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Review of advances in development of vanadium alloys and MHD insulator coatings

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Abstract

Recent progress in the development of low activation vanadium alloys and MHD insulator coatings for a Li-self cooled blanket is reviewed. Research progress in vanadium alloys is highlighted by technology for fabricating creep tubes, comparison of thermal creep in vacuum and Li, understanding impurity transfer between vanadium alloys and Li and its impact on mechanical properties, behavior of hydrogen and hydrogen isotopes, low dose irradiation effects on weld joints, and exploration for advanced vanadium alloys. Major remaining issues for vanadium alloys are thermal and irradiation creep, helium effects on high-temperature mechanical properties and radiation effects on low-temperature fracture properties. Er_2O_3 showed good compatibility with Li, and is promising as a MHD insulator coating on vanadium alloys. Significant progress in coating technology for this material has been made. Recent efforts are focused on multi-layer and in-situ coatings. Tests under flowing lithium conditions with a temperature gradient are necessary for quantitative examination of coating performance.

1. Introduction

Vanadium alloys are attractive blanket structural materials for fusion power systems because of their low induced activation characteristics, high temperature strength and high thermal stress factors [1,2]. Recent efforts were focused on developing V–4Cr–4Ti alloy as a reference composition. Many of the critical issues for the vanadium alloys have been resolved by recent research. As a result, the feasibility of vanadium alloys as fusion blanket structural materials has been enhanced.

The leading blanket concept using vanadium alloys as a structural material is the Li-self cooled

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system. One of the critical materials issues for this system is the development of an insulator coating for mitigating the magneto-hydrodynamic (MHD) pressure drop [3,4]. Significant progress has been made in MHD coating development in recent years including identification of new candidate materials, improvement of the coating technology for vanadium alloy substrates and demonstration of long-term stability in Li.

This paper highlights recent progress with vanadium alloys and MHD coatings for use in a Li-self cooled blanket system with a vanadium alloy structure. Remaining critical issues are also discussed.

2. Recent progress in the development of vanadium alloys

2.1. Fabrication technology

In recent years, several large heats of V–4Cr–4Ti were produced in the US [5], Japan [6] and Russia [7], followed by fabrication of products such as thin and thick plates, rods and wires, and tubes and weld joints [2,8]. Through these efforts, fabrication technology for vanadium alloys has been largely advanced. The examination of microstructural changes during the processing of V–4Cr–4Ti ingots into various products showed that optimization of the size and distribution of Ti–CON precipitates is crucial for good mechanical properties [9]. Plates, sheets, rods and wires were fabricated while minimizing the impurity pickup and maintaining grain and precipitate sizes.

A recent highlight in this area is the effort to fabricate pressurized creep tubes in Japan [10] and the US [11]. Thin tubes, including those for pressurized creep tube specimens, were successfully fabricated while maintaining low impurity levels, fine grain size and straight distribution of precipitate bands by maintaining a constant reduction ratio between the intermediate heat treatments [12]. Through efforts for fabricating creep tubes including plugging of end caps, fine-scale electron beam welding, the technology of this alloy was advanced [10]. In the US, increase in the oxygen level during commercial-scale cold drawing and annealing to fabricate thin-walled tubing of V–4Cr–4Ti have been investigated as a function of the vacuum level during intermediate annealing [11].

Successful joining of V–4Cr–4Ti by gas tungsten arc (GTA) [13] and laser welding [14] methods was demonstrated without the necessity of a post weld heat treatment (PWHT). A low pressure plasma-spraying method was applied for coating W on V–4Cr–4Ti for use on plasma-facing surfaces [15].

2.2. Interactions with Li environment

The properties of vanadium alloys can be changed during exposure to the Li environment via transfer of impurities across the interface between Li and the vanadium alloys [16,17]. This might not be a concern if all inner surfaces of the alloy are covered with an insulating MHD ceramic coating. However, it is necessary to evaluate this effect to analyze data from reactor irradiation or thermal creep in a Li environment. Moreover, an idea to cover the MHD coating again with a thin vanadium or vanadium alloy layer was presented, as will be shown in Section 3, for the purpose of preventing liquid lithium from intruding into the cracks in the ceramics coating. For this case, the interaction of vanadium alloys with liquid lithium needs study.

It is known that the corrosion of vanadium alloys in liquid lithium is highly dependent on the alloy composition and lithium chemistry [16,17]. Recent thermal creep experiments, to be shown in the following section, provided opportunities to examine the change in chemistry and properties of V–4Cr–4Ti alloys by exposing coupon specimens to Li together with the creep tubes. Fig. 1 shows impurity contents, tensile stress and elongation of US (US-832665) and Japanese (NIFS-HEAT-2) heats of V–4Cr–4Ti as a function of exposure time to Li at 1073 K [18,19]. The original compositions of the heats are given in Ref. [2]. Difference in the rate of N enrichment and O depletion is seen between the two alloys. However, the total and the uniform elongation of the both alloys seem to saturate around 20% and 10%, respectively. The difference in the impurity level of the two alloys can be partly attributed to the difference in the thickness of the samples for impurity analyses, 3 mm for US-832665 and 0.25 mm for NIFS-HEAT-2. However, precipitate structure or initial impurity distribution could also be factors. Both alloys were heat treated at 1273 K, where precipitation was maximized for US-832665 but a significant fraction of the fine precipitates (mostly Ti–O) was dissolved in NIFS-HEAT-2 [20]. An increased level of solid solution oxygen is considered to result in the enhanced depletion of oxygen in NIFS-HEAT-2 during exposure to Li.

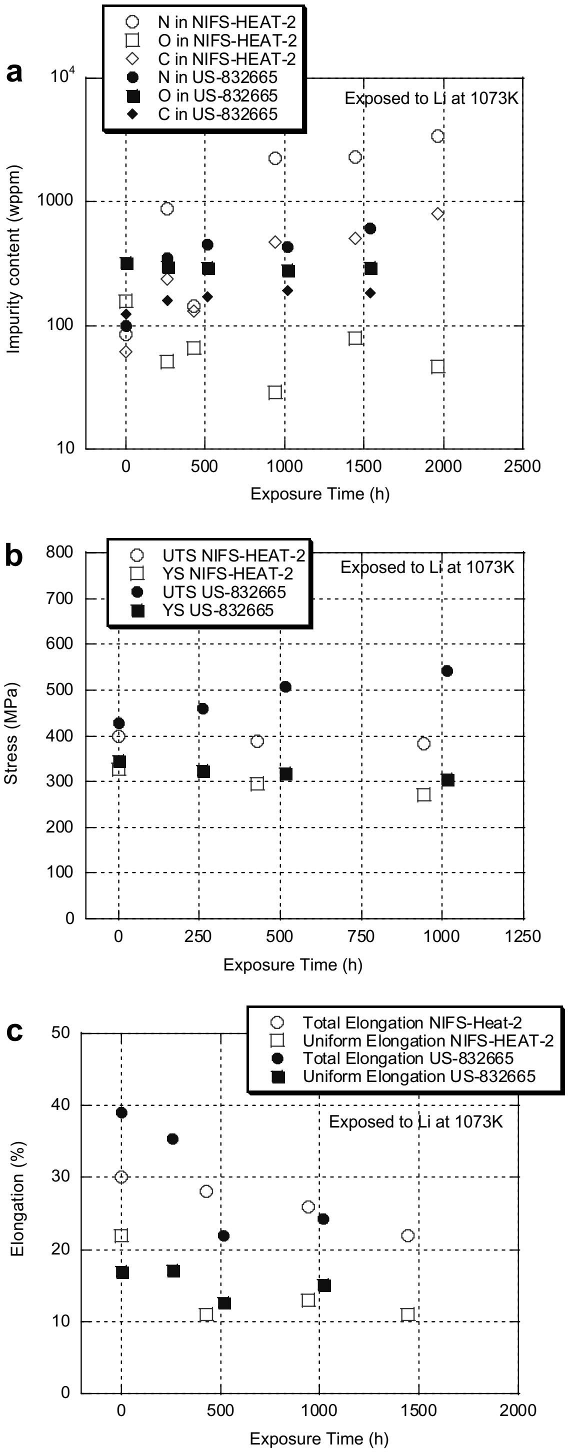

Fig. 1. (a) Impurity contents, (b) tensile stress and (c) elongation of Japanese (NIFS-HEAT-2) and US (US-832665) reference V–4Cr–4Ti alloys as a function of exposure time to molten Li at 1073 K [18,19]. It should be noted that the thickness of the samples for impurity analysis was 0.25 mm and 3 mm for NIFS-HEAT-2 and US-832665, respectively.

Analysis of partitioning of N and O in V–4Cr–4Ti showed that most of N was stored in globular precipitates and O is stored in globular and fine precipitates and matrix [21,22]. This is consistent with the results shown in Fig. 1 that large increase in N content did not cause remarkable hardening, because most of N transferred from Li to V–4Cr–4Ti is thought to be stored in the globular precipitates scarcely influencing the N level in matrix. Softening caused by loss of O in matrix seems to balance with or exceed the hardening by N.

Compositional dependence of the corrosion of V–*x*Cr–*y*Ti in Li was examined previously [16,17]. A new effort which included optimization of the Li removal technique after the exposure showed that V–(4–7)Cr–4Ti had good compatibility under these conditions [23].

2.3. Thermal creep

The progress in technology for fabricating high quality V–4Cr–4Ti creep tubes helped research on thermal creep performance of V–4Cr–4Ti alloys, including the effects of heat-to-heat variation, thermal and mechanical treatments, uni-axial (tensile) and bi-axial (creep tube) tests, and the test environment. Previous data on thermal creep in vacuum was summarized in Ref. [1]. A new apparatus for bi-axial creep testing in Li provided opportunities for examining creep deformation in vacuum and in Li [18]. However, the correlation of creep data is subject to the alloy heat and manufacturing processes as well as test methods and environments. Fig. 2 shows the comparison of the NIFS-HEAT-2 creep strain rate vs. creep strain data for tests in vacuum and Li environments at 1073 K, for the same batch of NIFS-HEAT-2 creep tubes [18,24]. The Figure clearly shows reduced strain rate in Li environments, which can be attributed to an increased level of N. However, as shown in Fig. 1, NIFS-HEAT-2 samples eventually showed no hardening but a little softening by exposure to Li. Further investigation is necessary for understanding the environmental effects on impurity redistribution and creep performance. A possible factor could be the surface hardening after exposure to Li [19], which is thought to be induced by N pick-up and could influence the creep processes.

Microstructural observations of the creep tube specimens tested at 1123 K showed free dislocations and dislocation cells at 100 MPa and 150 MPa, respectively [25]. This change of dislocation struc-

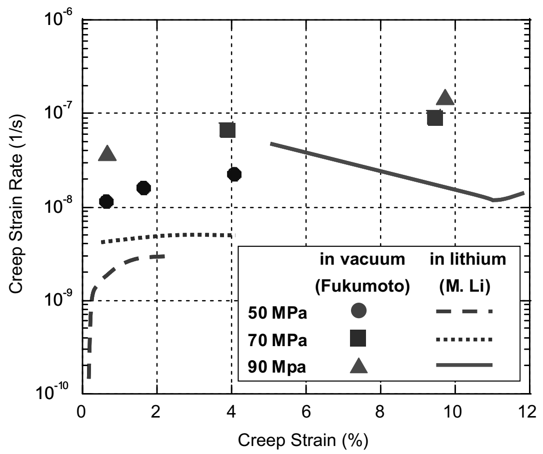

Fig. 2. Comparison of creep deformation in vacuum [24] and lithium [18] at 1073 K using the same batch of pressurized creep tubes fabricated with NIFS-HEAT-2 (V–4Cr–4Ti).

ture can cause the change in power law creep behavior [26].

2.4. Diffusion, retention and desorption of hydrogen and its isotopes

The behavior of hydrogen and its isotopes in vanadium alloys is a concern for tritium retention in the first wall and tritium inventory in the blanket. Deuterium ion implantation followed by thermal desorption showed that deuterium retention of V–4Cr–4Ti is much higher than and comparable to those of other PFC candidate materials (graphite and tungsten) at 380 K and 773 K, respectively [27]. A hydrogen absorption study showed that the rate of absorption is highly influenced by prior heat treatment, inducing Ti surface segregation. The formation of Ti oxide on the surface significantly suppressed the absorption rate [28].

Recent progress in detecting tritium by means of imaging plate (IP) enhanced the understanding on the tritium behavior in vanadium alloys: tritium is preferentially absorbed in Ti-rich precipitates [29]. The tritium distribution profile of a specimen after diffusion annealing measured with IP showed the tritium diffusion coefficient of V–4Cr–4Ti [30].

2.5. Radiation effects

Neutron irradiation can significantly influence the performance of vanadium alloys during operation in fusion reactors. Void swelling is known to be small if V is alloyed with Ti. Among the feasibility issues of radiation effects of vanadium alloys are loss of ductility at lower temperature, embrittlement enhanced by transmutated helium at high temperature, and irradiation creep at intermediate to high temperature.

The loss of uniform elongation of vanadium alloys irradiated at relatively low temperature (<673 K) was accompanied by a dislocation channel microstructure, implying flow localization during the deformation [31]. Although the mechanism of the flow localization is not well understood, it is inferred that interaction of dislocations with high densities of fine radiation-induced defect clusters is responsible. The defects were commonly observed as black-dots by TEM in V–4Cr–4Ti irradiated with neutrons below 673 K. Recent elemental analysis of the clusters by Atom Probe Elemental Analysis showed that they are enriched with Ti, O and TiO [32].

Helium embrittlement is a critical issue which may determine the upper temperature limit for vanadium alloys. The past experimental evaluation of the severity of helium effects varied from weak to very strong [1]. Since techniques to generate helium and displacement damage simultaneously are limited, recent progress in experimental evaluation of the helium effect is also limited. Clearly a 14 MeV neutron source is essential to evaluate the helium effects under fusion conditions.

Irradiation creep data are also very sparse. The data are limited to relatively low temperature and low dose. However, progress in irradiation creep tests is being made partly because of the progress in fabricating high-quality pressurized creep tube specimens with reduced impurity levels. Further data on irradiation creep at high temperature will be available from HFIR and JOYO reactor experiments.

Only low fluence irradiation data are available on weld joints. Welding results in dissolution of most precipitates, thus enhancing the level of solid-solution impurities. An enhanced defect cluster density and heterogeneous precipitate distribution in the weld metals by irradiations were reported [33]. The effects of post-irradiation annealing are shown in Fig. 3 [34]. Post-irradiation annealing showed a higher recovery temperature of the absorbed energy in the impact tests for the weld metal than that for the base metal. Microstructural observation during post-irradiation annealing showed that the dislocation loops formed by the irradiation were stable to

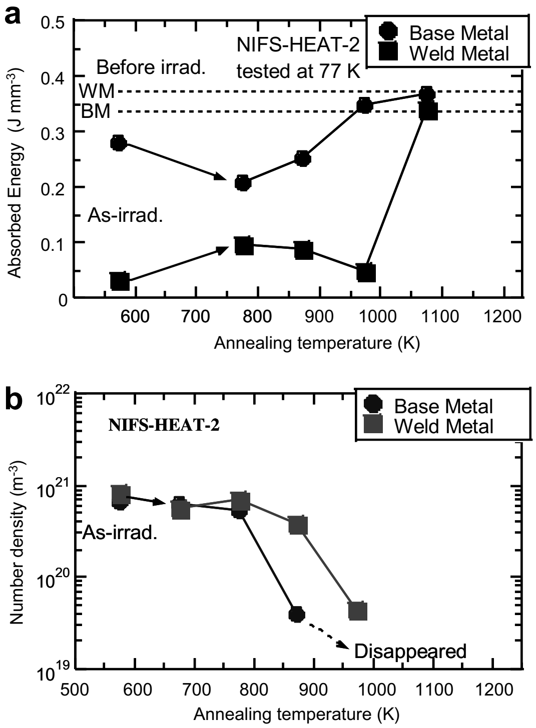

Fig. 3. (a) Absorbed energy in the impact test at 77 K and (b) number density of dislocation loops, for NIFS-HEAT-2 after neutron irradiation to 0.08 dpa, as a function of post-irradiation annealing temperature [34]. DBTT of the as-irradiated weld metal was 113 K.

a higher temperature in the weld metal, which can be attributed to the increased decoration of impurities at the loops in the weld metals.

2.6. Improvement of alloy properties

Generally larger ranges of operating temperature for structural materials allow us to design compact blanket systems with higher efficiency. Therefore, efforts have been made to develop advanced vanadium alloys with larger operating temperature windows, especially for potential use at higher temperature.

An increase in Cr level in V–Cr–Ti is known to increase high temperature strength, at the expense of a loss of ductility at low temperature [35]. A recent detailed survey in V–xCr–4Ti alloys showed that the strength at high temperature increases with small change in the DBTT with the Cr level to ~7% [36].

Addition of Y to V–4Cr–4Ti has been investigated for potential reduction of oxygen levels in the matrix and resulting increase in radiation resistance. Systematic fabrication of V–4Cr–4Ti–xY followed by impact tests showed an optimum level of 1.5% for Y [37]. Based on the results, a 15 kg ingot of V–4Cr–4Ti–1.5Y was made by levitation melting followed by characterization [38].

Mechanically alloyed V–Y alloys were fabricated and their irradiation response was examined. Fine grains and oxide dispersions increased high temperature strength and inhibited formation of interstitial loops in the matrix during neutron irradiation, because of the enhanced defect sinks [39]. Recent results showed that the further addition of Ti to V–Y enhances strength [40].

Other efforts to explore new alloying elements include W additions for suppressing hydrogen embrittlement susceptibility [41], and Zr for enhancing impurity gettering by co-precipitation [42]. Thermal and mechanical treatment conditions for forming a high density of fine precipitates [43] or oxides [44] have been explored for the purpose of enhancing high temperature strength of the conventional V–4Cr–4Ti alloys.

3. MHD coating development

Previously, CaO was regarded as the leading candidate for MHD coating material. However, recent research showed that it has a corrosion problem in liquid lithium at high temperature [45]. Thus, efforts in recent years were focused on identifying new candidate materials that can withstand corrosion by Li at high temperature and on developing coating technologies for the new candidate compositions.

Recent Li immersion tests of bulk specimens identified Er_2O_3 and Y_2O_3 as promising candidate ceramics, stable to approximately 1073 K in liquid lithium [3]. Feasibility of coating Er_2O_3 and Y_2O_3 on V–4Cr–4Ti was demonstrated by EB-PVD [3], arc source plasma deposition [46] and RF sputtering [47]. Especially, Er_2O_3 fabricated with arc source plasma deposition showed promising results as shown in Fig. 4 [48]. By deposition on a substrate at higher temperature, high crystalline Er_2O_3 coating was produced, which was shown to be stable in Li to 1000 h at 973 K.

Recent numerical estimates showed that tolerable crack density of the coating could be very low [49]. The result encouraged the development of double-layer coatings, i.e. an insulator layer covered by vanadium overlay, and revisiting of the in-situ

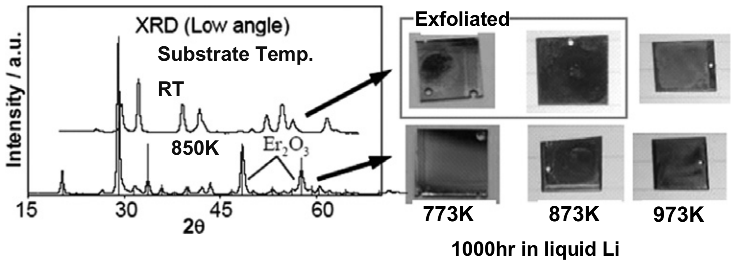

Fig. 4. Change of Er_2O_3 coating on V–4Cr–4Ti by exposure to Li at 773, 873 and 973 K for 1000 h. The coating was made using Arc Source Plasma Deposition method with substrate temperature at RT and 850 K. Crystalline structure of Er_2O_3 was observed only in the case of the high substrate temperature. Remarkable change was not observed in the coating at high substrate temperature after exposure to Li [48].

healing concept. Double layers with V on Er_2O_3 produced by EB-PVD showed satisfactory resistivity in molten Li to 873 K [50]. The composition of the alloys for the surface layer is being investigated for maximizing the compatibility with Li as discussed in Section 2.2.

The in situ coating method is a quite attractive technology because it will enable coating of complex surfaces after fabrication of components and has the potential to heal cracks in the coating without disassembling the component. In addition to the physical deposition methods, in situ coating with Er_2O_3 on V–4Cr–4Ti is being developed [51]. In this process, a Er_2O_3 thin insulating layer was formed on V–4Cr–4Ti during its exposure to liquid lithium by reaction of pre-charged oxygen in the vanadium alloy substrate and pre-doped Er in Li. Also demonstrated was the self-healing capability. The results showed significantly higher stability of the coating compared with the CaO in situ coating [52]. In the optimum condition for oxygen pre-charging to V–4Cr–4Ti a net-structure of Ti–O precipitates oriented in $\langle 100 \rangle$ directions was formed, and acts as a reservoir of oxygen for formation of the oxide coating. The mechanisms and kinetics for the formation of the structure were analyzed [53].

The degradation of electrical resistivity of the coating during irradiation can potentially deteriorate performance of the insulator coating. Radiation-induced conductivity of the candidate bulk ceramics and coated samples of Er_2O_3, Y_2O_3 and $CaZrO_3$ on V–4Cr–4Ti was evaluated during irradiation with D–T neutrons [54], fission neutrons [55] at ambient temperature, and γ-rays at high temperature [56]. Results showed that the change in the resistivity is not an issue for application in the fusion blanket.

Most of the recent corrosion characterizations of the coatings have been made by static immersion tests. Static tests are valuable for identifying candidates but not sufficient for verifying their suitability for use in blanket conditions. For example, erosion rate of the candidate coating ceramics in liquid lithium could be significantly different between static and flowing conditions. Thus, experiments in flowing Li with a temperature gradient and impurity control are essential for characterizing the performance of MHD coating in fusion blanket conditions. The efforts are being made to characterize the recent candidate coating systems in flowing Li [57,58].

4. Remaining critical issues and future directions

As a result of the recent progress in developing vanadium alloys, a limited number of critical issues remain. Available data on thermal and irradiation creep, helium effects on high temperature mechanical properties and radiation effects on fracture properties are insufficient. Especially for the helium effects, conclusive evaluation of the irradiation properties is possible only with the use of 14 MeV neutrons, motivating the construction of a 14 MeV neutron source.

The thermal creep tests under various conditions suggest that the current reference V–4Cr–4Ti alloys would allow a maximum operating temperature of ~973 K [1]. Potential ways to improve the creep performance of V–4Cr–4Ti would include an increase in Cr level (but not to a point where manufacturing ability and low temperature ductility are significantly degraded), applying thermal and mechanical treatments by which a high density of fine precipitates are formed, and adding new elements. Controlling impurity (C, O, N) levels and optimizing precipitate (Ti–CON) size and distribution are crucial for the mechanical properties of the alloy products and weldments at low and high temperatures. Systematic studies to optimize the microstructure and mechanical properties are necessary for enhancing the performance of vanadium alloys.

The tritium inventory in vanadium alloy blanket structures is thought to be a minor issue for Li-self cooled blanket because of low tritium partial pressure in Li. However, this might need to be re-examined taking irradiation effects and the impact of potential alloying elements such as Y into account. The tritium inventory would be a critical issue for the case using Li–Pb or Flibe as a breeding material because of higher tritium partial pressure. An effort is being made to explore a blanket concept using vanadium alloy and Flibe keeping low tritium partial pressure throughout the system [59].

Development of MHD insulator coating is a critical feasibility issue for the Li-self cooled blanket. Although there has been substantial progress in MHD coating, further intensive efforts are necessary for validating the performance of the current leading candidates, multi-layer coatings and in-situ coating concepts. Tests in flowing lithium conditions with a temperature gradient are necessary for quantitative evaluation of coating performance.

5. Summary

Vanadium alloys are attractive candidate structural materials for Li-self cooled breeding blankets of fusion reactors. Efforts in developing vanadium alloys have focused on V–4Cr–4Ti alloys as the reference material. Recent work has successfully resolved many of the critical issues and enhanced the feasibility of the alloys as fusion blanket structural materials. Research progress is highlighted by advances in fabricating creep tubes, joints and W-coating, comparison of thermal creep in vacuum and Li environments, understanding of impurity transfer between vanadium alloys and Li and its impact on mechanical properties, behavior of hydrogen and hydrogen isotopes, and exploration of advanced vanadium alloys for further enhancing high temperature strength, low temperature ductility or radiation resistance. Major remaining issues of vanadium alloys are thermal and irradiation creep, helium effects on high temperature mechanical properties and radiation effects on low temperature fracture properties. For conclusive characterization of the irradiation properties, use of International Fusion Materials Irradiation Facility (IFMIF) is essential as well as continuous use of fission reactors.

A new promising candidate of Er_2O_3 was identified for MHD insulator coating on vanadium alloys. It showed good compatibility with Li. Progress in coating technology for the new candidate material has been made. Also being advanced are development of two-layer coating with Er_2O_3 and vanadium overlay, and in-situ coating of Er_2O_3 coating for the purpose of protecting or healing cracked areas in the Er_2O_3 layer. Verifications of the performance of the candidates have been conducted mostly using static immersion tests. Tests in flowing Li conditions with a temperature gradient are necessary for quantitative examination of the performance.

Acknowledgements

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