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Status of vanadium alloys for fusion reactors

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Abstract

Advantages of vanadium alloys for fusion reactor structural applications are: low induced activation, excellent thermal stress factor, high strength at elevated temperatures, and superior ductility at low temperatures. Resistance to irradiation damage is also very impressive, i.e. very small DBTT shift by irradiation and low swelling. Research and development of vanadium alloys have made a remarkable progress in recent years partly supported by ITER-related activities. Composition range centered about V–4Cr–4Ti is the target of many of the studies conducted in the US, Japan and RF. Most of the studies have demonstrated the superior performance of this alloy family, while some alloys containing large amount of impurities have shown relatively poor properties. In addition to the baseline mechanical properties, neutron irradiation effects, e.g., swelling and radiation embrittlement are covered in this paper. Of particular importance is the study on the effects of dynamically-charged helium on mechanical properties and swelling. Recent developments of insulator coatings and welding are also covered. The importance of mechanistic studies of the damage behavior is emphasized for efficient alloy development and prediction of materials life in service.

1. Introduction

Successful development of low activation material is one of the most important tasks for the realization of fusion power system. Vanadium alloys are inherently low activation materials and have a number of advantageous properties over other alloys. Therefore, these alloys have been the target of many studies [1–3]. Although the primary objective of vanadium alloy research is to develop materials for fusion DEMO or power reactors, R&D of vanadium alloys has recently been substantially accelerated by the ITER-related activities. In the background of these activities, there are substantial effort categorized as fundamental studies. It should be acknowledged that properly directed fundamental studies are essential for efficient vanadium alloy development and prediction of their performance.

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Importance of vanadium alloys resides in the fact that these are the only realistic materials for use in commercial reactors which can possibly acquire economical competitiveness in the middle of the next century. Estimated cost of electricity of fusion power reactors projected from the current material performance is much higher than those of advanced PWR [4]. Environmental friendliness or public acceptance may compensate this surplus cost of fusion energy and may result in improved competitiveness of fusion energy. Thus, materials with long life, high operation temperature and low induced activation are the key to the realization of commercial fusion reactors. The operating temperature of ferritic steels, lower than 550°C, may be too low for good thermal efficiency. Although SiC/SiC compounds potentially have very attractive features, there are many uncertainties with these materials especially in their irradiation responses.

There are also disadvantages for vanadium alloys. First of all, the lack in industrial basis is rather substantial and unless some other application of vanadium alloys is devised, the cost to surmount this hurdle will be rather high.

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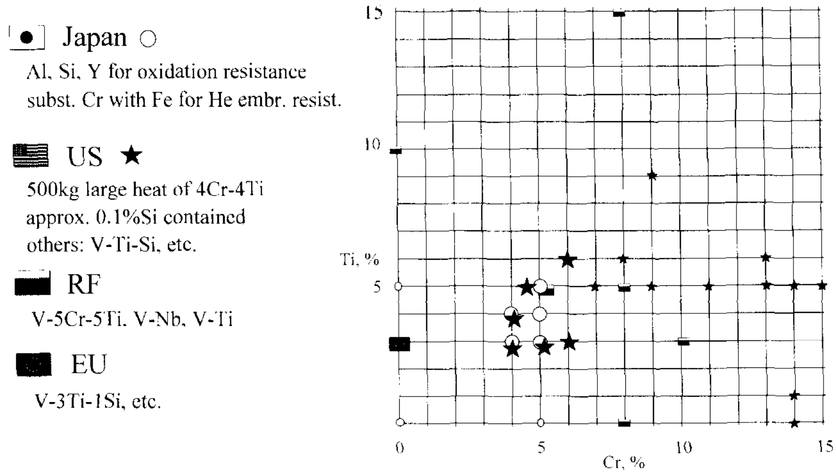

Fig. 1. Range of the V–Cr–Ti ternary alloy composition studied in different sectors of the world.

Other than this, vanadium alloys even overcompensate their demerits with benefits. Research issues which are addressed in this paper are: (1) baseline mechanical properties, (2) irradiation response, (3) helium effects, (4) corrosion and compatibility and (5) welding.

Most promising alloy composition at present is centered about V–4Cr–4Ti [2]. This alloy family is characterized by very low DBTT and small shift in DBTT by irradiation [5]. Research and development efforts in Japan [6,7], US [8] and RF [3] are all focusing on this alloy family, with some different emphasis on relatively minor alloy components. Fig. 1 shows the range of the ternary alloy composition studied recently in different sectors of the world. Larger symbols represent greater emphasis compared to smaller ones. It is seen from this figure that current efforts are centered near V–4Cr–4Ti. A large (500 kg) heat of this alloy has been produced in the US and the experiments conducted on this heat in various laboratories including those outside the US consistently showed excellent properties of this alloy. A still larger heat has been obtained recently for a diverter application in the DIIID machine at General Atomics.

In the present paper, an overview of the recent progress in vanadium alloy development is presented. Since there are several excellent reviews [1,2,9] on this subject, those topics which were not extensively covered in previous review papers will be emphasized.

2. Baseline mechanical properties

2.1. Ductility

Vanadium–chromium–titanium alloys with composition centered on 4%Cr–4%Ti generally have excellent ductility [2] as mentioned above. In ITER-driven activities, these alloys have been extensively studied in several laboratories, and remarkable progress has been made. Through these activities, substantial common knowledge base has been established on material composition, heat treatment, test method, and specimen size and geometry.

Charpy tests of these vanadium alloys are commonly done using a few different specimen geometries; their notch angle/root radius/notch depth combination is 45°/0.25/0.61 (1/3 CVN), 30°/0.08/0.61 (1/3 CVN), or 30°/0.03/0.45 (1.5 CVN). Here, CVN stands for Charpy V-Notch specimens and 1/3 CVN has dimensions (in mm) $3.3 \times 3.3 \times 25.4$, and those of 1.5 CVN are $1.5 \times 1.5 \times 20$; the latter is often used in Japanese university programs. While the notch geometry is expected to influence DBTT behavior, the former two types of 1/3 CVN specimen turned out to give almost identical results from each other, only slightly greater impact energy for 30° notch angle specimens in the higher temperature region [10]. Direct comparison between 1.5 CVN and 1/3 CVN is not yet available for vanadium alloys. In a correlation study [11] using 10Cr–2Mo–1Ni ferritic/martensitic steel, almost identical DBTT was obtained from both types of specimens, i.e. 30°/0.08/0.61 (1/3 CVN) and 30°/0.03/0.45 (1.5 CVN). This correlation is assumed to hold in the discussions below.

V–Cr–Ti ternary alloys have been extensively studied especially at ANL. Fig. 2 shows a 3D plot of the DBTT for the ternary alloys as a function of titanium and chromium based on the data obtained at ANL [12,13] and IMR/Tohoku Univ [7]. Only data on appropriately-prepared specimens are included in this plot. The recommended specimen fabrication procedures are given in the next section. It can be seen from this plot that there is a fairly large flat bottom region extending from 0%Cr–0%Ti to 5%Cr–5%Ti. Titanium is needed for the resistance against swelling and hydrogen embrittlement, and

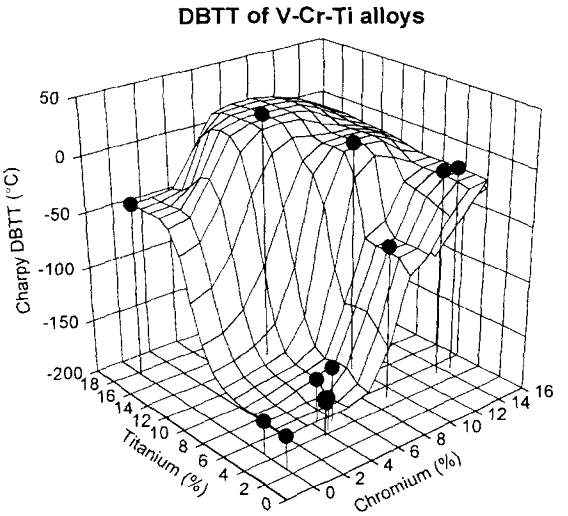

Fig. 2. A 3D plot of the DBTT for the ternary alloys as a function of titanium and chromium based on the data obtained at ANL and IMR/Tohoku University.

chromium is for strength and some corrosion resistance. Excessive amount of titanium is not desirable also because it leads to lower strength and creep life. Some margin is needed for DBTT shift by irradiation and also for the transmutation of vanadium into chromium at an expected rate of 0.01 appm/dpa in a fusion spectrum. This is the reason that many of the researches are focusing on the V–4%Cr–4%Ti composition.

Optimization of heat treatment procedures has also been pursued. Fig. 3 shows ductile to brittle transition curves of V–4Cr–4Ti–0.1Si alloy measured using 1.5 CVN specimens with different annealing temperatures [7]. The DBTT is rather high for specimens annealed at 1100°C, while it gradually decreases by lowering the annealing temperature. For specimens annealed at 950°C, DBTT is below −200°C. Fracture surface was transgranular cleavage type in the brittle region and no sign of intergranular fracture was observed. This shift in DBTT by annealing at different temperatures in this case has been ascribed to the smaller grain size when annealed at low temperatures based on metallographic examination. Thus, thermo-mechanical treatment is very important for the performance of vanadium alloys as well as any other engineering materials. The optimum procedure recommended by ANL is as follows [14]: (1) Extrude ingot in a stainless steel jacket at 1150°C into ca. 65 mm thick slabs. (2) Subsequent warm rolling at 400°C to desired intermediate dimensions. (3) No more than 15% thickness reduction per each rolling step. (4) No more than 50% thickness reduction accumulated between annealing. (5) Anneal at 1050–1070°C for 2 h in a high vacuum furnace between rolling. (6) Anneal at 1050°C for 2 h in high vacuum only for selected final products thicker than 3.6 mm. Other products deliver in as-rolled condition. The vacuum during annealing has a large effect on the DBTT behavior [10]. The 500 kg heat of V–4Cr–4Ti does not show DBTT down to −200°C if annealed in ion-pumped system at 1050°C for 1 h, while the DBTT of the same alloy but annealed in an oil-diffu-

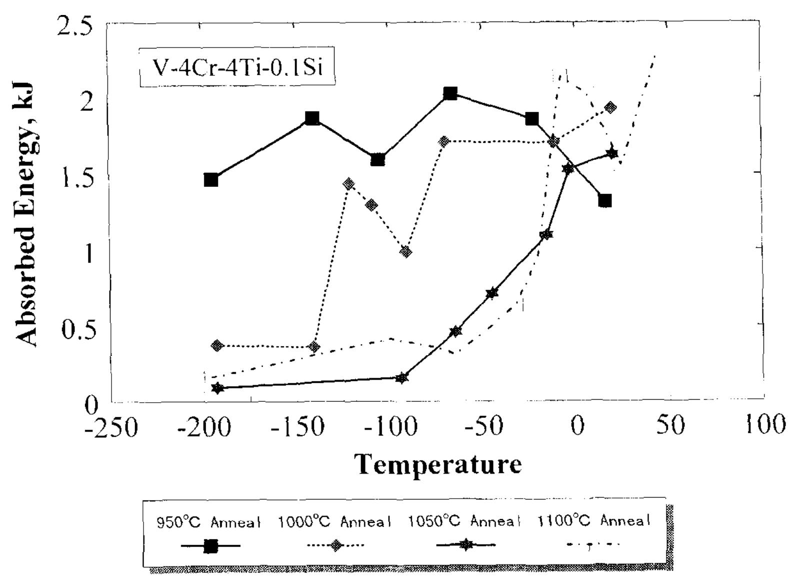

Fig. 3. Ductile to brittle transition curves of V–4Cr–4Ti–0.1Si alloy measured using 1.5 CVN specimens with different annealing temperatures.

sion pump system for 2 h is at about −170°C. The cause for this difference is suspected to be due to impurity pickup during the annealing.

2.2. Creep

Creep data on V–4Cr–4Ti alloy are currently being accumulated but very time consuming by nature, so that the progress in the last 1–2 years is not very impressing. Here, only a general remark will be made. Comparison with other materials such as 316 stainless steel or ferritic steels show a definite advantage for this alloy in high temperature mechanical properties. For instance, rupture stress for 10000 h at 600°C is 400 MPa for this alloy while the corresponding stress for 316 steel or HT-9 is only 120–130 MPa [9]. It is further encouraging that the data for this alloy for a longer period tend to approach those of very high strength V–15%Cr–5%Ti alloy.

3. Irradiation response

3.1. Swelling

Selected vanadium alloys have superior swelling resistance. Since the alloy V–4Cr–4Ti is relatively new, the swelling data on this alloy is limited to low damage levels. Fig. 4 shows the swelling data obtained for V–Cr–Ti alloys. The swelling of V–4Cr–4Ti at 30 dpa is about 0.3%, which is lower than for the other two alloys in this figure which have higher chromium content. Interestingly, the swelling level appears to become smaller for higher dpa levels. This finding has been interpreted in terms of the change in sink balance due to the radiation-induced fine precipitation of titanium silicide particles [15]. This observation implies that cavities once grown to some size shrink by further irradiation damage. Since this set of data has been obtained from a series of different irradiations,

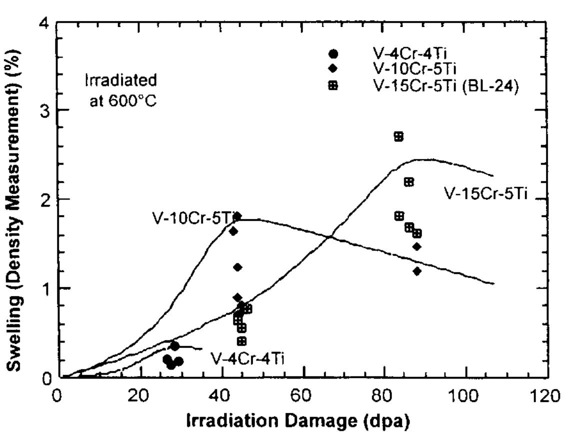

Fig. 4. Swelling of V–Cr–Ti ternary alloys.

possible effects of damage rate, temperature and irradiation history, etc. cannot be excluded convincingly. This phenomenon, nevertheless, is worth closer study since this may be a clue leading to the development of alloys resistant to swelling at very high damage levels relevant to power reactors. If, in contrast, these radiation parameters such as dpa rate have such a pronounced effect on swelling, this will be another important topic of research, since the mechanism underlying this phenomenon may invalidate many of the data obtained using similar procedures.

In some vanadium-based binary alloys with undersized solutes, e.g. iron or chromium, very large swelling has been observed [16]. In the ternary alloy, i.e. V–4%Cr–4%Ti, swelling is very effectively suppressed by the titanium addition. In order to predict the performance of the material at high damage levels, it is important to understand the mechanism of the large swelling of the binary alloy and the mechanism of swelling suppression by titanium.

The mechanism of the large swelling has been studied by utilizing computer simulation [17]. It has been found experimentally that the large swelling is due to the large dislocation bias in these alloys. A strong correlation between solute atomic size factor and swelling has also been found experimentally; small solutes yield large swelling. In a recent theoretical treatment [18], the dislocation bias in bcc metals is larger than in fcc metals, contrary to the general belief, so far. Assuming this is true, the observed large swelling in V–5%Fe is inherent to the bcc lattice of this alloy, and there must be some mechanism which suppresses the bias and hence swelling in other low swelling alloys. By a series of molecular dynamics computer simulation, it has been found that in pure bcc iron and vanadium, self interstitial atoms (SIA) assume dumbbell configuration in a perfect lattice. In contrast, the stable configuration of SIA in the expansion side of, and close enough to an edge dislocation with $a/2 < 111 >$ Burgers vector is crowdion of which axis is parallel to the Burgers vector of the dislocation. This result is quite understandable since the energy of dumbbell configuration is only marginally lower than crowdion in an otherwise perfect lattice, and the compressive stress field of a crowdion is favorably accommodated in the expansion strain field of a dislocation. This conversion to crowdion has a decisive effect on the migration of the SIA. Since only one dimensional to-and-fro motion along its axis is possible for a crowdion, it can never approach the dislocation core; the path for the SIA approaching the dislocation is perpendicular to the axis of the crowdion. In other words, the dislocation bias is effectively suppressed by this conversion to crowdion.

This dislocation-aided conversion to crowdion does not occur in a simulated vanadium–iron alloy lattice; SIAs make reorientation and migration keeping essentially $< 110 >$ dumbbell configurations, and are eventually absorbed by the dislocation. This means that the inherent large dislocation bias is in full effect in vanadium with undersized solute of iron. The probability of oversized solutes associated with SIA is much less than undersized solutes and the frequency of interception from conversion to crowdion is accordingly much lower than the latter case. Thus, the large swelling found in vanadium-based binary alloys with undersized solutes are at least qualitatively understood in terms of the large inherent dislocation bias and bias suppression by dislocation-aided conversion to crowdion in pure metals, and the absence of this conversion in undersized solute alloys. The production bias theories might also be applied to explain this large swelling [19]. It appears, however, difficult to explain the correlation between atomic size factor and swelling in terms of the production bias model.

Titanium addition has been known to have a striking swelling suppression effect [20]. This effect has been also demonstrated for the V–5%Fe system [21]. In this study, 1%, 3% and 5% titanium was added to V–5%Fe alloy and irradiated in EBR-II at 600°C to 11.3 dpa. The swelling of V–5%Fe in this condition was 10.8% while it decreased down to zero by adding only 3% of titanium. In this respect, V–Cr–Ti alloys are in quite similar situation as V–Fe–Ti alloys; the high swelling V–Cr binary alloys is suppressed by titanium addition. The mechanism of swelling suppression by titanium has been argued based on the radiation-induced precipitation of titanium oxide which was supposed to act as vacancy absorber. From our recent data of positron annihilation, it seems more likely that titanium in solution is playing the major role in this swelling suppression. The mechanism of swelling and swelling suppression by titanium should be studied more closely in order to evaluate the performance and lifetime

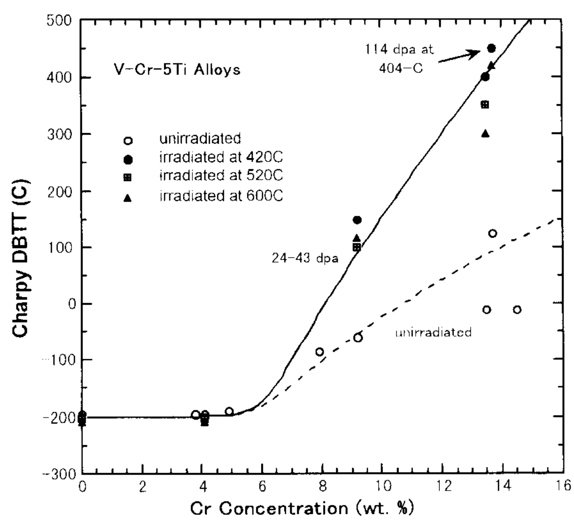

Fig. 5. DBTT of V–Cr–(4–5)Ti alloys before and after irradiation to 24–43 dpa at 420, 520 and 600°C as a function of chromium concentration.

of alloys in the V–4Cr–4Ti family from limited irradiation resources.

3.2. DBTT shift by irradiation

Shift of DBTT for V–4Cr–4Ti alloy by irradiation is rather small. Although the neutron irradiation data on this alloy is also limited to 34 dpa, the existing data shows virtual immunity of this alloy to irradiation damage. Fig. 5 shows DBTT of V–Cr–(4–5)Ti alloys before and after irradiation to 24–43 dpa at 420, 520 and 600°C as a function of chromium concentration [22]. Data on V–15Cr–5Ti after irradiation to 114 dpa are also included. The excellent resistance to radiation embrittlement of the alloys with less than 6% chromium content is clearly seen from this figure.

3.3. Effects of helium

The effects of helium generated during fusion neutron irradiation on void nucleation and high temperature ductility are well known at least qualitatively. Swelling may not be very severe in selected vanadium alloys even with a significant amount of helium, while at higher dpa levels and helium concentrations corresponding to several tens of MWa/m^2, the material response should be examined more closely. High temperature helium embrittlement may be the life-limiting process in vanadium alloys as well as in other materials, e.g. austenitic stainless steels. Furthermore, there is a slight concern about possible low temperature helium embrittlement, analogous to those allegedly-found in ferritic steels. Thus, in vanadium, there are several distinct issues associated with helium.

Since the type of helium clusters involving vacancies will very much dependent on the way how helium and displacement damage are introduced, the resulting effects on the microstructural development and mechanical properties will also depend on the mode of helium implantation, or He/dpa ratio. A new experimental method to generate helium in vanadium alloy specimens concurrently with neutron irradiation, a DHCE (dynamic helium charging experiment) technique [23] has been devised. In this technique, tritium-containing vanadium alloy specimens are irradiated in a fast reactor. By choosing appropriate concentration of tritium in the specimens, the decay of tritium into helium-3 during neutron irradiation can be adjusted to a desired value. This technique was applied for the first time to vanadium candidate alloys as well as model binary alloys in FFTF/MOTA [1] under the auspices of Japan/US collaboration. In this first run, the displacement damage level was 18 and 31 dpa and the helium

[1] Material Open Test Assembly in Fast Flux Test Facility at Westinghouse Hanford Company, Richland, Washington.

concentration ranged from a few appm to 177 appm depending on the alloy type.

Segregation of helium bubbles are usually observed along grain boundaries in specimens doped with helium by conventional tritium trick technique [24]. In specimens irradiated using DHCE technique, bubble segregation was not observed in all the alloys studied. Because of the very limited space available in the DHCE capsules, no Charpy specimens were irradiated and only a small number of tensile specimens were irradiated. Attempts were made to obtain toughness-related properties of vanadium alloys irradiated using DHCE technique by utilizing TEM disks and pieces of tensile specimens [25]. The fraction of fracture surface area showing ductile features was used as a measure of ductility. No brittle behavior was observed at temperatures above −150°C in V–4Cr–4Ti (ANL code: BL-47) specimens after irradiation by DHCE between 425 and 600°C to 18–31 dpa where He/dpa ratio was 0.4 to 4.2. Predominantly brittle fracture morphologies were found only at −196°C in some specimens irradiated to 31 dpa at 425°C. Thus, helium co-implanted under a condition similar to that in fusion reactors does not cause any additional reduction of ductility of candidate vanadium alloy at low temperatures. Although this conclusion is reasonably convincing, because of the nature of the non-standard test method used, it seems necessary to confirm this conclusion by using larger specimens appropriate for fracture toughness or Charpy tests in future irradiations.

Tensile tests of the same alloy after identical DHCE irradiations were also done at temperatures up to 600°C and the result is encouraging. Tensile tests conducted at the irradiation temperature showed little effects of helium on total elongation. Since significant ductility reduction sets in only above 600°C in many helium-charged vanadium alloys by tritium trick, this result indicates that no unforeseen embrittlement occurs under helium co-implantation condition of DHCE. Tensile specimens of the alloy V–5%Cr–5%Ti added with aluminum, silicon and yttrium were also irradiated in the same DHCE run [26]. For comparison, specimens doped with helium by tritium trick were also irradiated in the same irradiation environment. Fig. 6 summarizes the results of tensile tests conducted near 400°C; 0.2% proof stress, UTS and total elongation are shown. Data for control specimens and helium-doped specimens by cyclotron injection are also included in this figure. The total elongation of the specimens helium-injected by cyclotron to 50 appm decreased from 23% (control) to 10% despite rather small damage level of 0.02 dpa by the cyclotron implantation. By neutron irradiation at 407°C to 48.7 dpa, the total elongation is reduced to 5.8%, which is somewhat lower than the data obtained for the US alloy of V–4Cr–4Ti (BL-47). The reduction in total elongation was greatest, i.e. down to 4.2%, for specimens helium-pre-injected by tritium trick and neutron irradiated at 519°C to 42.9 dpa. DHCE at 430°C where helium concentration and dpa level was 177 appm and 24.4 dpa, respectively, resulted in the total elongation of 8.1%. Since the change in tensile property generally saturates at a damage level below 20 dpa, comparison of these data may be interpreted that the effect of dynamically charged helium on the ductility of V–5Cr–5Ti–SiAlY around 400°C is small.

There still remains an issue of high temperature creep life under fusion-relevant helium generation condition. This issue could be tackled by using a technique combining DHCE and pressurized tube creep. Post-irradiation test of DHCE specimens at high temperatures, e.g. 650 to 700°C could also provide useful information on this issue and some progress is expected in the near future. Although the damage level was limited to less than 20 dpa, it has been concluded that helium does not have significant effect on the low temperature ductility of V–4Cr–4Ti alloy.

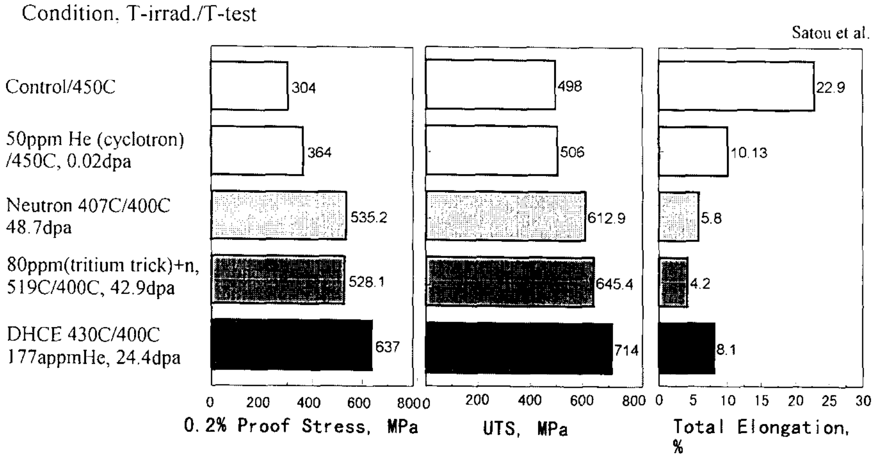

Fig. 6. Results of tensile tests conducted near 400°C; 0.2% proof stress, UTS and total elongation are shown. Data for control specimens and helium-doped specimens by cyclotron injection are also included in this figure.

4. Corrosion and compatibility

4.1. Insulator coating

The development of self-healing insulator coating on vanadium alloys may be one of the most challenging tasks in the fusion reactor material development. Substantial effort is being undertaken in the Argonne National Lab. Several different approaches have been examined to develop an AlN coating on vanadium substrate [27]. These approaches include PVD, CVD, reaction sputtering, coating with aluminum metal followed by nitriding, etc. The reaction deposited AlN layers in a partial pressure of high-purity N_2 gas were fairly compact and uniform thickness in the range of 8–12 μm. Although this coating disappeared during exposure to liquid lithium, a thermal-hardening treatment at 700–900°C made this coating adherent and kept intact even after exposure to liquid lithium. The resistivity of these coatings were measured and the resistivity was one to five times larger than the minimum value required for ITER application.

Calciumoxide has four orders of magnitude greater electrical resistivity than AlN and has a high thermodynamic stability in liquid lithium. Therefore, attempts were made to form CaO in situ in liquid lithium [28], where oxygen-enriched vanadium alloy was allowed to react with calcium contained in liquid lithium. Microcracks develop in the coating during cooling from temperatures above 400°C. After heating, the cracks exhibited self-healing within a 10 h period at 360°C and less than 1 h at 500°C, as evidenced by restoration of the resistivity to its original value. Thus, the development of self-healing insulator coating appears to have made a rather substantial progress in a relatively short period of time. Although there are a number of issues to be studied, e.g. the self-healing process under strong magnetic field and neutron and gamma-ray flux, the baseline characteristics of the self-healing coating, calcium oxide, in particular, appears to be very promising.

4.2. Welding

Since vanadium alloys generally have rather poor oxidation resistance, welding is usually done in inert atmosphere or in vacuum. Several welding methods, i.e., tungsten inert gas (TIG), electron beam (EB) and laser, have been applied to this material and all show good welding properties. Tensile properties, i.e. yield stress and total elongation are equal or greater in V–Cr–Ti ternary alloys after TIG welding [29]. EB and TIG methods have been applied to an alloy V–5Cr–5Ti (ANL code: BL63) [30]. This alloy has been known to show relatively high DBTT, i.e. about +80°C after annealing at 1125°C because of high impurity level. TIG welding in high purity argon resulted in a further increase in DBTT and the post weld annealing at 400°C intended to expel hydrogen did not improve the ductility. Annealing at 950°C resulted in a dramatic improvement of ductility; DBTT became even lower than that of base metal. Charpy test of the same alloy after EB welding showed surprisingly low DBTT, i.e. below −200°C. The reason is not clear yet, but the authors noted that post weld annealing may not be needed after EB welding. In summary, although welding procedures have not been optimized yet, existing data shows rather good welding properties of the vanadium alloys near V–4Cr–4Ti composition.

5. Summary and future perspective

A remarkable progress have been made in the research and development of vanadium alloys in recent years. A database centered on V–4Cr–4Ti is being accumulated rapidly and most of the acquired data demonstrate the superior properties of these alloys. Many of the engineering issues, e.g., manufacture, welding, insulator coatings, etc. are being successfully resolved. There are also substantial amount of fundamental studies supporting the alloy development and evaluation of alloy performance at very high damage levels, which is not easily accessible by experiments.

It is not clear, however, if this level of activity is going to be maintained in the future, when the ITER application of vanadium alloys is abandoned. Fusion material development is a long term program and continuous effort should be exerted to further develop vanadium alloys for fusion DEMO or power reactors. In order to secure resources for vanadium alloy R & D, flexibility in the use of this material should be enhanced. Although blanket designs using vanadium alloys usually adopt self-cooled liquid lithium breeder concept, it is also desirable to develop vanadium alloys which can be used with helium gas, light water, molten salts, or other coolant and/or breeding materials. Compatibility with coolants other than liquid lithium may be enhanced by cladding or by using surface coatings. The latter may be needed anyway in order to reduce MHD loss even when used together with liquid lithium coolant.

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