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Section 5. Vanadium alloys

Vanadium alloys – overview and recent results

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Abstract

This paper reviews recent progress in research on vanadium alloys with emphasis on V–4Cr–4Ti as a reference composition. New high purity V–4Cr–4Ti ingots and products (NIFS-HEATs) were made. The improved purity of the alloys made a practical demonstration of enhanced feasibility of recycling as a method of handling after use in fusion reactors. Significant progress has been made in the understanding of physical metallurgy of V–4Cr–4Ti and effects of O, N and C on the alloy properties such as low and high temperature mechanical properties, welding properties and low temperature irradiation effects, by means of including the comparison of various large heats and model alloys with different impurity levels. The effects of other trace impurities on some of the properties are also discussed. Other current efforts to characterize V–4Cr–4Ti, to improve its properties and to explore advanced vanadium alloys are reviewed. Issues remaining for the future investigations are discussed.

1. Introduction

Vanadium alloys are recognized as attractive candidate materials for neutron interactive structural components of fusion energy systems, because of their high temperature strength, high thermal stress factor, low activation property and so on [1–6]. High compatibility of vanadium alloys with liquid Li makes it possible to design concepts of liquid Li blanket using vanadium alloys, which have the potentiality of high thermodynamic efficiency, high reliability and availability because of high operation temperature and no need for neutron multiplier and ceramic breeder, both of which must be replaced periodically owing to burn-up [7].

Based on past results, a V–4Cr–4Ti ternary alloy is considered to be a leading candidate material. Recent efforts on vanadium alloy development have been focused on characterizing the existing V–4Cr–4Ti heats to establish performance limit and operation window. The efforts include the evaluation of low and high temperature mechanical properties with and without irradiation, improvement of the alloy properties by compositional and microstructural optimization, and exploration of new alloys by changing composition or applying new fabrication processes.

Following the large V–4Cr–4Ti ingot production organized by the US-DOE program (US-HEATs) [8,9], efforts have been made in Japan (NIFS) [10–12] and Russia (Bochvar) [13] to produce 30–200 kg V–4Cr–4Ti heats with improved purity. The results from new Japanese heats of V–4Cr–4Ti (NIFS-HEATs) show various benefits by reducing the level of oxygen and other trace impurities. International collaboration is in progress, in

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which those heats are being characterized by a number of research groups. In relation to the alloy production and characterization activity, understanding of fundamental aspects such as defect formation, evolution and recovery, impurity redistribution and precipitation during heat treatment, plastic deformation and irradiation has been enhanced. Technology for vanadium alloys as materials for components of fusion blanket systems is also in progress, e.g. welding and coating for mitigating magnetohydrodynamic (MHD) pressure drop and for corrosion protection, using the new alloy products.

This paper highlights recent progress in the fundamental and alloy performance knowledge on V–4Cr–4Ti. The MHD coating issues are not covered in this paper because they are overviewed in another paper [14]. The efforts to improve the alloy by modification of the composition or fabrication process and to explore advanced vanadium alloys are also reviewed. Current issues and research requirements are also discussed.

2. Alloy production, impurity level and low activation properties

In Japan, following the production of high purity 30 kg V–4Cr–4Ti (NIFS-HEAT-1) [10,11], a pair of V–4Cr–4Ti ingots with a total weight of 166 kg (NIFS-HEAT-2) were produced [12]. The chemical composition of NIFS-HEAT-1, NIFS-HEAT-2 and two large heats produced by the US program (US832665 [15] and US832864 [9]) are compared in Table 1. Plates and sheets with various thicknesses (26, 6.6, 4.0, 1.9, 1.0, 0.5 and 0.25 mm) and wires with diameters of 2 and 8 mm were fabricated from NIFS-HEATs and distributed to collaboration partners including those in the US, Russia and China as well as Japanese universities and research institutes.

The significant difference in the oxygen level between the NIFS-HEATs and US-HEATs makes it possible to investigate the oxygen effects on various properties. The fact that other impurity levels are lower in NIFS-HEATs would facilitate realistic consideration on potential low activation properties of vanadium alloys.

Recycling and reuse are a commonly accepted strategy for handling radioactive materials from fusion reactors [16]. It was repeatedly pointed out that vanadium alloys would have a potentiality of recycling and reuse [17,18]. Dolan and Butterworth [19] proposed recycling criteria by surface contact dose as Hands on (<0.01 mSv/h), Quasi-Hands-on (workers with added precautions for dose rate reduction) (<0.1 mSv/h), Quasi-Remote (workers in shielded cabs) (<1 mSv/h) and Full Remote (<10 mSv/h) and showed that extremely pure V–Cr–Ti ternary and removal of ^{42}Ar during remelting would facilitate the hands-on recycling. On the other hand, Grossbeck et al. [15] showed that V–4Cr–4Ti cannot meet the hands-on recycling criteria because of activation from Al, Nb, Ag and Mo even in the case of advanced laboratory processing. Cheng and Muroga [20] have described multiple-recycling criteria for V–4Cr–4Ti.

Fig. 1 shows the levels of Al, Nb, Ag and Mo in US-HEATs, NIFS-HEATs and raw V, Cr and Ti metals used for producing NIFS-HEATs. The impurity levels are shown as relative values to the recycling limit proposed by Dolan and Butterworth [19]. The figure shows that all alloys meet the criteria for full remote recycling, and NIFS-HEATs are close to meet the criteria for quasi-hands-on recycling. Thus NIFS-HEATs are considered to have made a practical demonstration of the feasibility of relatively economical recycling. Fig. 1 also implies the source of the impurities. Since Al is volatile and evaporates during the alloying processes, reduction of Al in raw metals will not be crucial. Higher Nb level in the alloys than those in raw metals implies that Nb was introduced from the facilities used for the alloying. A major source of Ag seems to be raw Cr metal. Thus, careful cleaning of the facility and selection of raw Cr

Table 1
Chemical composition of NIFS-HEAT-1, NIFS-HEAT-2, US832665 [14] and US832864 [9]

ID	C	O	N	B	Na	Mg	Al	Si	V	Cr	Mn	Fe	Ni	Cu
NIFS-HEAT-1	56	181	103	7	17	<1	119	280	Bal.	4.12	<1	80	13	4
NIFS-HEAT-2	69	148	122	5	<1	<1	59	270	Bal.	4.02	<1	49	7	2
US832665	170	330	100	3.7	0.01	0.17	355	785	Bal.	3.25	0.21	205	9.6	0.84
US832864	37	357	130	–	<2	<1	193	273	Bal.	3.8	–	228	–	<50
	As	Zr	Nb	P	S	Ca	Co	Ag	Sn	Sb	Ti	W	Mo	Ta
NIFS-HEAT-1	1	<10	1.4	16	9	3	2	<0.05	<1	<1	4.13	<1	23	58
NIFS-HEAT-2	<1	2.5	0.8	7	3	12	0.7	<0.05	<1	<1	3.98	<1	24	13
US832665	1.4	<46	60	33	16.5	<0.26	0.30	0.078	0.24	0.17	4.05	25	315	<19
US832864	–	–	106	<30	–	4	–	–	–	–	3.8	–	<50	

For the NIFS-HEATs metallic impurities were measured by GD-Mass analysis except Nb, Ag and Zr, which were measured by ICP analysis. (Cr, Ti: wt%, others: wppm).

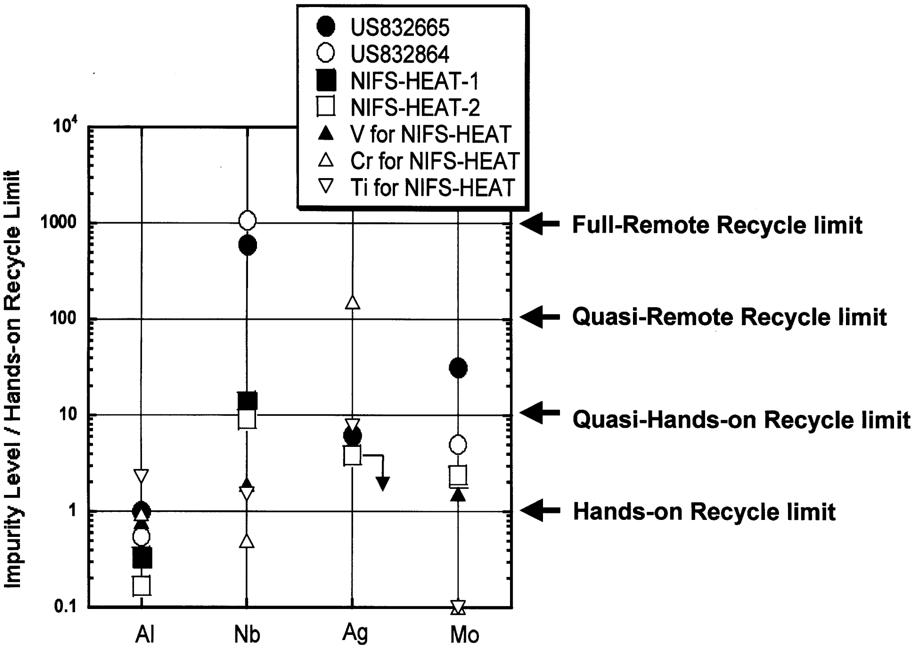

Fig. 1. Impurity levels of US-HEATs, NIFS-HEATs and the raw metals used for production of NIFS-HEATs, as relative to the hands-on recycle limits determined by Dolan and Butterworth [19], in which the limit for Al, Nb, Ag and Mo were 353, 0.1, 0.013 and 10 wppm, respectively. Note that Ag level of NIFS-HEATs is below the detection limit.

would increase further the potential of economical recycling of vanadium alloys.

Wu et al. [21] compared the dose rate after shutdown of reactors in the cases of NIFS-HEATs and US832665 and showed that Co in NIFS-HEATs will dominate the dose to 50 years. Thus reduction of Co level would be necessary if the cooling time before re-processing is to be <50 years.

3. Behavior of interstitial impurities and influence on properties

Although it was well recognized [22,23] that interstitial impurities such as O, N and C can have strong impacts on various properties of vanadium alloys, the research for V–4Cr–4Ti was limited partly because of the lack of specimens with systematic variation of the impurity levels. Fig. 2 shows the level of O and N in V–4Cr–4Ti alloys recently available for the property tests. Comparison of US and NIFS-HEATs makes it possible to carry out tests on oxygen effects on various properties including those which require massive specimens. Sixty gram V–4Cr–4Ti heats with various oxygen and nitrogen levels were made [24]. The tests using the heats were limited to small specimen experiments because of the limitation in volume. Also produced were extremely high purity alloy sheets made by the Zr-foil treatment method [25,26] in which the V–4Cr–4Ti sheet specimens were sandwiched with Zr foils and annealed for removing O, N and C impurities. The tests with these specimens were confined to microstructure observations and micro-indentations [27,28].

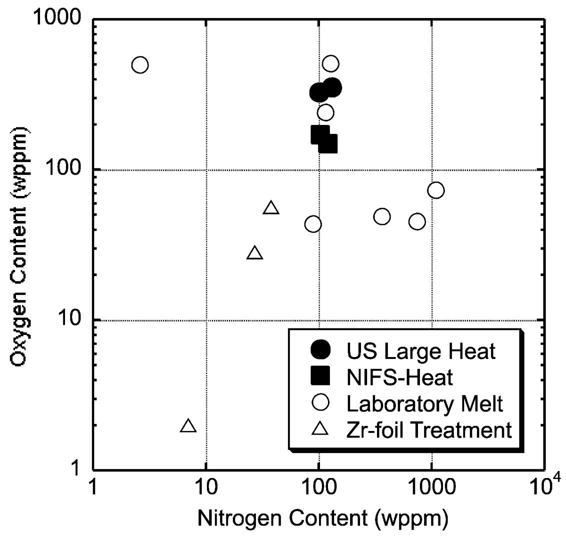

Fig. 2. O and N levels in V–4Cr–4Ti alloys available for recent investigations. Laboratory-melt V–4Cr–4Ti with variation of O and N levels [24] and Zr-foil treated specimens [27,28] are newly available.

The understanding of precipitation and impurity redistribution during plastic deformation and thermal treatment is crucial for optimizing the thermomechanical treatment conditions of alloys produced. Recent detailed characterization of US-HEATs [29,30] and NIFS-HEATs [31] provided comprehensive understanding of the precipitation behavior. Fig. 3 is summary of the precipitation during the fabrication of NIFS-HEAT-1 products [11,31]. Two types of precipitates, Ti-rich blocky precipitates, which were composed of Ti, C, N and O, and thin precipitates, which were composed of Ti, O and C (N was under detection limit), were observed in the matrix after heat treatment at 1273 K. The two types of precipitates were also observed in US-HEATs and their structural analysis has been reported by Hoelzer [32]. The blocky precipitates formed during the initial fabrication process. The precipitates aligned along the working direction during the forging and the rolling processes, forming band structures, and were stable above 1373 K. In the case of NIFS-HEAT-1, bands of small grains aligned along the rolling direction were observed at the annealing temperature of 1223 K. The grains became homogeneous at 1273 K. The thin precipitates were formed at about 973 K and disappeared at 1273–1373 K. At 1373 K, new precipitates, which were composed of V and C, were observed at grain boundaries. They seem to be formed as a result of redistribution of C induced by dissolution of the thin precipitates. The impact of the inhomogeneous microstructure on the mechanical properties was investigated by Donahue et al. [33].

The evaluation of various properties as a function of heat treatment conditions was carried out for NIFS-HEATs including tensile and impact properties, hardness and microstructures [31,34,35]. The optimum heat treatment temperature of 1223–1273 K was suggested.

Research with model V–4Cr–4Ti alloys doped with O and N provided information on the partitioning of O and N into the precipitates and matrix [24]. The density of the blocky precipitates and thin precipitates increased with N and O levels, respectively. Hardness after annealing at 1373 K, where only the blocky precipitates were observed in the matrix, increased to a certain extent with O level ($\simeq$4.5 HV/100 wppm O) but only very weakly with N level ($\simeq$0.9 HV/100 wppm N). These data suggest that most of N should be included in the blocky precipitates and stable to >1373 K. On the other hand, O should be in the matrix, the blocky and the thin precipitates, and the partitioning changes with the heat treatment. Thus, for the purpose of the property control of V–4Cr–4Ti, the level and distribution of N are not important but those of O are crucial. The fraction of the

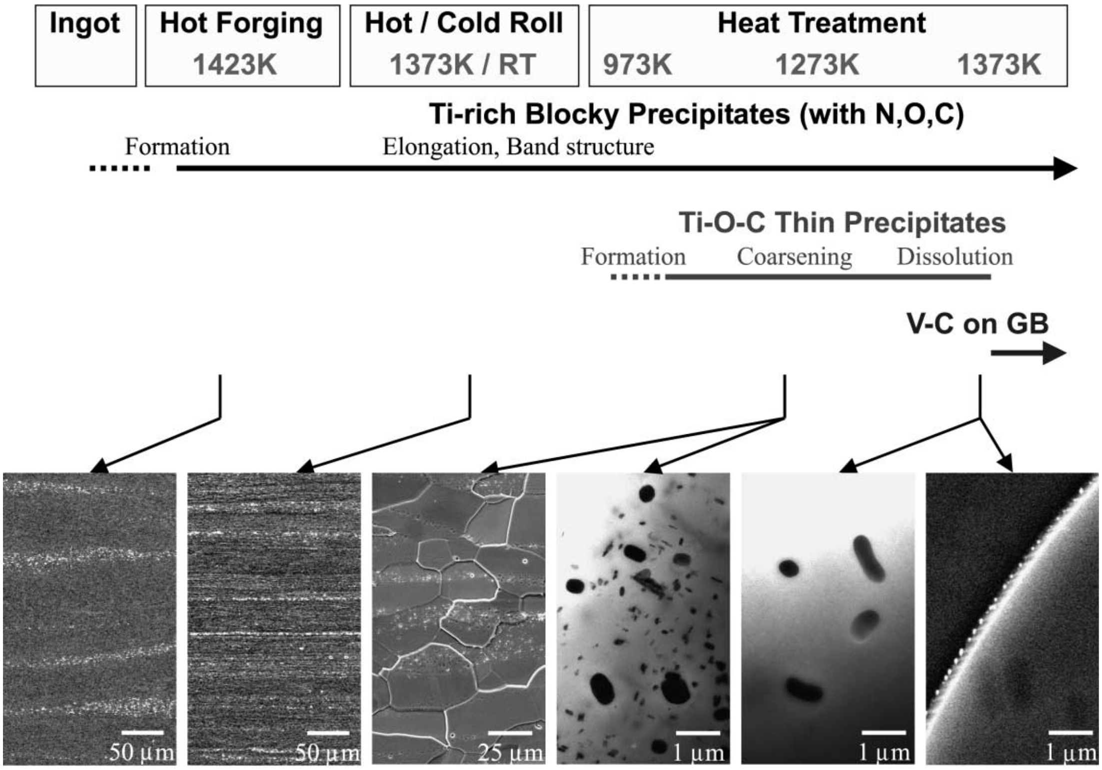

Fig. 3. Precipitate formation and distribution during the fabrication process of NIFS-HEAT-1 [11,31].

impurities in solution and in precipitates are in good agreement with the stereology measurements and calculation [36].

Fundamental information on the impurity distribution and interaction with solutes and dislocations is obtained by serrated flow in tensile deformation. Temperature and strain rate dependence of the flow showed that the serrated flow above 673 K is related to C and O, and above 773K to N [37]. Small serration height at 673 K for NIFS-HEAT-1 ($1 \sim 3$ MPa) relative to that for US832665 ($\simeq$9 MPa) was observed and attributed to the difference in O level [38].

One of the concerns by reducing the impurity levels in vanadium alloys is a possible reduction in strength at high temperature. The tensile tests of NIFS-HEAT-1 at RT to 1100 K showed that the tensile strength and the uniform elongation are near the lower and upper limit of the distribution bands of other V–4Cr–4Ti alloys, respectively. Thermal creep properties of NIFS-HEAT-1 were similar to those of other V–4Cr–4Ti alloys and $\simeq$200 MPa at the Larson–Miller parameter of 23×10^{-3} [38].

4. Welding, hydrogen embrittlement and oxidation

Recent efforts devoted to welding technology are focused on gus-tungsten-arc (GTA) welding and laser welding techniques. GTA is a suitable technique for joining large structural components. GTA welding technology for vanadium alloys made a significant progress recently by improvement of the atmospheric control [39]. The oxygen level in the weld metal was controlled by combined use of plates of NIFS-HEAT-1 or US8332665, and filler wire of NIFS-HEAT-1, US8332665 or a high purity model alloy (36 wppm O). The fact that the DBTT of the joint and the oxygen level in the weld metal had a clear positive relation motivated further purification of the alloys for improvement of the weld properties [40]. However, since a recent study showed upward shift of DBTT by thermal aging (e.g. 100 K shift by annealing at 1073 K for an hour) in the case of welding with low oxygen level [41], thermal aging effects should be investigated.

Laser welding is an attractive joining technology because of flexible, in-field, automated and remote operation with lenient need for environmental control, and small weldment and heat affected zone. Recent highlight of the laser welding study is the significant difference in impact properties of the joints of NIFS-HEAT-1 and US-HEATs [42]. Since the difference in oxygen level in the weldments is not large, contribution of other impurities (Si, Al, Mo and Fe, whose levels in NIFS-HEAT-1 are significantly lower than those in US-HEATs as shown in Table 1) is under examination [43].

The effect of the oxygen level on tensile properties of hydrogen-doped V–4Cr–4Ti was investigated [44,45]. Recently, new data on NIFS-HEAT-2 and SWIP-heat (O: 900 wppm) were reported by Chen [46]. In all cases, the total elongation of alloys with high oxygen levels (800–900 wppm) decreased rapidly with the increase in the hydrogen level and to $\simeq$2% at 100 wppm of hydrogen, and those with lower oxygen levels (158–670 wppm) were >20% at 200 wppm of hydrogen.

The oxidation kinetics and influence on mechanical properties were investigated for the purpose of evaluating the degradation of the pipe exterior and the impact of vacuum leak [47,48]. The data also can be applied to predict the performance in non-Li coolant systems such as He and water. The oxidation kinetics obeyed parabolic or linear kinetics depending on temperature and partial pressure of O. Since a surface oxide layer is not formed or, if formed, not protective against the internal oxidation, alloying with other oxide-formers is necessary for improvement. The addition of Si, Al or Y was shown to suppress significantly the weight gain during exposure to air above 873 K [49]. However, the addition of these elements were not effective in suppressing corrosion in water. An increase in Cr level was shown to be effective, instead [50].

5. Loss of elongation by irradiation at lower temperatures

A large increase in tensile strength and a drastic decrease in the elongation for V–4Cr–4Ti irradiated below $\simeq$700K was reported [51,52] and their mechanisms have been investigated. Based on microstructural characterization [53,54], the high density of defect clusters or radiation-induced precipitates were considered to be responsible for the formation of dislocation channels and the reduction in work-hardening capability.

Limited data suggest that addition of Al, Si and Y, which scavenge interstitial impurities, to V–4Cr–4Ti improved the uniform elongation after irradiation [55]. Microstructures after micro-indentation of ion irradiated NIFS-HEAT-1 in the as-received (O: 144 wppm, N: 124 wppm, C: 54 wppm) and the Zr-foil treated (O: 2 wppm, N: 6 wppm, C: 3 wppm) conditions showed less dislocation channels in the Zr-foil treated specimens at 473, 573 and 673 K [56]. The results support the idea that the impurity reduction may be a possible method to enhance radiation resistance in the low temperature regime. However, since the data were obtained by micro-indentation, other important parameters such as the uniform elongation were not derived.

The comparison of microstructures of ion-irradiated V–4Cr–4Ti in the as- received and as Zr-foil treated conditions showed that the dislocation loop density of the two specimens are almost the same, and that the precipitate density in Zr-foil treated specimen was significantly lower than that in the as-received specimen [28]. Thus a possible improvement of the tensile property of V–4Cr–4Ti by impurity reduction may occur via a reduction of the density of radiation-induced precipitates or of the impurity segregation at loops which might enhance barrier strength of the loops against dislocation glide during plastic deformation. Verification of the ideas using neutron irradiation is necessary.

The electron irradiation study using a high voltage electron microscope showed that the loop density in NIFS-HEAT-1 in the as-received and in the Zr-foil treated conditions were almost the same but significantly different from that of the specimens produced from another laboratory-melt V–4Cr–4Ti [27]. The result implies that trace impurities other than C, N and O would have dominant effects on the loop density. Since the comparison has not been made by either ion or neutron irradiations, it is not certain if this is only the case for electron irradiation.

6. Creep

Creep resistance is an important property for determining the maximum operation temperature of vanadium alloys. Thermal creep property of V–4Cr–4Ti has been investigated by uniaxial tensile [57] and biaxial pressurized tube [58] specimens. The data from the two methods are reasonably consistent with each other showing a 3.7–7 power law for the stress dependence of the creep rate, and suggesting climb-assisted processes. Since these creep experiments were carried out in vacuum, an introduction of oxygen into the specimens during the test is a common concern. The first series of the pressurized creep tests in Li are in progress [59].

Irradiation creep data are still very limited. Recent ATR and HFIR-12J irradiation experiments on pressurizedtube specimens yielded some creep data at 473, 573 and 773 K to a maximum dose of 6 dpa [60]. In contrast to the previous experiments, which showed bilinear strain rate with stress [61], the data showed a continuous increase of the strain with the stress. Microstructural examination of irradiated and unirradiated pressurized creep tubes were carried out for mechanistic investigation [62]. However, higher fluence data are necessary to reduce uncertainty in the creep behavior.

7. Beyond single phase V–4Cr–4Ti

Efforts are being made to improve the V–4Cr–4Ti alloy or to develop alternative alloys by changing minor, major compositions or fabrication processes. The common goal is to enhance the high temperature strength, oxidation resistance and irradiation resistance, which will enable to extend the operation windows of the alloys and to use the alloys in more oxidizing environments such as He cooled systems.

As already mentioned, the addition of Si, Al and Y on V–4Cr–4Ti has been reported to increase the uniform elongation after irradiation below $\simeq$700 K [55]. However, the data were limited to small specimen tests such as miniaturized tensile tests, since only small button samples were available. Recently high quality V–4Cr–4Ti, Si, Al, Y alloys of 2.5 kg each were made by the levitation melting technique. With the products, systematic impact property tests became possible. Some preliminary data showed good impact properties (lower DBTT and higher upper-shelf energy) before and after irradiation relative to those of the previous specimens produced by the button melting [63].

High strength vanadium alloys were made by addition of Y, O and N to vanadium followed by mechanical alloying and HIP [64]. The addition of Y, O and N was intended to enhance the mechanical properties by dispersion of Y_2O_3 and YN and scavenging O and N from the matrix. Alloys produced by optimization of the processes had small grains and homogeneously dispersed particles, and showed a higher tensile strength than those of NIFS-HEATs with moderate uniform elongation, both at room temperature and 1073 K [65].

The V–Ti–Al system has been examined as possible alloys yielding superior oxidation resistance and high temperature creep resistance [66]. The development is, however, still in the scoping phase.

8. Critical issues remaining for the future research

Although the long term strategy toward fusion materials development is different in different countries, it is commonly accepted that some critical issues should be resolved before proceeding to the advanced step of the development.

As for the remaining critical issues, it should be pointed out that lack of irradiation data is the most serious issue. In the low temperature regime, unified models of deformation and fracture are in progress [67]. Coupling of the models with irradiation experiments should be enhanced. The verification of the idea that a reduction of interstitial impurities would enhance radiation resistance at lower temperatures is necessary and will be carried out with NIFS-HEATs and other model alloys. Irradiation creep data are confined to low temperature and low dose. Data to high fluences at low temperatures, and to low and high fluences at higher temperatures are highly needed to evaluate the performance limit. He effects are the most uncertain factors for predicting the alloy performance under fusion conditions. Careful evaluation of available techniques for simulating helium effects and establishing experimental strategy is of vital importance. Since it was pointed out, as a possible problem of V–4Cr–4Ti used in fusion environments, that most of helium produced by transmutation cannot be maintained in the matrix but migrate to grain boundaries causing grain boundary embrittlement [66], characterization of V–4Cr–4Ti in helium generating irradiation environments is an immediate need.

A reduced impurity level of NIFS-HEATs demonstrated enhanced feasibility of recycling. As the next goal, careful cleaning of facilities to remove Nb and selection of Ag-free raw Cr would make it possible to demonstrate further enhanced feasibility of recycling.

Significant progress has been made in the understanding of the physical metallurgy of V–4Cr–4Ti, especially interstitial impurity effects during fabrication and the following thermal and mechanical treatments. The comparative characterization of V–4Cr–4Ti with different interstitial impurity levels has enhanced and will enhance further the understanding of the interstitial impurity effects on the alloy properties. This understanding should then be extended to apply for optimizing the alloy composition and the production process. It was reported that DBTT starts to increase with $\mathrm{Cr} + \mathrm{Ti}$ exceeding about 10% [23] for V–Cr–Ti ternary. The increase in DBTT was considered as a design tradeoff versus the higher tensile and creep strength achievable in the alloys containing higher $\mathrm{Cr} + \mathrm{Ti}$ levels. However, the optimum condition presently regarded as V–4Cr–4Ti was based on the data derived using binary or ternary alloys with oxygen levels exceeding 300 wppm. A reduction of the oxygen level may change the story and the optimum ternary composition may exist at $\mathrm{Cr} + \mathrm{Ti} > 10\%$. Care should be also given to other trace impurities which can have effects on some properties.

The interaction with environmental impurities and its influence on properties are still critical issues and should be addressed. Especially oxidation and synergistic of oxygen and hydrogen-induced property changes should further be investigated especially with respect to the effects on the first wall and blanket environmental conditions. Database on the impurity pick-up in various environments (vacuum with various O partial pressures, air, water, He and Flibe) would facilitate various designs using vanadium alloys. The fact that an environmental exposure up to four years in DIII-D at the peak service temperature of about 623 K did not cause change in tensile properties [68] is encouraging for the use of vanadium alloys in various positions of various systems including the present and near-term machines.

For technologies relevant to the component fabrication, such as working, joining and coating, the control of interstitial impurity is also known to be a key issue. Progress in the technologies was made with improved impurity control. Further improvement in the technology is essential for demonstrating that vanadium alloys are technologically feasible structural materials for fusion reactors. Insulating coating, although not discussed in this paper, is one of the most important technologies necessary to be developed. Irradiation effects on welding and coating have scarcely been investigated. Since introduction and redistribution of impurities would take place during the fabrication of welding and joining, it is of vital importance to check the irradiation effects. Timely acquisition of data on irradiation response would enhance the soundness of the development of welding and coating.

The development of alloys with different alloy compositions or fabrication processes from those of the present single phase V–4Cr–4Ti should be enhanced. The effort may extend the operation window of vanadium alloys and the possibility of using in oxidizing environments. Strengthening the relation with other alloy development programs is crucial for an efficient promotion of the vanadium alloy development. One of the possible partners would be hydrogen storage materials [69], in which the reduction of impurity levels is also a critical issue [70].

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