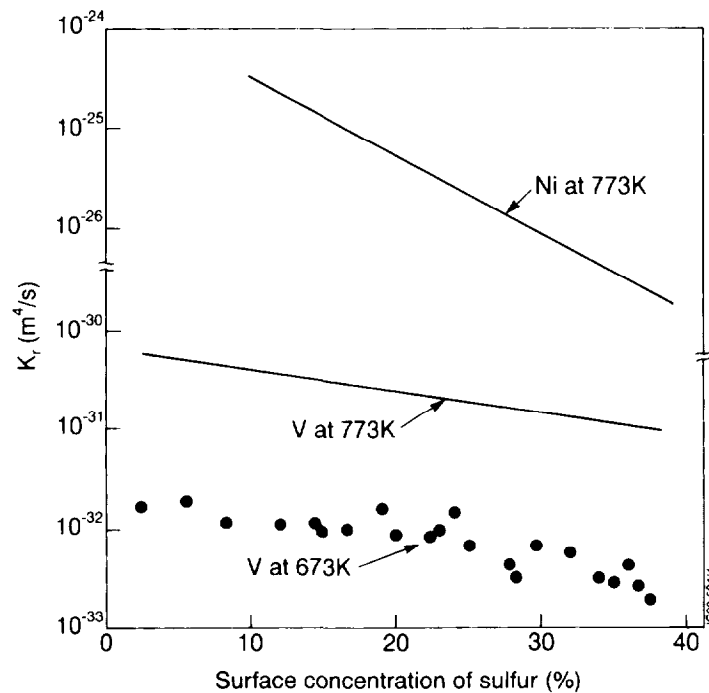


Fig.4. Recombination coefficient for hydrogen in vanadium and nickel as determined by Yamawaki et al. using permeation techniques. Squares are data from gas-driven permeation experiments [25], while the solid lines are fits to ion-driven permeation data [28].