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Factors involved in the use of vanadium alloys  
and ferritic steels for the ITER first  
wall/blanket and divertor

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## **REPORT A**

**Factors involved in the use of a vanadium alloy for the ITER first wall/blanket and divertor (including compatibility problems associated with impurities)**

## **REPORT B**

**Factors involved in the use of ferritic steel (9 Cr – 1 Mo) for the ITER first wall/blanket and divertor**

by

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**These two reports, A and B, were prepared in 1994 by Ron Bullough at the request of General Atomics USA and therefore refer only to our state of knowledge prior to that date. However, it was felt that their content may be of some historical interest if only as an indication of the progress since 1994 of our understanding of the various material property requirements for ITER. The text of each report has been modified, but not technically changed, from the original versions prepared in 1994.**

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## **REPORT A**

### **Factors involved in the use of a vanadium alloy for the ITER first wall/blanket and diverter (including compatibility problems associated with impurities)**

#### **Preamble**

This will not be a comprehensive review of the properties of vanadium alloys, but will attempt to highlight and discuss only those specific properties of such alloys that pertain directly to the use of such alloys for the ITER first wall/blanket and diverter; because of this restriction it is possible that certain properties may be overlooked that have unexpected synergistic relevance to the application. We will deliberately focus the discussion on the V-Cr-Ti alloys and, in particular, if data is available, on the relevant properties of the specific V-5Cr-5Ti alloy identified by Dr. Rebut in a design ITER study carried out in 1994. The required research to obtain information or understanding will be identified in **bold** in each section.

#### **The Required Material Properties**

To survive in the operating conditions and to be consistent with design requirements, the first wall \* material must have the following properties: (1)

- a) High heat load capability
- b) Mechanical property capability
- c) Ease of fabrication
- d) Resistance to radiation damage
- e) Low activation
- f) Chemical compatibility

\* (abbreviation for first wall/blanket and diverter)

To structure the review, we will attempt to relate the known or conjectured properties of the vanadium alloys to each of these required properties. Some duplication or repetition is inevitable because many of the required properties are interdependent. As indicated in the preamble, we will draw attention to any lack of understanding or knowledge of properties.

### **High heat load capability**

The first wall must be able to withstand a full plasma disruption. This implies an ability to withstand high thermal shock:

High thermal conductivity

Low thermal expansion

Low elastic modulus

High strength

All these material properties are reasonably well satisfied by the V-5Cr-5Ti alloy, particularly in the un-irradiated condition. However, as we shall see below when resistance to radiation damage is discussed, it is important to know if the limited swelling observed in V-Cr-Ti alloys is due to very few gas bubbles or voids being nucleated (as occurs in ferritic/martensitic steels) or to the presence of a very high density of such cavities (as occurs in many b.c.c. refractory metals such as molybdenum, niobium or tantalum when the so-called void -lattice often forms). Even without the ordering typical of a void - lattice the presence of a very high density of small bubbles or voids in irradiated vanadium alloys could severely reduce their thermal conductivity. The elastic moduli and intrinsic strength can also be degraded by such cavity densities.

**Thus need to know: effect of neutron irradiation on thermal conductivity, moduli and strength.**

### **Mechanical property capability**

The relatively high thermal conductivity of V-Cr-Ti alloys, compared with ferritic or stainless steels, permits a vanadium alloy first wall to be up to 4mm thick compared to 2mm for ferritic and only 1mm for stainless. In the absence of irradiation the vanadium alloys do have adequate UTS and creep strength up to very high temperatures (700°C). In addition, again in the absence of irradiation and, in particular, in the absence of impurity elements such as oxygen, nitrogen, helium and hydrogen (some of which, such as helium and hydrogen will definitely be generated by transmutations under high energy (14 MeV peak) neutron irradiation and all of which can be absorbed from the liquid lithium and helium gas associated with the blanket design) it is clear that the other required mechanical property capabilities - adequate ductility and fracture toughness and fatigue resistance - are satisfied by the vanadium alloys. The possible degrading effects of irradiation and adverse chemical compatibility on the mechanical capability will be discussed below. Suffice it to emphasise that:

**We need to have: detailed knowledge of the possible degrading effects of irradiation and the various and often associated interstitial impurities on the mechanical capability of V-5Cr-5Ti.**

### **Ease of fabrication**

There is obviously much less experience of fabricating complex structures out of vanadium alloys than out of ferritic or stainless steels. However, there are no known indications of any insuperable problems. Attention probably should be focused on the heat affected zones associated with welds. Thus grain growth in these zones may result in a large local DBTT shift and in contrast to the ferritic steels the (presumably) single phase vanadium alloys will not undergo martensite formation within the zones during cooling and thereby compensate for the grain growth. Needless to say such grain growth, that certainly would happen in pure vanadium, may be severely

inhibited in the alloy by the presence of precipitate pinning phases on the grain boundaries- unless, of course, any such precipitates themselves dissolve within the zones!

**Thus need:**

- 1. Study of grain growth in heat affected weld zones and tests for local DBTT shifts in V-5Cr-5Ti.**
- 2. Study of any non-equilibrium phase stability in this alloy that might be induced during cooling.**

It is possible that this problem could be overcome by using a very narrow weld with an associated highly localised heat affected zone.

Because of activation problems it is essential to ensure that the concentration of residual niobium in the vanadium alloy is very low (<0.1 ppm). The feasibility of achieving such a low concentration of niobium will obviously depend on the ore -> metal extraction route and may well impose a severe economic penalty.

The range of fabrication routes of vanadium alloys have been described by various authors (2, 3). It is clear that the development of such alloys for fusion applications will involve complex processing (thus hot forming of such alloys requires careful and highly specific protective coating procedures to avoid the formation of vanadium oxide phases) to prevent contamination by unwanted interstitial pick up.

### **Resistance to radiation damage**

The relevance of requiring low swelling, low radiation creep, acceptable loss of strength, ductibility and fatigue properties depends on the relative importance attached to the technology phase compared to the physics phase of ITER operation. We will

assume here that the former phase is important and that it is required to run ITER with significant neutron loading on the first wall ( $\sim 2\text{MW/m}^2$ ).

Before discussing the various radiation damage effects it is important to emphasise that, in contrast to the material development programme for fast reactor core components, there is no available fusion materials test reactor and therefore any first wall materials development programme will have to rely on simulation data; that is radiation damage data obtained with particles (electrons, light ions, heavy ions or fast neutrons), all with a damage rate or energy spectrum that is not that to which the first wall will be exposed. To make use of such data for reliably predicting the response of the first wall material to the actual fusion reactor neutron spectrum inevitably requires a comprehensive understanding of the various microstructural evolutionary processes that can prevail, coupled with the systematic construction of appropriate and consistent physically based models. Such a systematic approach has been proposed (2) and we consider that it is still appropriate to the current situation - suitably modified to include our currently more complete realisation of the range of physical processes that can prevail. In practice it is obvious that when a fusion reactor is built an alloy for the first wall will have to be selected on the basis of simulation data; a primary role of such a reactor must then be to validate the alloy choice made (by exposure to its own fusion neutron spectrum) and to use the opportunity to test systematically a wide range of alternative alloys for their radiation damage response to the actual fusion spectrum. Naturally, such a reactor would provide the best investment if the risk of failure to its first wall was minimal and we therefore need to identify what simulation data should best be sought to re-inforce our present (rather sparse) knowledge of the proposed V-Cr-Ti alloy.

### **Relevant radiation damage processes to be expected**

The resistance to radiation damage in any material is dependent on the physical and chemical changes that occur in the material due to the irradiation. These changes are

many, varied and often complex. It follows that a complete vindication of the suitability of any material is probably quite impossible. The best we can do is to indicate the wide range of R&D studies that are necessary based on any present data and general understanding we may have. As we have explained above such R&D studies must yield both data and understanding, since without the latter we cannot hope to deduce the material response in the real fusion environment. Let us therefore identify very briefly some of the processes in relation to the vanadium alloy situation.

1. **The presence of precipitates can significantly influence swelling, ductility and toughness**

The precipitate situation in irradiated vanadium alloys is complex (5). The precipitates in titanium containing alloys ensure very low swelling but are deemed responsible for large DBTT shifts (6). The morphology and density of these precipitates needs careful study particularly since they appear to be quite different in FFTF neutron irradiated material (5) compared to those in alloys irradiated with 40 MeV vanadium ions (7). Precipitation can enhance or inhibit the nucleation and growth of cavities (and hence swelling). Coherent precipitates (as in PE16 alloys) are probably effective recombination sites for self-interstitials and vacancies and hence reduce the swelling of such alloys. On the other hand, when precipitates are incoherent they can provide preferred sites for the nucleation of embryonic voids (small helium/hydrogen bubbles or sites for surface active gas accumulation) which can then develop into bias driven neutral voids and copious swelling. Such precipitates can greatly reduce the required "gas driven" incubation period defining the transition from relatively slow gas driven bubble growth to rapid bias driven void growth (swelling).

**Thus need: to fully understand the morphology and role of the Ti containing precipitates in the V-Cr-Ti alloy**

Needless to say their role in influencing the mechanical properties also depends critically on their morphology and chemical content.

When Ti is absent such alloys (V-Cr) can manifest enormous swelling (at rates greater than 1%/dpa) (8) and therefore the importance of the Ti containing precipitates cannot be over-emphasised. However, the large swelling often observed in alloys without titanium, with sparse indication of precipitation in that case, does suggest that vanadium is not an intrinsically low swelling pure material (as is pure iron and other refractory metals such as molybdenum, niobium and tantalum). We shall discuss this point later when we review the suitability of ferritic steels (Report B) for the first wall. Suffice it to comment here that we believe the low swelling in these latter materials is not due to a precipitation controlled process but is a more intrinsic feature of such b.c.c. materials.

## 2. **The influence of helium and hydrogen**

Both helium and hydrogen will be generated by transmutation reactions when vanadium alloys are exposed to the fusion neutron spectrum. The swelling under a fast reactor neutron spectrum with helium generation simulated by the "tritium trick" doping procedure is certainly increased by the presence of such helium (9). The helium appears to be dispersed with only a fraction associated with the grain boundaries and with most trapped (at precipitates?) in the matrix. There is thus considerable dispersion hardening but not severe helium embrittlement. Both helium embrittlement and creep rupture are usually thought to be due to the accumulation of gas on the grain boundaries. Needless to say these studies are not an accurate representation of the actual helium (and hydrogen) generations in a fusion reactor and the trapped helium within the matrix could, under continuous generation of helium and hydrogen, lead to the nucleation and subsequent growth of cavities (and hence significant swelling). In fact there is now (dual beam) evidence of synergistic effects of helium and hydrogen leading to significant swelling in pure vanadium (10).

**Thus need: dual beam simulation data, as reported in reference 10, for the V-Cr-Ti alloy. See also the effects of helium and hydrogen below on the compatibility of the vanadium alloy.**

3. **Damage spectrum effects**

The fusion neutron energy spectrum is very hard with its peak near 14 MeV compared with ~1 MeV for the fast reactor. This shift to higher energies produces both higher P.K.A. energies and a much higher generation rate of both helium and hydrogen. We have referred to the various issues associated with the transmutation gases but to these we have to add the absence of an appropriate neutron source with the hard fusion spectrum. We must therefore rely on simulation plus understanding to anticipate the effects of the inevitable high energy cascades.

**Thus we need: the morphology and defect yield of such cascades.**

Molecular dynamics studies would seem an attractive approach and certain, albeit limited, progress has been made.

**We need: acceptable potentials to represent the inter atomic forces in vanadium (let alone in the required V-Cr-Ti alloy) and very large (parallel) computing facilities to deal with the dynamics of very large cascades (many millions of atoms).**

In addition, some basic principles concerning the significance of the so-called "production bias" in relation to the usual "dislocation interstitial bias" needs settling, in order to interpret and exploit many of these molecular dynamic modelling results.

Although there is no direct neutron evidence to correlate with molecular dynamic studies in vanadium there are heavy ion damage studies (11) using 80 KeV<sup>+</sup> ions

which indicate that the defect yields and cascade collapse efficiencies in vanadium are very small and more like  $\alpha$ -iron, in sharp contrast to many of the other refractory metals.

**We need: heavy ion studies in the V-Cr-Ti alloy)**

#### 4. Miscellaneous effects and questions

There are many relevant radiation effects that we have not discussed, largely because their relevance for study will only become clear (both in purpose and in method) when we have answers to the above identified "needs". Thus, for example:

- a) The effects of toughness depend on hardening which in turn depends on the results of the precipitation studies.
- b) Segregation effects depend on understanding the complex transport by the intrinsic point-defects and these will require the detailed "rate theory" modelling of the evolving micro structure.
- c) Irradiation creep will almost certainly be "gas driven" leading to creep rupture. However, it does need "in pile" studies - which will require "in beam" simulation. The design of such experiments must be preceded by helium (and hydrogen) distribution studies as suggested above.
- d) Stress driven segregation of helium and other embrittling elements to microcracks or fatigue cracks have been studied theoretically but the relevance of such segregation to the first wall conditions and the vanadium alloy needs further consideration when some of the above "needs" are satisfied.
- e) Potential internal fatigue crack initiation is a possibility under the periodic thermal stress and periodic radiation damage prevailing in the first wall. Again to assess this possibility we obviously need to know precisely where the helium and hydrogen are located in the evolving microstructure in the vanadium alloy.

### **Low Activation**

The inherent low induced activity in the vanadium alloy is clear. We have previously referred to the need to reduce the niobium concentration below 0.1 ppm which undoubtedly raises economic problems that may be difficult to solve. In addition, of course, the inherent low activity advantage of vanadium must not be corrupted by adding alloy elements with unacceptable activity detriment, in order to improve its intrinsic suitability for the first wall. The situation has been reviewed by Butterworth and Giancarli (12) and they conclude that the titanium limit must be less than 5 wt%, aluminium must be reduced below 170 ppm together with the above niobium reduction. It should be noted that niobium has traditionally been used in the fabrication of many high strength vanadium alloys. An extensive review of the current situation for the design of low activation vanadium alloys for fusion applications has been given by Atkin and Rand (13).

### **Chemical Compatibility**

Interstitial elements such as oxygen, carbon and nitrogen can either act as solid solution strengtheners, or react with the alloy additions in vanadium alloys (14). The yield strength of vanadium can increase five-fold with increases of dissolved oxygen (0.005 to 0.22 wt%) or with increases of dissolved nitrogen (0.005 to 0.11 wt%) over the temperature range 0-600° C (14). However, the situation is somewhat confusing since Loomis et al (15) found no such sensitivity of the mechanical properties of the V-15Cr-5Ti alloy to oxygen and nitrogen concentrations and considered variations of mechanical properties were due to different alloy production routes ! It is clear that alloying additions with strong affinities for interstitials will reduce the solid solution hardening contribution from the interstitials but concomitantly increase the precipitation strengthening.

There is no doubt that the rapid oxidation of vanadium in the presence of oxygen to form the volatile oxide  $V_2O_5$  is a major drawback to its use in a high temperature oxygen environment.  $V_2O_5$  melts at  $670^\circ\text{C}$  and contact with air at temperatures above  $650^\circ\text{C}$  can result in severe oxidation. The corrosion rate and associated embrittlement is particularly severe in the presence of hydrogen (16) at levels to be expected in a flowing helium coolant such as proposed for ITER and moreover the severity of these effects is greater in the V-Cr-Ti alloy than in pure vanadium. The results obtained by Bell and Bishop (16) show clearly that when hydrogen is present at 70 vppm in helium flowing over V-Cr-Ti alloys severe embrittlement of the alloy occurs; the hydrogen will be present in such helium coolant and thus hydrogen embrittlement of vanadium alloys (even in the relative absence of oxygen) would appear to be a very serious problem. In addition, even when hydrogen was not deliberately added to the helium (to remove the oxygen) embrittlement of the alloy still recurred (particularly in the surface layers of the alloy samples). Thus hydrogen in the helium led to complete embrittlement and helium containing oxygen with no added hydrogen led to partial embrittlement of the V-Cr-Ti alloys. Clearly since hydrogen will inevitably be present in any helium coolant these results must impose serious doubts upon the suitability of the V-Cr-Ti alloy for the first wall.

**We need: as concluded by Bell and Bishop (16), to discover the critical hydrogen level that can be permitted in the helium coolant - note that hydrogen will almost certainly have to be added to the helium coolant to improve tritium recovery via isotopic exchange.**

Finally, of course, in the high energy fusion neutron environment we cannot, in any case, avoid the transmutation generation of both hydrogen and helium in the V-Cr-Ti alloy. The reported severity of the embrittlement of such alloys under flowing helium containing hydrogen re-inforces the need for the dual beam irradiation damage

(hydrogen and helium) study suggested above when we discussed the influence of helium and hydrogen on the radiation damage resistance.

## References

1. R. F. Mattas & D. L. Smith. J. Nucl. Mater. 191-194 (1992) 139
2. J. C. LaVake & C. T. Wang. Vanadium Purification, CEND - 3742-356, Combustion Engineering, Nov. 1969
3. H. R. Thresh, A. G. Hins & D. L. Smith. J. Nucl. Mater. 155-157 (1988) 608
4. R. Bullough, B. L. Eyre & G. L. Kulcinski. 68 (1977) 168
5. H. M. Chung & D. L. Smith. J. Nucl. Mater. 191-194 (1992) 942
6. N. S. Cannon, M. L. Hamilton, A. M. Ermi, D. S. Gelles & W. L. Hu. J. Nucl. Mater. 155-157 (1988) 987
7. D. J. Mazey, W. Hanks, T. Atkins, W van Witzenburg, D. K. Lurcock and B. C. Sowden. Technology Task Report LAM5, A.E.A. FUS. 144 (1991)
8. F. A. Garner, D. S. Gelles, H. Takahashi, S. Ohnuki and H. Kinoshita. J. Nucl. Mater. 191-194 (1992) 948
9. M. Satou, K. Abe and H. Matsui. J. Nucl. Mater, 191-194 (1992) 938
10. F. Kano, Y. Arai, K. Fukuya, N. Sekimura and S. Ishino. J. Nucl. Mater 203 (1993) 151
11. W. J. Phythian, B .L. Eyre and D. J. Bacon. J. Nucl. Mater 155-157 (1988) 1274
12. G. J. Butterworth and L. Giancarli. J. Nucl. Mater 155-157 (1988) 575
13. T. Atkins and M. Rand. Technology Task Report LAM 5, A.E.A. FUS 160 (1991)
14. D. L. Harrod and R. F. Gold. International Metals Reviews No. 4, 1980, p163, Review No. 255
15. B. A. Loomis, B. J. Kestel and D. R. Dierks. J. Nucl. Mater, 141-143 (1986) 523
16. G. E. C. Bell and P. S. Bishop. "Corrosion 93" The NACE Annual Conference Proceedings, March 8-12, 1993, New Orleans, Paper No. 177

## **REPORT B**

### **Factors involved in the use of ferritic steel (9 Cr - 1 Mo) for the ITER first wall/blanket and divertor**

#### **Introduction**

In this report we will attempt to focus on those properties of 9Cr-1Mo ferritic steels, such as T91, that pertain directly to their use for the ITER first wall/blanket and, again, where information, data or understanding is lacking we will identify the research needed. These 'needs' will then provide the foundation for the 'Research Requirement Summary' which completes the report. It must be emphasized that, in contrast to the vanadium alloys, an enormous amount of research, both experimental and theoretical, has been undertaken to validate the use of ferritic steels for the first wall of fusion reactors and for structural components (cladding and/or wrappers) of fast fission reactors (1-3). It is perhaps appropriate at the outset to give the 'consensus' view which we believe can be summarized as follows:

**Ferritic steels have many advantages over austenitic stainless steels but there is nevertheless real concern that, in presently developed ferritic steels, helium generated by transmutation in the fusion neutron spectrum may cause an unacceptable degrading of the fracture toughness with very large shifts of the DBTT in the irradiation environment.**

In order to develop ferritic steels that will maintain their toughness during 'fusion' irradiation it is essential to understand the role of helium in the embrittling process. This in turn implies that, of all the required material properties the first wall must have in order to survive in the operating environment and enumerated below, we should focus attention on:

- 1) Resistance to irradiation damage in the presence of the helium and hydrogen that *will* be produced by the high energy fusion neutrons and
- 2) Chemical compatibility - particularly in relation to a liquid lithium breeder and a helium coolant gas stream. Before doing so, however, it is appropriate to comment briefly on each of the remaining required material properties.

### **High heat load capability**

The ferritic martensitic steels such as 9Cr-Mo have, in general, superior thermal stress resistance compared with austenitic stainless steels (1-3). Their relatively high thermal conductivity and low thermal expansion coupled with high strength and low elastic modulus means that a sufficiently thick first wall can be constructed to withstand the thermal shock associated with a full plasma disruption. However, as we shall see when we discuss their resistance to irradiation damage, it is important to know whether any of these essential properties, that determine the first wall's high heat load capability, are severely degraded by high energy fusion neutron irradiation. It is perhaps worth saying immediately that because the low swelling in the ferritic steels occurs due to a dearth of voids (rather than an excess of voids, as in many of the refractory metals) it would seem most unlikely that the intrinsic thermal conductivity would be severely reduced. Unfortunately, as we shall discuss below, the same confident and immediate prognostication cannot be made with regard to possible loss of mechanical property capability due to the irradiation.

**Thus we need to know: effect of high energy neutron irradiation on the thermal conductivity (probably small) and on mechanical property capability (discussed below under resistance to irradiation)**

### **Mechanical property capability**

In the absence of irradiation the ferritic steels have adequate UTS and creep strength. However, as we shall discuss below, at temperatures up to  $\sim 450^{\circ}\text{C}$ , irradiation can lead to hardening with embrittlement and loss of strength (4,5). Above this temperature there is evidence of irradiation softening (6) and irradiation can actually inhibit the formation of Laves phase (4,7) with their associated embrittling effects. The probable effects of helium concomitant with such irradiation exposure are complex and varied and will be discussed below when we consider the specific effects of irradiation.

### **Ease of fabrication**

There is an enormous wealth of knowledge and experience concerning the fabrication of ferritic steels and there is no reason to believe that the first wall could not be successfully fabricated. In particular any potential loss of ductility in the heat affected weld zones by grain growth etc. is known to be adequately compensated for by the hardening due to martensite formation during cooling. For these reasons and improved intrinsic resistance to hydrogen embrittlement, the presence of dual phases in the 9Cr-Mo would seem an advantage - this is usually achieved by reducing the carbon and nitrogen content (8).

### **Resistance to radiation damage**

We consider that the response and potential degradation of the ferritic steels when exposed to the high energy fusion neutrons is the most serious issue facing their use as first wall materials. As we remarked in our discussion of this issue for the vanadium alloys (Report A), the relevance of requiring low swelling, low irradiation creep, acceptable loss of strength, ductility and fatigue properties depends on the relative importance attached to the technology phase compared to the physics phase of ITER operation. As previously, we shall assume that the technology phase is important and that ITER will be expected to survive with significant neutron loading on the first wall ( $\sim 2\text{MW}/\text{m}^2$ ).

Furthermore we cannot overemphasize the importance of simulation testing of materials with the necessary concomitant physical modelling of the microstructural evolutionary process. Without the latter modelling, conclusions drawn from simulation experiments, no matter how close they approach the actual fusion environment, must be treated with great caution. The absence of a fusion materials testing facility (analogous to the fast reactor materials testing reactors used so successfully to test and develop fast reactor core component materials) forces us to use the above more systematic approach with understanding development continuously coupled to the accumulation of simulation data. Needless to say, even this approach is not foolproof since there is always a finite risk that new unexpected phenomena may occur in materials exposed to the precise fusion environment that are totally absent from materials tested under simulation conditions quite close to that environment; thus all we can hope to do is to adopt an approach that gives us sufficient confidence in the wisdom of choosing a 9Cr - 1Mo ferritic steel such as T91 for the first wall.

### **Radiation damage processes in ferritic steels**

In almost complete contrast to the vanadium alloys there is a huge amount of data and knowledge of radiation damage processes in ferritic steels. In fact the amount of information is so large that to give a comprehensive review of it would be both impractical and completely indigestible. A very extensive source for such information is provided by the recent 'Proceedings of the Workshop on Ferritic/Martensitic Steels' referred to in Reference 3. In the present discussion we will focus on the processes and mechanisms that have been identified or conjectured and suggest where further research could yield appropriate insight or clarification.

### **Swelling, micro structural evolution and helium effects**

Reviews of the swelling observations in ferritic steels have been published recently (1,9) or will appear shortly (10); the salient features and conclusions may be summarized as follows:

- 1) Ferritic/martensitic (henceforth referred to as FM steels of which the 9Cr - 1Mo, T91 steel is of specific concern here) steels are all much more resistant to void swelling than are austenitic steels.
- 2) The maximum swelling rate ( $\sim 0.1\%/dpa$ ) is always lower than that for austenitic steels ( $> 1\%/dpa$ ) and the swelling rate and peak swelling temperatures ( $\sim 420^\circ C$ ) are both relatively insensitive to chromium content below 10% concentration. It follows that the chromium in solid solution is not primarily responsible for the swelling resistance of T91 and that the resistance is probably an intrinsic property of such FM steels and mechanistically akin to that of pure  $\alpha$ -iron.
- 3) The swelling of FM steels is enhanced under 1MeV electron and high energy ion irradiation (11). The enhancement under electron irradiation may be due to premature void assisted nucleation by ingress of gaseous impurities through the surface of the thin foil (it is well known that surface active impurities can often enhance void or bubble nucleation - for example, the high density void lattice formed in molybdenum under neutron or ion irradiation must have been nucleated by such impurities - where there is insufficient transmutation helium gas present). The enhancement under heavy ion irradiation is more puzzling but differences in dissolved gaseous impurities (e.g. oxygen or nitrogen ) may be responsible.
- 4) When helium is deliberately introduced by adding  $^{58}Ni$  to T91 (Irradiating a steel with 2% nickel in HFIR results in a helium/dpa ratio similar to that expected in the ITER environment) the swelling rate does show an increase (5) although again, this increase has been attributed to variations in dissolved gaseous impurities rather than due directly to the influence of the enhanced helium concentration on void nucleation.

**Thus need: careful irradiation experiments on the same batch (heats) of T91 steels to clarify the direct effect, if any, of helium and the relative importance of surface active gaseous impurities.**

Needless to say, the addition of 2% nickel creates a *different* alloy and nickel, being a well known austenite stabilizer, may be influencing the swelling propensity in ways other than by simply increasing the helium generation rate. Analogous dual ion-beam experiments by Asano et al (12) on 12Cr-1Mo steel (without the addition of nickel) confirm this concern that adding nickel may have other retrograde effects since Asano et al found only a very small increase in void concentration when the helium beam was present.

**Thus need: dual beam simulation experiments to high irradiation doses on T91 to confirm the relative insensitivity of the void concentration and swelling on the helium/dpa ratio.**

5) The combined affects of high input of helium and hydrogen have been studied using a triple beam (13) and show a rather significant increase in the peak swelling rate in 12Cr-1Mo steel.

**need: Triple (with hydrogen and helium) beam studies on T91 at gas injection rates close to those expected in ITER.**

It is important to emphasize that although the increase in peak swelling rate in the presence of hydrogen/helium is considerable, its absolute value is still modest (<0.1%/dpa) and much less than swelling rates observed in austenitic steels.

**Thus it is concluded that FM steels with bcc structure exhibit very good swelling resistance to dose levels exceeding 100dpa and the T91 steel is a particularly low swelling material.**

### **Mechanism of the swelling resistance**

Various mechanisms to explain the swelling resistance of FM steels have been suggested and have been reviewed in some detail by Odette (14). These include the notions that:

- a) the intrinsic interstitial-dislocation bias is lower in bcc iron than in fcc iron (15).
- b) solute atoms such as carbon or nitrogen have a high binding energy with vacancies in bcc iron and can thereby retard swelling or alternatively they can screen the dislocations with impurity atmospheres and reduce the bias (16,17).
- c) the grain boundaries may be strong recombination sites (18).
- d) when the void density is high (the only example in the FM steels appears to be FV548 when subjected to electron irradiation), the sink density can be so dominant that nearly all the radiation produced vacancies, and interstitials recombine *at* the sinks; this can also happen if the dislocation density remains high (19).
- e) the dual nature of the interstitial dislocation loops created by the aggregation of radiation-produced interstitials can impose a profound restraint on the swelling (20,21).

Of the mechanisms e) is, we believe, the most likely intrinsic reason for the observed void swelling resistance of FM steels and there is now direct observational support for its fundamental validity. The basic feature of the mechanism is (20,21) that in bcc iron interstitial loops with two possible Burgers vector, viz.  $a/2\langle 111 \rangle$  and  $a\langle 100 \rangle$ , can form by alternative unfauling shears from a common faulted  $a/2\langle 110 \rangle$  loop nucleus. The shear to produce the (100) loop with its large Burgers vector is much greater than that needed to produce the usual (111) loop. Hence within the continuously generated interstitial loop population only a very few (100) loops will form amongst the vast majority of (111) loops. Because the Burgers vector of the (100) loops is much greater than the (111) loops, the (100) loops will have a preferential bias for interstitials and grow, whereas the (111) loops will be relatively neutral sinks and absorb a net flux of vacancies and have a finite lifetime. There are thus *two 'dislocation' sinks* of different bias always present in the evolving

microstructure and the probability of a third sink type (even more 'neutral' than the (111) loops) such as voids, nucleating and growing is small. This dynamic situation will prevail to very high irradiation doses with a gradual build-up of a dislocation network of a  $\langle 100 \rangle$  dislocations as the (100) loops preferentially continue to grow and eventually impinge upon each other. The transition to such a network is found in FM steels (20,23) and direct TEM observations of preferential interstitial loss to (100) loops have been made (24).

Clearly this mechanism is consistent with the relative insensitivity of the void concentration to the generation rate of helium and/or other gases since void formation is simply difficult in such a dual sink microstructure when a relatively neutral sink (the (111) loop) is being continuously produced in abundance.

Before concluding this brief discussion of void swelling and microstructural evolution, it is appropriate to comment on the recoil spectrum sensitivity of these phenomena. The situation seems very fortunate since in bcc metals and in  $\alpha$ -iron in particular, cascade collapse with the formation of vacancy loops does not easily occur (25). This fact and the sheer difficulty of forming vacancy planar aggregates (by quenching or radiation) in  $\alpha$ -iron encourages one to think that there may be a reasonably weak dependence on the recoil spectrum of the microstructural evolution in FM steels. This conjecture is also consistent with the more direct and interpretable correlations that can often be made between simulation and fast reactor neutron data in such steels.

**Thus need: recoil spectrum insensitivity very carefully substantiating for T91 steel.**

### **Tensile and Fracture Properties**

In the absence of large amounts of helium, irradiation to high dpa levels does not seriously degrade the tensile and fracture properties of FM steels (1). Studies of the dependence of tensile properties on helium have been made in T91 by adding nickel

and irradiating in HFIR. The yield stress is increased by about 20% in the presence of helium but with a distinct saturation at doses above  $\sim 10$  dpa. The precise way that helium causes hardening is not known but it could be stabilizing a proportion of the small (111) interstitial loops and thereby inhibiting their shrinkage (or even just prolonging their lifetime). Since helium (and hydrogen) is so important in FM steels a precise understanding of the location of the helium within the microstructure must be important - especially for assisting the modelling of the concomitant microstructural evolutionary processes.

**Thus we need: microstructural studies to determine the location of helium to explain its effects on hardening.**

It might be ideal if this could be done by dual (or triple) beam irradiation or by linking recent helium implantation studies (26) with beam irradiations. Incidentally, in the absence of simultaneous irradiation, implanted helium appears to form very small bubbles on the dislocations and on some boundaries (26) - such effects could be looked for in the irradiated materials. From these latter results it would seem important to maintain the first wall temperature above  $\sim 300^\circ\text{C}$  since below this temperature hardening by helium implantation certainly occurs.

Traditional helium embrittlement, caused by the aggregation of helium on grain boundaries as bubbles, leading to premature creep rupture for example (or irradiation-gas driven creep rupture) or to intergranular fracture, is a feature of austenitic steels when irradiated at high temperatures. Such traditional helium embrittlement (27) (or hydrogen embrittlement (27,28)) is not considered to be a serious problem in FM steels (1). The study in reference (28) also suggest that little or no synergistic embrittlement effects of helium and hydrogen exist in these materials.

Kimura et al (29) have reported rather severe hydrogen embrittlement in 9Cr-2W steel under neutron irradiation conditions which they attribute to radiation-induced segregation of phosphorous and/or sulphur influencing the efficiency of the hydrogen embrittlement process.

**Thus need: corresponding hydrogen embrittlement study for T91 steel.**

In fact irradiation effects on toughness are considered to be the greatest concern for the fusion applications of FM steels (1). In T91 steel the DBTT shift and decrease of the upper shelf energy can be significant. For example, when irradiated in EBR II at 390° C to 13 dpa a DBTT shift of 52° C was observed; the shift does, however, appear to saturate with dose (54° C at 26 dpa). Much larger shifts were found in a 12Cr -1Mo steel. In this respect the reduced activation FM (Cr - WVTa) steels have lower shifts than T91 steels. Corresponding irradiations of nickel-doped T91 steel in HFIR to generate helium yielded a massive 204° C shift with no sign of saturation (350° C shift at 40 dpa irradiation dose!). The fracture mode is now intergranular - thus any hardening due to helium has saturated and the reduction of toughness (embrittlement) is directly due to a lowering of the fracture stress across prior austenitic or lath boundaries. Needless to say, an understanding (atomic in nature or due to bubble growth) of this loss of cohesive strength across the boundaries is of crucial importance and may well be inextricably mixed with the radiation enhanced segregation processes of other embrittling solute (such as phosphorus and/or sulphur). **Thus need: Auger studies or accurate STEM analyses to determine precisely what (and how much) has segregated on the boundaries.**

Such measurements can then be carefully correlated with various segregation models - such as for example the extensive studies of Bullough, Rauh and Hipsley (29,30). Thus it may be necessary to remove critical embrittling solute impurities such as sulphur or phosphorus from T91 steel - at any rate , the effect of such removal is well worthy of study.

### **Chemical Compatibility**

A great deal of data and understanding of the factors controlling the corrosion of structural steels in liquid lithium and other breeder materials now exists and has been recently reviewed (31,32). The important conclusion is that the FM steels are, in general, much more resistant to corrosion than are the austenitic steels; the former dissolve at a much slower rate and their surfaces remain macroscopically much less

pitted than the latter. However it is now recognized that solubility-driven reactions are not the only significant degradation mechanism for steels in lithium (31). In fact, the mass transport due to solubility reactions can be substantially affected or masked by reactions involving solute such as nitrogen or carbon, which are particularly important at temperatures below 500°C (33,34). The chemical reactions involving these solute are complex and an accurate estimate of effective dissolution (corrosion) rates in FM steels and in T91 steel in particular requires an awareness and understanding of them.

**Thus need: the total mass transfer burden of T91 steel in the ITER temperature range and the purity requirements on the lithium.**

At temperatures above 400°C there are no observed deleterious effects of liquid lithium on FM steels.

The potential absorption of hydrogen from the helium coolant is an obvious potential embrittlement problem - particularly in the presence of radiation induced segregation processes. The need to have such data and to understand hydrogen embrittlement in irradiated T91 steel has been identified above previously.

### **Summary of Research Needs for T91 steel**

These have been identified (in bold) in the text and are collated here with further remarks.

1. Effect of high energy neutron irradiation on the thermal conductivity. (In view of the probable relative insensitivity of the microstructural evolution in T91 to recoil spectra a fast reactor neutron spectrum should provide reliable data. However, to induce a more reliable void/bubble concentration the appropriate helium could also be introduced with nickel doping. Alternatively, a dual (or triple) beam simulation study may be considered).

2. Ascertain the true effects of helium on void concentration by using T91 specimens from the same 'heats'. A careful study of the laboratory data records may be sufficient to clarify this issue (Reference (5)).
3. Perform dual beam simulation experiments to high irradiation doses to confirm the relative observed insensitivity of the void concentration and hence swelling on the helium/dpa ratio.
4. Perform triple beam simulation experiments (hydrogen and helium) at gas injection rates close to that in ITER. The possibility of synergistic effects between hydrogen and helium must be clarified.
5. Check that the microstructural evolution in specifically T91 steel is 'reasonably' insensitive to recoil spectrum.
6. Microstructural study of location of helium as a source of irradiation hardening.
7. A study of hydrogen embrittlement in T91 steel akin to that reported in reference 29.
8. Auger and/or STEM studies to determine the extent of radiation induced segregation on boundaries in T91 and the synergisms with helium and/or hydrogen. The loss of cohesive strength across the boundaries dominates the DBTT shift and must be understood.
9. Determine the mass transfer burden of T91 in the ITER temperature range and the purity requirements on the lithium liquid breeder.

Finally we conclude the report by a 'Miscellaneous effects and questions' section which is similar to the analogous section of our report on vanadium alloys (Report A).

### **Miscellaneous effects and questions**

There are many relevant radiation effects that we have not discussed, largely because their relevance for study will only become clear (both in purpose and in method) when we have answers to the above identified "needs". Thus, for example:

- a) The important segregation effects depend on understanding the complex transport by the intrinsic point-defects and these will require detailed "rate theory" modelling of the evolving micro structure.
- b) Stress driven segregation of helium and other embrittling elements to microcracks or fatigue cracks have been studied theoretically but the relevance of such segregation to the first wall conditions and T91 steel needs further consideration when some of the above "needs" are satisfied.
- c) Potential internal fatigue crack initiation is a possibility under the periodic thermal stress and periodic radiation damage prevailing in the first wall. Again to assess this possibility we obviously need to know precisely where the helium and hydrogen are located in the evolving microstructure in T91 steel.

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## **References**

1. R. L. Klueh, K. Ehrlich and F. Abe, J. Nucl. Mater. 191-194 (1992) 116.
2. D. R. Harries, G. J. Butterworth, A. Hishinuma and F. W. Wiffin, J. Nucl. Mater. 191 -194 (1992) 92
3. F.W. Wiffin in "Proceedings of the Workshop on Ferritic/Martensitic Steels" JAERI Tokyo, Japan 26-28 October 1992 (Vols I and II). Eds: F. Abe, A. Hishinuma, A. Kohyama and M. Suzuki. (together with many other papers in these proceedings).
4. A.E. Little and L. P. Stoter in: Proceedings of 11th Intern. Symp. on

- "Effects of Radiation on Materials", ASTM-STP 782, eds H.R. Brager and J.S. Perrin (ASTM, Philadelphia, 1982) 207
5. P. J. Maziasz and R. L. Klueh, in: Proceedings of 14th Intern. Symp. on "Effects of Radiation on Materials", ASTM-STP 1046, eds N.H.Packan, R. E. Stoller and A. S. Kumar (ASTM, Philadelphia, 1989) 35
  6. R.L. Klueh and J.M.Vitek, J.Nucl. Mater. 182 (1991) 230
  7. P.J. Maziasz, R. L. Klueh and J. M. Vitek, J. Nucl. Mater. 141-143 (1986) 929
  8. N. Igata, J. Nucl. Mater. 133-134 (1985) 141
  9. E. A. Little, in Proc. Symp. "Materials Modelling: from Theory to Technology", (Inst. of Physics, Bristol), (1992), 141
  10. E. A. Little, J.Nucl. Mater. Accepted for publication.
  11. E. A. Little, Phys. Stat. Sol. (a) 87 (1985) 441
  12. K. Asano, Y. Kohno, A. Kohyama and G. Ayrault, J. Nucl. Mater. 155-157 (1988) 912
  13. K. Farrell and E. H. Lee, ASTM Spec. Publ. No. 955(1987) 498
  14. G. R. Oddette, J. Nucl. Mater. 155-157 (1988) 921
  15. J. J. Sniegowski and W. G. Wolfer, in Proc. Topical Conf. on Ferritic Alloys for use in Nucl. Energy Technologies, (USA: AIME), (1984) 579
  16. E. A. Little and D. Stow, J. Nucl. Mater. 87 (1979) 25
  17. M. R. Hayns and T. M. Williams, J. Nucl. Mater. 74 (1978) 151
  18. P. J. Maziasz, R. L. Klueh and J. M. Vitek, J. Nucl. Mater. 141-143 (1986) 929
  19. L. K. Mansur and E. H. Lee, J. Nucl. Mater. 179-181 (1991) 105
  20. E. A. Little, R. Bullough and M. H. Wood, Proc. Roy. Soc. A372 (1980) 565
  21. R. Bullough, M. H. Wood and E. A. Little, ASTM Spec. Publ. No. 725 (1981) 593
  22. B. L. Eyre and R. Bullough, Phil. Mag. 11 (1965) 31

23. T. S. Morgan, E. A. Little and R. G. Faulkner, ASTM Spec. Publ. No 1175 (1993)
24. T. Muroga, A. Yamaguchi and N. Yoshida, ASTM Spec. Publ. No. 1046 (1989) 396
25. C. A. English, J. Nucl. Mater. 133-134 (1985) 71
26. A. Hasegawa, N. Yamamoto and H. Shiraishi, J. Nucl. Mater. 202 (1993) 266
27. H. Schroder and H. Ullmaier, J. Nucl. Mater. 179-181 (1991) 118
28. K. K. Bae, K. Ehrlich and A. Moslang, J. Nucl. Mater. 191-194 (1992) 905
29. C. A. Hipsley, H. Rauh and R. Bullough, Acta Met. 32 (1984) 1381
30. H. Rauh and R. Bullough, Proc. Roy. Soc. A427 (1990) 1
31. T. Flament, P. F. Tortorelli, V. Coen and H. U. Borgstedt, J. Nucl. Mater. 191-194 (1992) 132
32. See paper in Session 2D of the Workshop on Ferritic/Martensitic Steels (Ref (3))
33. O. K. Chopra and D. L. Smith, J. Nucl. Mater. 141-143 (1986) 584
34. P. F. Tortorelli and J. H. DeVan, Proc. Topical Conf. on Ferritic Alloys for Use in Nuclear Energy Technologies (AIME, 1984) 215