

# **Characterization and Qualification of Neutron Radiation Effects – Summary of Japan-USA Joint Projects for 40 years –**

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## **Abstract:**

The Joint Projects under the Japan-USA Fusion Cooperation Program started in 1981 and continues for more than 40 years. In the Joint Projects, although wide range of fusion materials and engineering issues were covered, neutron radiation effects on fusion reactor materials have always been the major research emphases, and the neutron irradiation facilities in the US were jointly used by Japanese and US researchers. Japanese test facilities including neutron and charged particle irradiation facilities were complementarily used.

Initial focus of the Joint Projects was on fundamental fusion neutron radiation effects and irradiation correlation. Systematic comparison of fission and fusion radiation effects in comparable damage levels and the effects of transmutation-induced helium were investigated. The collaboration was then focused on the effect of dynamic irradiation effects in variable conditions. In addition to the relatively fundamental studies, the Joint Projects contributed largely to development of candidate materials such as RAFM steels, vanadium alloys, SiC/SiC composites, and tungsten alloys, by a mechanism-oriented approach. The Joint Projects also covered issues specific to materials application to fusion blankets and plasma-facing components including neutron radiation effects such as tritium retention and permeation of neutron-irradiated plasma-facing materials. Various irradiation technologies were developed and applied to the irradiation experiments including those for in-situ testing.

Considering that high energy neutron sources, such as A-FNS and IFMIF-DONES, now have high viability, the fundamental research supporting the neutron source programs are essential. The knowledge obtained through the Joint Projects is valuable and should be advanced for this purpose. To this end, it is of urgent necessity to launch an international scientific program focusing on fundamentals of fusion neutron radiation effects.

## **Key words:**

neutron radiation effects, irradiation correlation, low activation materials, transmutation

## 1. Introduction

Under the agreement between the Japanese and the US governments on the collaboration for research and development in the research field of energy and related subjects (1979), the Fusion Cooperation Program has been carried out as one of the key research activities. Since 1981, a series of “Joint Projects” have continuously been carried out in the Program changing the main subjects every 6 to 8 years, in addition to personnel exchanges and workshops. Presently the Joint Projects are operated under the implementing agreement between Japan-MEXT (The Ministry of Education, Culture, Sports, Science and Technology) and US-DOE (Department of Energy). For the Projects, the use of unique facilities in the US, which are not available in Japan, has been the necessary condition. There have been selection processes by the fusion community of both countries to start new Projects. Six Projects were carried out so far and now the seventh Project is going on. All the Joint Projects selected and carried out have targeted fusion materials and engineering [1].

The Joint Projects covered wide range of fusion materials and engineering issues including structural materials, functional materials, blankets, and plasma-facing components (PFC). However, throughout these Projects, neutron radiation effects on fusion reactor materials have always been the major research emphases. For this purpose, neutron irradiation facilities in the US were jointly used by Japanese and US researchers. Japanese infrastructure including neutron and charged particle irradiation facilities were complementarily used.

A summary of the past and ongoing projects is given in Table 1. Overviews of past Joint Projects are available for the RTNS-II [2], the FFTF/MOTA [3], the JUPITER [4], the JUPITER-II [5], the TITAN [6], and the PHENIX [7] Projects. The ongoing FRONTIER Project (2019-2024) was launched as of April 2019, in which the interface issues for the solid and liquid divertors are the major research targets [8].

It should be noted that the Joint Projects are science-oriented program, for which NIFS (National Institute for Fusion Science) and Universities are responsible on Japanese side, in contrast to Japan-USA collaboration program on HFIR [9], which is a more material development-oriented program carried out on Japanese side by QST (National Institute for Quantum and Radiological Science and Technology), formerly JAEA (Japan Atomic Energy Agency), under agreement with US-DOE. This program is hereafter referred as QST (JAEA)-DOE collaboration program.

This paper presents a historical overview of the Joint Projects but focuses on their contribution to characterization and qualification of neutron radiation effects of fusion reactor materials. The Joint Projects also covered non-neutron activities, such as fabrication and joining/coating technology of materials and components, high heat-flux and plasma-wall

interaction tests of diverter/first-wall materials and components, thermofluid of blankets and divertors, and corrosion and compatibility issues, which are reported in the overview articles.

## 2. Outline of the Joint Projects

In the RTNS-II Project, fusion neutron irradiation experiments using RTNS-II (Rotating Target Neutron Source-II), the world-largest D-T neutron source in the US, were carried out. Japanese advanced technologies such as electron microscopy and small specimen test technology were utilized to characterize the generation, accumulation, and recovery processes of the defects induced by the D-T neutron irradiation. Because the fluence of RTNS-II is high relative to other D-T neutron sources, although much lower than the necessary fluence for testing in fusion reactor conditions, comparison of fission and fusion neutron irradiation effects in comparable dose levels became possible, which largely enhanced irradiation correlation studies.

The main theme in the FFTF/MOTA Project was the effects of high-dose neutron irradiation up to 100 dpa (displacement per atom), which is the goal irradiation dose level of the fusion reactor materials, using FFTF/MOTA (Fast Flux Test Facility/Materials Open Test Assembly), the world-largest fast experimental reactor. It is intended to obtain understanding of the microstructural evolution and mechanical property change covering low dose to high dose irradiation regimes. The radiation performance of candidate materials including austenitic stainless steels, low activation ferritic steels, and vanadium alloys were studied as well as model alloys and pure metals. Also carried out were studies of the effect of transmutation-induced helium.

In the JUPITER Project, dynamic behavior of materials which appears only during irradiation, and change in material performance under varying conditions in response to steady and non-steady reactor operation were investigated using HFIR (High-Flux Isotope Reactor), which is a water-cooled mixed spectrum reactor having a high control and monitoring capability. Under this scope, in-situ measurements of the electrical and thermal properties of ceramic materials under irradiation and effects of varying temperature under irradiation on materials performance were studied. Also carried out were irradiation creep measurements. Various irradiation technologies to carry out these unprecedented experiments were developed.

The JUPITER-II Project was established to provide the scientific foundations for understanding the behavior of blanket structural materials interacting with breeding and cooling materials under conditions specific to fusion reactors, including interactive neutron irradiation effects. For this purpose, high-temperature coolant flow, heat and mass transport in blanket systems, and coolant chemistry and its interactions with the containing materials were

investigated. The results were integrated based on modeling and system design effort for advancing fusion blanket concepts.

The TITAN Project extended the scope of the JUPITER-II Project by including not only the blanket issues but also the interface issues among first wall, blanket, and recovery systems. A particular interest was placed on obtaining fundamental understanding for establishing tritium and thermofluid control. The experiments were designed for testing under conditions specific to fusion, such as intense irradiation, high heat/particle flux, and circulation of liquid breeders or coolants in a high magnetic field. The results were applied using integrated modeling to advance the design of tritium and heat control in magnetic and inertial fusion energy systems.

The goal of the PHENIX Project was to address the technical feasibility of the helium-cooled divertor concepts using tungsten as the armor material for fusion reactors. The research included heat transfer in helium-cooling conditions, material properties under high heat load and neutron irradiation, and tritium behavior in the PFC and first-wall conditions including neutron irradiation, using fission reactors, tritium test facilities, high heat flux and heat transfer test facilities. Also carried out were to develop computational models, predictive design, and analysis tools. The results were applied to the technical issues not only for helium-cooled divertors but also for various PFC and first-wall concepts.

The FRONTIER Project started in April 2019 to provide the scientific foundations for reaction dynamics in interfaces of plasma facing components for DEMO reactors. In this Project, modifications of microstructure, tritium transport properties, and mechanical strength of the interfaces, induced by neutron irradiation, are being investigated. Issues specific to liquid metal divertors such as corrosion under neutron irradiation is covered using a liquid-metal filled irradiation capsules for HFIR, to contribute to developing engineering models of advanced divertors.

Throughout these Joint Projects, neutron irradiation effects have been the core research activity, in which materials delivered from Japan and the US were irradiated in the neutron irradiation facilities in the US, and were partly shipped back to Japan, mainly to the Oarai Center of Tohoku University for Post-Irradiation Examinations (PIE) using Japanese unique infrastructure. From the TITAN Project, specimens irradiated in HFIR in ORNL (Oak Ridge National Laboratory) were shipped to STAR (Safety and Tritium Applied Research) facility in INL (Idaho National Laboratory) for tritium transfer and plasma exposure experiments.

### 3. Fundamentals of fusion neutron radiation effects and fission-fusion correlation

#### 3-1. Fundamentals of D-T neutron irradiation effects

In the RTNS-II Project, extensive irradiation experiments were carried out, showing characteristic defect production and microstructural evolution induced by high energy collision cascades produced by D-T neutrons. Observations with Transmission Electron Microscope (TEM) were extensively carried out. The examples include defect cluster formation in pure metals, model alloys, engineering alloys, and non-metals and interaction of defect clusters with pre-introduced defects, such as dislocation loops, line dislocations, and cavities. Efforts were made to correlate the structure of defect clusters with PKA (Primary Knock-on Atom) energy spectra [10,11].

A unique and aggressive effort made in the Project was to irradiate the samples at liquid helium or nitrogen temperature followed by TEM observation without raising the temperature (cryo-transfer experiment) [12,13]. In-situ annealing TEM observations showed that a recovery stage existed below room temperature in aluminum and copper and new defect clusters appeared in addition to disappearance of the existing defect clusters in copper. In addition, new defect clusters appeared during observation in TEM at low temperature with electrons much lower than the threshold energy for atomic displacement in nickel, copper, and aluminum, showing rearrangement of submicroscopic defects or defect clusters under low energy electron irradiation, in other words electron illumination.

Since the irradiation volume of RTNS-II was too small, development of small specimen test technology was highly motivated as a necessary tool to characterize mechanical property change by irradiation [14].

### 3-2. Strategy of irradiation correlation

In the early period of the Joint Projects, a strategy of irradiation correlation to predict high fluence fusion neutron irradiation effects was established as shown in Fig. 1 [3]. The correlation study was composed of three activities.

- (1) Low dose fission-fusion correlation by highly controlled irradiation experiments.
- (2) Low dose to high dose correlation using surrogate fission reactors including fast reactors and mixed-spectrum reactors.
- (3) High dose fission-fusion correlation with emphases on simulation of transmutation-induced helium effects.

It should be noted that charged particle irradiation experiments and modeling/simulation are the necessary components to reinforce the correlation effort.

Since RTNS-II was far more accessible and controllable than most of the nuclear reactors with respect to materials irradiation testing, highly controlled irradiation data could be derived, although irradiation dose and test volume were quite limited. Thus, for the fission-fusion correlation, the quality of fission neutron irradiation test needs to be enhanced, motivating

development of irradiation test rigs for precise low fluence fission neutron irradiation. For this purpose, efforts were made to increase quality of the irradiation data from JMTR (Japanese Materials Test Reactor). The advanced technology developed for irradiation in JMTR was widely used not only for fission-fusion correlation but also for low to high dose correlation, in-situ monitoring of the property changes under irradiation, and estimate of the temperature transient effect.

### 3-3. Irradiation correlation – low dose and low temperature

Using RTNS-II and some fission reactors, defect cluster evolution by fission and fusion neutrons were compared at low dose and low temperature in pure metals, model alloys, and engineering materials. In many materials, the cluster density was proportional to the neutron fluence, which is an indication that the visible clusters were produced directly from the displacement cascades. The fluence dependence of defect cluster accumulation then declined to a square root relation, indicating interaction among adjoining cascades [11]. There was no significant difference in the defect cluster density between D-T and fission neutron irradiations on dpa basis for copper, nickel, and austenitic steels in their low temperature regime ( $\sim 373$  K for copper and  $\sim 473$  K for nickel and steels) [15-17]. Fig. 2 is the comparison of the number density of defect clusters produced by irradiations with D-T neutrons in RTNS-II and fission neutrons in JMTR and OWR (Omega-West Reactor), a water-cooled experimental fission reactor, for nickel at 473 K [16] and AISI 316 SS at 363 K [17]. In both cases, the fluence dependence almost follows the square root relation.

D-T neutrons and fission neutrons produce PKAs having energies of several hundreds of keV and several tens of keV, respectively. Computer simulation showed that a 100~200 keV cascade is characterized as a collection of 5~20 keV cascades (subcascades) [18]. Thus, if defect clusters are directly formed from cascades with 5~20 keV, the number of defect clusters would become similar for D-T neutron and fission neutrons on dpa basis. However, this is not the case for heavy ions. Fig. 3 (a) compares D-T neutrons and high energy heavy ion irradiations in copper, showing significant difference in the defect cluster density on dpa basis [19]. Since large fraction of PKAs for the heavy ions are with the energy below 1 keV, the difference is understandable assuming only small fraction of PKAs having energy of 5~20 keV or higher can produce the defect clusters observable with TEM. Fig. 3 (b) compares the defect cluster density as a function of 20 keV subcascade density estimated by PKA energy spectra and computer simulations calculating the number of subcascades [19]. 20 keV was selected because a computer simulation showed that cascade splitting into subcascades starts at  $\sim 20$  keV in copper [20]. Fig. 3 shows much better correlation by the subcascade density than that by dpa. This is

an example showing that use of model-based parameters can enhance the correlation and thus predictability.

Fission-fusion correlation of mechanical properties for nickel, copper, iron, and austenitic stainless steels showed that there was no significant difference between D-T and fission neutrons on dpa basis with respect to yield strength. However, correlation of other mechanical property parameters such as uniform elongation and strain hardening exponent was not as simple as that of the yield strength [21].

### 3-4. Irradiation correlation – low dose and high temperature

At high temperature, defect clusters directly produced by collision cascades are unstable and rearranged to form lower density of extended defects such as dislocation loops and voids. These are induced by interaction of surviving freely migrating defects. Fig. 4 compares void swelling in nickel as a function of dpa comparing RTNS-II at 563 K and JMTR at 573 K [16], and RTNS-II at 723 K and JOYO (Japanese Fast Experimental Reactor) at 673 K or 773 K [22]. Clearly, void swelling by fusion neutrons is higher than that by the fission neutrons on dpa base. However, as shown in the figure, the damage rate (dpa/s) of the fusion neutron irradiation was about two order of magnitude lower than that of the fission neutron irradiation. This apparent difference in fission and fusion irradiation data is understandable because, with the increase in the damage rate, fraction of recombination of vacancies and interstitials increases suppressing their contribution to developing the secondary defects. Thus, in this case, the damage rate effects dominated over fission-fusion difference.

Similarly, the correlation can be misleading if damage rate effect is not appropriately included. An example is seen in ref. [23], in which simple collection of the void size data seemingly indicated highly scattered results. But considering the damage rate difference among the data, the cavity evolution process was understandable in a unified way. One of the successful correlation efforts is to compare dislocation loop density in Fe-Cr-Ni austenitic alloys produced by fission, fusion, and charged particle irradiations [24]. For these alloys, it is known that the density of dislocation loops reaches saturated values which are proportional to square root of the damage rate in a wide range of temperature.

It is to be noted that the damage rate effect is also a remaining issue for the lifetime estimate of long-service components such as reactor pressure vessel [25].

### 3-5. Transmutation effects at high dose

As is well known, the key issue for the fission-fusion correlation at high dose is the effect of transmutation-induced helium. In the Joint Projects, various means were applied to generate

helium during fission neutron irradiation including the following methods. The goal was to realize fusion relevant helium production rate ( $\sim 10$  He (ppm)/dpa).

(1)  $^{10}\text{B}$  addition to produce  $^4\text{He}$  during fission neutron irradiation in FFTF/MOTA.

(2) Dynamic Helium Charging Experiment (DHCE) in FFTF/MOTA for vanadium and vanadium alloys.

It should be noted that other helium production techniques were also used in the Joint Projects, such as doping of Fe or Ni isotopes and In-situ Helium Implantation using NiAl foils in HFIR. But, in these experiments, the US side took initiative and Japanese contribution was limited. They are not covered in this section.

### 3-5-1. Boron addition

$^{10}\text{B}$  addition is a popular way to produce helium during irradiation as result of nuclear reaction of  $^{10}\text{B}$  with thermal neutrons. Because the cross section of the reaction is too large,  $^{10}\text{B}$  burns rapidly when irradiated in light water reactors where the thermal neutron flux is high. Therefore, peripheral positions of FFTF/MOTA were selected as the irradiation locations of this experiment, where the thermal neutron flux was much lower than that in light water reactors but higher than that in the mid-core positions of FFTF/MOTA, enabling fusion-relevant and steady helium production rate during irradiation.

One of the issues for the  $^{10}\text{B}$  addition technique is that boron can have an effect to change the materials properties (non-nuclear effect) in addition to the transmutation-induced helium effect (nuclear effect). It is possible to estimate the nuclear and non-nuclear effects separately by comparing doping of  $^{10}\text{B}$  and  $^{\text{n}}\text{B}$  (natural boron: 20% $^{10}\text{B}$ ). Comparison of  $^{10}\text{B}$  and  $^{\text{n}}\text{B}$  doped nickel showed that boron addition can have two competing effects of enhancing void density as a nuclear effect and suppressing void density as a non-nuclear effect, and that the latter effect decreases with the irradiation temperature [26].

Boron addition experiments were carried out for austenitic steels [27], vanadium alloys [28], reduced activation martensitic alloys [29], and copper alloys [30], showing various helium-induced effects on microstructure and mechanical property changes.

### 3-5-2. Dynamic Helium Charging Experiment (DHCE)

DHCE is a quite unique method to produce helium during neutron irradiation [31]. A Li-filled capsule for irradiation in FFTF/MOTA was fabricated in which, in addition to test specimens, tritium-doped mother vanadium was inserted. With the start of irradiation, temperature of the capsule increases and the tritium doped in the mother vanadium is released and, through Li, doped to the test specimens. During irradiation,  $^3\text{He}$  is produced in the

specimens by the decay of the tritium, which make it possible to realize fusion relevant He/dpa ratio. This method can only be applied to the materials which have high tritium solubility. In this Project, most of the materials tested were vanadium and vanadium-base alloys.

DHCE was carried out at 698 to 773 K and ~30 dpa and He/dpa ratio of ~0.1 to ~8. A selected results are shown in Fig. 5 for V-4.1Cr-4.3Ti [32] and V-4.0Cr-4.8Ti-0.9Si-1.0Al-0.8Y [33] alloys. The effect of helium production is not remarkable in these conditions. Interestingly, the total elongation increased by the helium generation in both cases. On the other hand, the samples subjected to the Tritium Trick experiment, in which tritium was doped and helium was produced by the decay of the tritium before the neutron irradiation (pre-doping of helium), showed lower elongation after irradiation than the DHCE samples for V-4.0Cr-4.8Ti-0.9Si-1.0Al-0.8Y [33]. Microstructural observation showed that, in the Tritium Trick case, helium bubbles were formed on grain boundaries which deteriorated the mechanical properties. It is expected that helium produced by the Tritium Trick experiment mostly diffused to grain boundaries before the neutron irradiation because the density of the trapping site for helium in the matrix was very low without irradiation.

### 3-5-3 The impact of compositional change by transmutation

Neutron irradiation can generate solid transmutation products, as well as helium and hydrogen, changing the materials compositions. Important compositional changes for fusion-relevant materials are production of chromium from vanadium, production of nickel and zinc from copper, and production of rhenium and osmium from tungsten. Neutronics calculation shows that these transmutation reaction rates in fusion reactor conditions are higher than those in FFTF irradiation conditions but much lower than those in HFIR irradiation condition [34]. Thus, FFTF irradiation is insufficient for simulating the effects, but HFIR irradiation is an exaggerated simulation of the effects.

Assessments of the effect of solid transmutation products were carried out in the Joint Projects. For vanadium alloys, irradiation in HFIR showed elevation of DBTT (Ductile-Brittle Transition Temperature) which was highly enhanced by the increase in chromium level induced by transmutation [35]. Since the chromium level in the candidate vanadium alloys was optimized from the point of radiation resistance, the effect of the increase in chromium level on the materials properties in fusion reactor condition needs to be elaborately assessed. The microstructures in unalloyed copper irradiated in HFIR, in which ~3% nickel and zinc were formed during irradiation, and in pre-alloyed Cu-Ni, Cu-Zn and Cu-Ni-Zn irradiated in FFTF/MOTA, in which little compositional change occurred, were observed to compare the effects of steady and increasing level of nickel and zinc during irradiation [36]. In the PHENIX and the FRONTIER Projects, the effect of transmutation-induced rhenium and osmium in

tungsten is one of the key issues. In the Projects, comparison is being made between thermal neutron-shielded and un-shielded irradiations in HFIR to evaluate synergistic neutron irradiation and solid transmutation effects in fusion relevant conditions [7].

#### 4. Irradiation temperature control and variation effects

The trigger of this activity was the peculiar microstructures observed in various materials after irradiation in FFTF/MOTA. For example, Fe-Cr-Ni austenitic alloys irradiated at 873 K showed high density of small defect clusters including dislocation loops and SFT (Stacking Fault Tetrahedra), which are defects typically observed by irradiation at much lower temperature. Quantitative analysis showed that the temperature transient during the shut-down process of FFTF caused additional irradiation at lower temperature to the samples, which caused the high density of defect clusters [37]. This means such clusters can also be produced during the start-up process which may influence the succeeding microstructural evolution. This motivated new experimental research on the effect of short time negative excursion of irradiation temperature on materials performance.

The first effort was made in JMTR to compare irradiations with improved temperature control using auxiliary electric heaters and those with conventional temperature control by nuclear heating. In some materials and conditions, large difference was observed between the two cases [38].

In the JUPITER Project, the varying temperature irradiation experiments, which is designed to assess the effect of 10% negative excursion of the irradiation temperature, were carried out. Using the horizontal symmetry of HFIR irradiation condition by the mid-plane, a symmetrical comparison of the steady and the varying temperature irradiation was carried out for 613 K (steady) /613 K (90%)-498 K (10%) (varying), and 793 K (steady) /793 K (90%)-633 K (10%) (varying). The negative temperature excursion took place 8 times during the 8 irradiation cycles of HFIR [39].

Some examples of the comparison of the steady and the varying temperature irradiations are shown in Fig. 6. The low temperature excursion resulted in (a) low density void formation in Fe-16Cr-17Ni austenitic alloy [40], and (b) change from void lattice to random void distribution for pure V [41] at 613K, and (c) change from void dominance to phosphide precipitate dominance for Fe-16Cr-17Ni-0.024P austenitic alloy [40], (d) change from low density voids to high density small cavities for pure vanadium [42], and (e) a finer size and higher density of Ti-rich precipitates for V-4Cr-4Ti [43] at 793 K. The tensile tests showed that the change shown in (d) resulted in significant hardening and loss of elongation [44]. The totally different microstructures shown in (c) can be attributed to the difference in temperature for nucleation of the voids and the phosphide precipitates. Precipitate nucleation can take place

rapidly at 633K, which then grow at 793 K dominating the microstructure and suppressing void formation. The change from void lattice to random void distribution observed for pure V, shown in (b), is interesting and may suggest possible mechanism of the void lattice formation, which is still controversial.

## 5. Contribution to development of fusion candidate materials

The Joint Projects largely contributed to the development of fusion candidate materials, especially radiation-resistant blanket structural materials. Since the Joint Projects are science-oriented, contribution to the material development has been made mainly by mechanism-based approach.

In the early period of the Joint Projects, austenitic stainless steels, such as 316 stainless steels and their modified versions, were one of the major candidate materials not only for ITER but also for fusion powerplants. Vast efforts have been made to qualify these materials in worldwide fusion materials community [45] including those by the JAEA-DOE collaboration program [46]. The efforts included evaluation of the effects of transmutation-induced helium. In contrast, the Joint Projects focused on mechanism-oriented irradiation studies such as detailed microstructural characterization of the candidate materials irradiated to high dose [47], and fundamental studies based on model Fe-Cr-Ni ternaries. The studies using model ternaries included the effects of minor element addition such as Ti and P [48,49].

In 1980s, the major target of fusion blanket structural materials moved to “low activation materials” such as Fe-Cr-W based RAFM (Reduced-Activation Ferritic/Martensitic) steels, vanadium alloys, and SiC/SiC composites.

The FFTF/MOTA Project contributed to the initial RAFM development phase. Irradiation tests using FFTF, including those under the FFTF/MOTA Project, clearly showed that 7-10% Cr range was most resistant to DBTT shift caused by neutron irradiation [50], and Fe-Cr-W based low activation steels had less shift of DBTT induced by neutron irradiation relative to conventional Fe-Cr-Mo based steels [51]. JAEA and Japanese Universities have the candidate RAFM materials of F82H and JLF-1, respectively. Extensive irradiation studies for F82H have been carried out by the JAEA/QST-DOE collaboration program, for the purpose of constructing irradiation database necessary for DEMO design [52]. In the Joint Projects, on the other hand, characterization of F82H and JLF-1 was carried out focusing on mechanism of radiation embrittlement and dimensional stability using FFTF/MOTA and HFIR [53,54].

Because other low activation materials are still in immature state of development, the Joint Projects took initiative in the relevant fundamental studies, which highly contributed to enhancing feasibility of those materials. In the case of vanadium alloys, early period of the Joint Projects focused on fundamental radiation response of pure vanadium and binary vanadium

alloys. The research was highly motivated by the finding of ~160 % huge swelling of V-5Fe by irradiation in FFTF/MOTA to ~50 dpa [55]. Various binary and ternary vanadium alloys were investigated to research into the mechanism of the high swelling [56]. In the vanadium research community, V-4Cr-4Ti alloys are regarded as the leading candidates according to their radiation resistance. The major theme in the subsequent research included transmutation helium effects, which were covered in section 3-5-2, and interstitial impurity (C, N, and O) effects on radiation response of V-4Cr-4Ti alloys. Significant increase in uniform elongation after irradiation for V-4Cr-4Ti by the addition of Al, Si, and Y, which are known as getters of interstitial impurities in the matrix, suggested that the reduction of the interstitial impurities in solution enhances radiation resistance [57]. One of the unique efforts being made in the Joint Projects was the irradiation creep tests using pressurized creep tubes (PCTs). Comparison of Japanese and US candidate V-4Cr-4Ti alloys and comparison of liquid lithium and liquid sodium environments were carried out for the irradiation creep parameters [58].

One of the initial highlights of irradiation tests of SiC/SiC composites in the Joint Projects was the effect of SiC fibers. The bend strength of the composite using advanced high purity and stoichiometric fibers did not degrade to ~10 dpa, in contrast to rapid degradation of the composites using conventional fibers [59,60]. One of the later emphases on radiation tests in the Joint Projects was the estimate of radiation creep parameters. Studies on neutron irradiation creep of SiC have so far been extremely limited and insufficient mainly because of difficulty in fabricating creep test samples such as PCTs. The bend stress relaxation (BSR) method had been developed to examine the thermal creep behavior of refractory ceramic fibers. In the Joint Project, the BSR method was applied for studying irradiation creep of high purity and stoichiometric SiC, demonstrating that the BSR method is effective to determine the irradiation creep parameters in wide range of irradiation conditions [61].

Other low activation structural materials investigated in the Joint Projects include Fe-Cr-Mn austenitic alloys and oxide dispersion strengthened (ODS) steels. The major emphases of these alloys for irradiation experiments in the Joint Projects were phase stability under irradiation [62] and radiation effects of joint interfaces [6], respectively

Later, the interest of the Joint Projects extended to plasma-facing component materials such as tungsten and tungsten-base alloys and copper-base alloys. Especially irradiation tests of advanced tungsten alloys have been actively carried out from the PHENIX Project. Initial efforts focused on the mechanical property change of pure W [63], but recently powder-metallurgical-processed K-doped W, W-3%Re, and K-doped W- 3%Re, produced by Japanese Universities, were examined after neutron irradiation, and K-doped W-3%Re exhibited a ductile fracture [64].

This chapter focuses on the neutron irradiation tests in the Joint Projects contributing to the materials development. However, it should be noted that, under the Joint Project framework,

fabrication and joining technology of the candidate materials were also largely enhanced because the necessity to prepare high quality irradiation test samples highly motivated.

## 6. In-situ measurements of electrical, thermal, and optical properties

The objectives defined for the Joint Projects highly motivated development of new irradiation test technologies. Especially, in-situ measurement technology of functional properties such as electrical, thermal, and optical properties was highly enhanced in the Joint Projects.

RTNS-II was a highly assessable neutron source, relative to fission reactors, and was used for various in-situ tests. For example, loss of light transmission of optical fibers were measured in-situ during irradiation [65]. Also carried out was the critical temperature measurements of in-situ irradiated Nb<sub>3</sub>Sn superconductors [66]. For these studies complementary experiments were carried out using Japanese irradiation facilities such as KUR (Kyoto University Research Reactor).

The JUPITER Project focused on transient and variable effects of neutron irradiation, in which in-situ measurements of electrical and thermal properties under irradiation were among the major emphases. In-situ measurement of electrical conductivity of insulating ceramics were carried out using a Temperature Regulated In-Situ Test (TRIST)-ER facility. Irradiation of various Al<sub>2</sub>O<sub>3</sub> (alumina and sapphire) specimens to ~3 dpa showed that RIC (Radiation-Induced Conductivity) is proportional to the dose rate and RIED (Radiation Induced Electrical Degradation) would not be a problem in fusion reactor relevant conditions [67]. In-situ thermal conductivity measurement of SiC and SiC/SiC composites were carried out using TRIST-TC1 facility as a collaboration with the JAEA-DOE collaboration program [68]. The experiments showed that the thermal conductivity undergo a rapid reduction with irradiation followed by saturation.

## 7. Neutron radiation effects on plasma-materials interactions

The major objective of this activity is to understand the effects of neutron irradiation on tritium behavior (diffusion, trapping, desorption, etc.) in plasma-facing materials. The TITAN Project started shipping of HFIR-irradiated samples from ORNL to INL followed by various experiments in STAR facility, including hydrogen isotope retention and desorption during or after exposure to plasma in TPE (Tritium Plasma Experiment). In addition to Thermal Desorption Spectroscopy (TDS) and permeation measurements, Nuclear Reaction Analysis (NRA) was carried out for depth profiling [69].

There are many experimental parameters, such as temperature and fluence of neutron irradiation, temperature, flux, energy, time, and isotopic ratio of plasma exposure, in addition to materials parameters. This activity has been continued through the TITAN, the PHENIX, and the FRONTIER Projects. Through these Projects, understanding on the contribution of these parameters to the hydrogen isotope retention has progressed. Initial experiments showed that significant level of deep traps for hydrogen isotopes were formed already by neutron irradiation at 323 K to 0.025 dpa [70]. The following research showed that the deuterium retention increased with the neutron fluence, decreased with neutron irradiation temperature and plasma exposure temperature. Detailed analysis of the TDS spectra in comparison with those by heavy ion irradiation made it possible to identify the deuterium trapping species [71].

This activity is continuing extending its scope including the effects of interfaces in the plasma-facing materials on retention and permeation of hydrogen isotopes.

## 8. Modeling of radiation effects

The irradiation experiments carried out in the Joint Projects have always been in close collaboration with the modeling and computer simulation activity. Since there are many cases showing that the modeling and computer simulation reinforced the irradiation experiments, only a limited examples are reported in this paper.

The examples include binary-collision computer simulation of cascade damage structure to support the low-dose fission-fusion correlation [20,72] (also shown in Fig. 3 [19]), molecular dynamics computer simulation on the helium-vacancy interactions to support prediction of helium effects on void evolution [73], kinetic models to investigate the influence of He/dpa and damage rate, which are critically important parameters for irradiation tests but difficult to investigate systematically [74], and microstructural evolution under varying temperature to support the varying temperature irradiation experiments [75,76]. In these cases, computer simulation provided overall picture of the parametric dependence of materials performance based on limited number of comparisons with experimental results. In the radiation-tritium synergism studies, the computer code TMAP4 provided information on characteristics of hydrogen isotope traps produced by neutron irradiations, which made it possible to evaluate tritium transport in materials irradiated with neutrons [77].

In the Joint Projects, efforts have been made to integrate theory, modeling, and experiments for the materials performance systematically. Among the examples are Integrated Focus on Fundamental Studies-Vanadium Initiative (IFFS-VI) in the JUPITER Project [78], Material System Modeling Subtask in the JUPITER-II Project [5], and System Integration Modeling Task in the TITAN Project [6].

## 9. Summary and present issues

The Joint Projects under the Japan-USA Fusion Cooperation Program have a history exceeding 40 years. They have covered wide range of fusion materials and engineering issues. However, throughout these Projects, neutron radiation effects on materials were always the main research emphases. Although the key activity has been the joint use of the unique neutron irradiation facilities in the US, complementary use of Japanese neutron and charged particle irradiation facilities and the use of Japanese unique materials and technology enhanced the collaboration both in quality and quantity.

Initial focus of the Projects was placed on fundamental fusion neutron radiation effects and irradiation correlation. Systematic comparison of fission and fusion radiation effects in comparable damage levels and simulation of helium production were carried out. The collaboration was then focused on the dynamic materials behavior under irradiation in variable conditions and obtained various unprecedented results.

The Joint Projects contributed largely to the fusion materials development adopting science-oriented approaches, especially for RAFM, vanadium alloys, SiC/SiC composites, and tungsten alloys. In addition to the characterization of materials performance under irradiation, issues specific to their application to fusion blankets and PFC were investigated such as heat transfer, compatibility, and tritium retention/permeation influenced by neutron irradiation. The collaboration also enhanced materials fabrication, joining, and coating technology, and various unique irradiation and post-irradiation test technology.

This overview places emphases on relatively early collaborations on fundamental radiation effects and irradiation correlation studies because, (1) although materials development and blanket/PFC oriented researches carried out in the later collaborations are being succeeded by the present research activity, the past knowledge on fundamental radiation effects and irradiation correlation is seemingly not well inherited, and (2) the key facilities of the early collaborations, such as RTNS-II and FFTF, were shut down soon after the Joint Projects ended, and the opportunity to conduct D-T and fast neutron irradiation of these dose levels was lost afterwards.

In the present fusion materials development programs, viability to construct and use high energy neutron sources, such as A-FNS [79] and IFMIF-DONES [80], is increasing more than ever as necessary tools for DEMO development. However, considering the limited volume and time available for irradiation with the neutron sources, it is obvious that the support from fundamental understanding on the materials performance, based on the fission neutron and other irradiation means, is essential to obtain high predictability of materials performance in DEMO conditions. This is also a reason for the focusing of this paper.

Development of neutron sources for fusion materials irradiation has a very long history. Materials scientists committed themselves largely to the neutron source programs organizing the users' community [81]. Fundamental understandings obtained through the Japan-US Joint Projects, as listed below, have been a driving force of the commitment.

- (1) Irradiation correlation with respect to primary damage formation and evolution (RTNS-II Project)
- (2) Low fluence – high fluence correlation including damage rate effect (FFTF/MOTA Project)
- (3) Transmutation effect (simulation and prediction) (FFTF/MOTA and JUPITER Projects)
- (4) Temperature transient effects and temperature control requirements (JUPITER Project)
- (5) Issues specific to fusion blanket and PFC applications (JUPITER-II, TITAN, PHENIX, and FRONTIER Projects)
- (6) Characteristics of the candidate materials, and irradiation and post-irradiation test technology (all Projects)

But unfortunately, it appears that the commitment of materials scientists to the neutron source programs at present is not as large as it was years ago. Now that A-FNS and IFMIF-DONES programs are making significant progress, reinforcing the users' community is of urgent necessity. For this purpose, it is of vital importance to launch an international scientific program focusing on fundamentals of fusion neutron radiation effects.

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Table 1. Summary of Japan-USA Joint Projects.

RTNS-II, Rotating Target Neutron Source-II; FFTF, Fast Flux Test Facility; EBR-II, Experimental Breeder Reactor-II; HFIR, High Flux Isotope Reactor; ATR, Advanced Test Reactor; HFBR, High Flux Beam Reactor; STAR, Safety and Tritium Applied Research; MTOR, Magneto-Thermofluid Omnibus Research Facility; TPE, Tritium Plasma Experiment; PISCES, Plasma Interactive Surface Component Experimental Station; PAL, Plasma-Arc Lamp facility; LAMDA, Low Activation Materials Development and Analysis; LLNL, Lawrence Livermore National Laboratory; PNNL, Pacific Northwest National Laboratory; ANL, Argonne National Laboratory; ORNL, Oak Ridge National Laboratory; INL, Idaho National Laboratory; BNL, Brookhaven National Laboratory; UCLA, University of California, Los Angeles; UCSD, University of California, San Diego; GIT, Georgia Institute of Technology.

Project Name	Period	Test Facilities	Core Subjects
RTNS-II	1981~1986	RTNS-II (LLNL)	Low dose D-T neutron irradiation Defect production and accumulation Fission-fusion correlation Microstructure-mechanical property correlation
FFTF/MOTA	1987~1994	FFTF (PNNL) EBR-II (ANL-West)	High fluence neutron irradiation Dimensional stability Mechanical properties Transmutation effects
JUPITER	1995~2000	HFIR (ORNL) ATR (INL) HFBR (BNL)	Transient and variable effects of irradiation Temperature variation effects In-situ conductivity of ceramics Irradiation creep
JUPITER-II	2001~2006	HFIR (ORNL) STAR (INL) MTOR (UCLA)	Key issues for advanced blankets Molten-salt blanket Li/V-alloy blanket He-SiC/SiC blanket
TITAN	2007~2012	HFIR (ORNL) TPE/STAR (INL) MTOR (UCLA) PISCES (UCSD)	Mass and heat transfer in first wall and blanket Tritium retention and transfer Thermofluid in magnetic field Coating & joining integrity under irradiation
PHENIX	2013~2018	HFIR (ORNL) PAL (ORNL) TPE (INL) He Loop (GIT)	Feasibility of He-cooled divertors Heat transfer in He-cooled divertors Radiation effects on tritium transport in PFC Radiation effects on tungsten alloys
FRONTIER	2019~2024	HFIR (ORNL) LAMDA (ORNL) TPE (INL)	Interface issues for solid and liquid divertors Radiation effects on interface properties Tritium transfer across interfaces Compatibility for liquid divertors

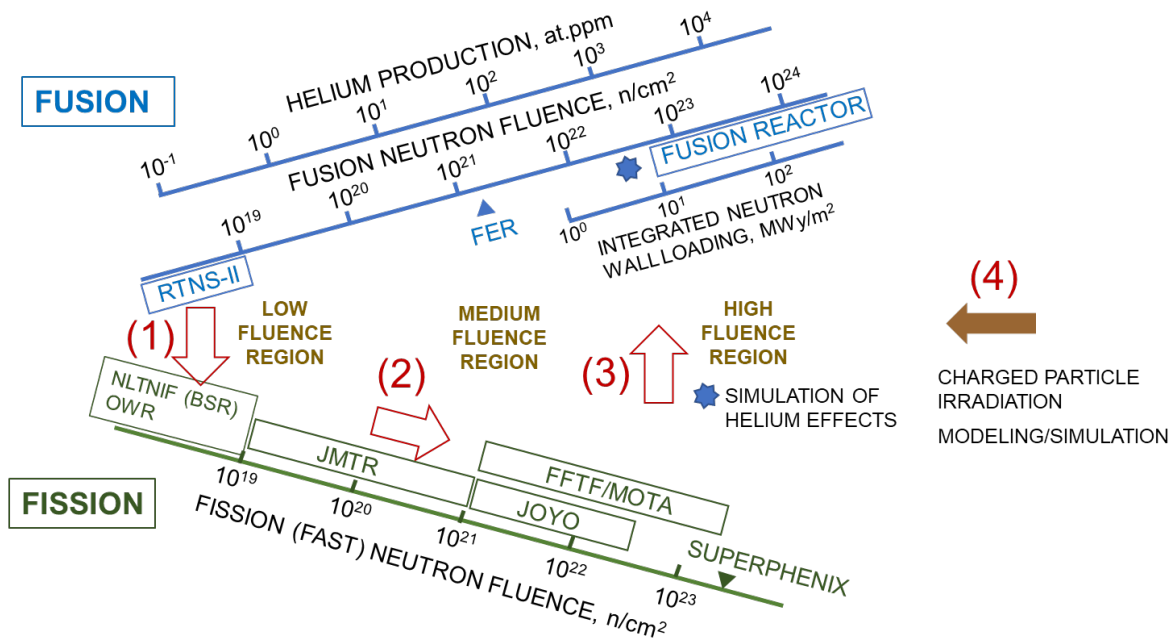


Fig. 1. A strategy of irradiation correlation to predict high fluence fusion neutron irradiation effects [3].

- (1) Fission-fusion correlation in low dose.
- (2) Low dose-high dose correlation with fission neutrons.
- (3) Fission-fusion correlation in high dose focusing on helium effects.
- (4) Support from charged particle irradiation and modeling/simulation.

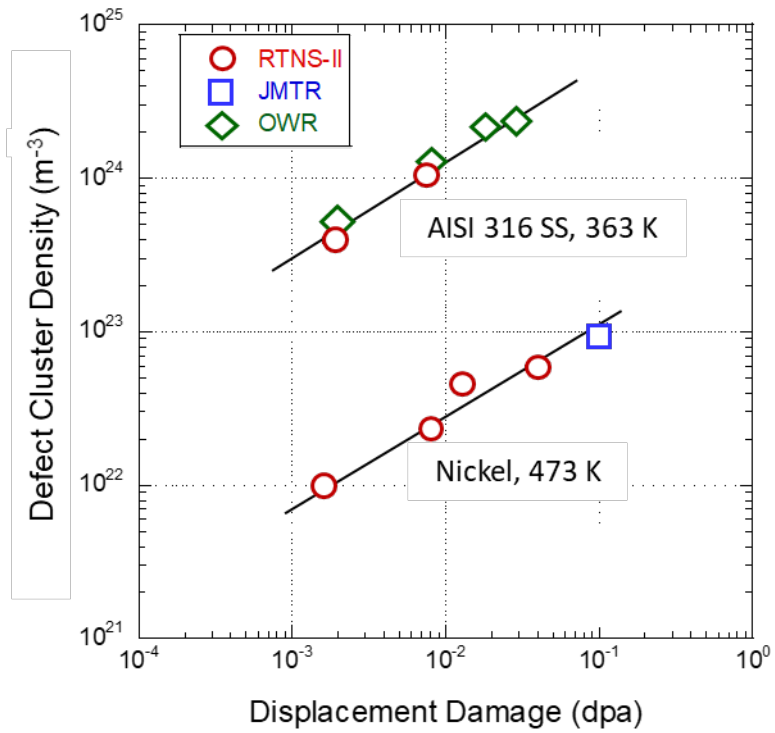


Fig. 2. Comparison of the number density of defect clusters produced by irradiations with D-T neutrons in RTNS-II and fission neutrons in JMTR and OWR for nickel at 473 K [16] and AISI 316 SS at 363 K [17].

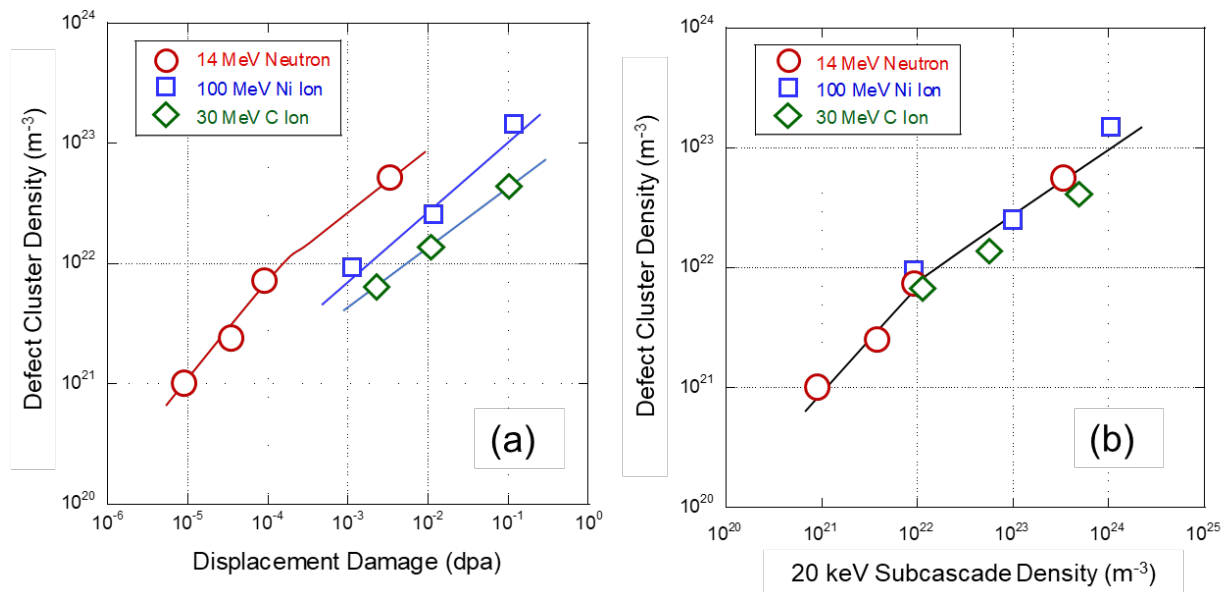


Fig. 3. Comparison of the number density of defect clusters produced by irradiations with D-T neutrons and high energy heavy ions in copper (a) as a function of dpa, and (b) as a function of 20 keV subcascade density estimated based on PKA energy spectra and computer simulation [19].

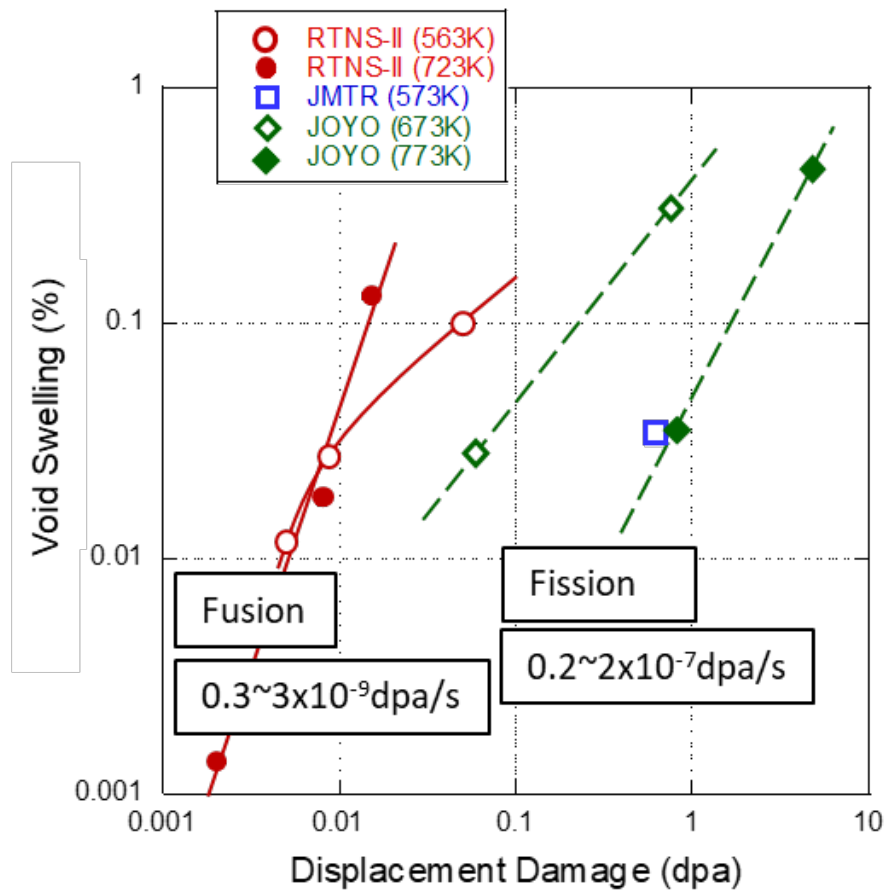


Fig. 4. Void swelling in nickel as a function of dpa, comparing RTNS-II at 563K and JMTR at 573 K [16], and RTNS-II at 723 K and JOYO at 673 K or 773 K [22].

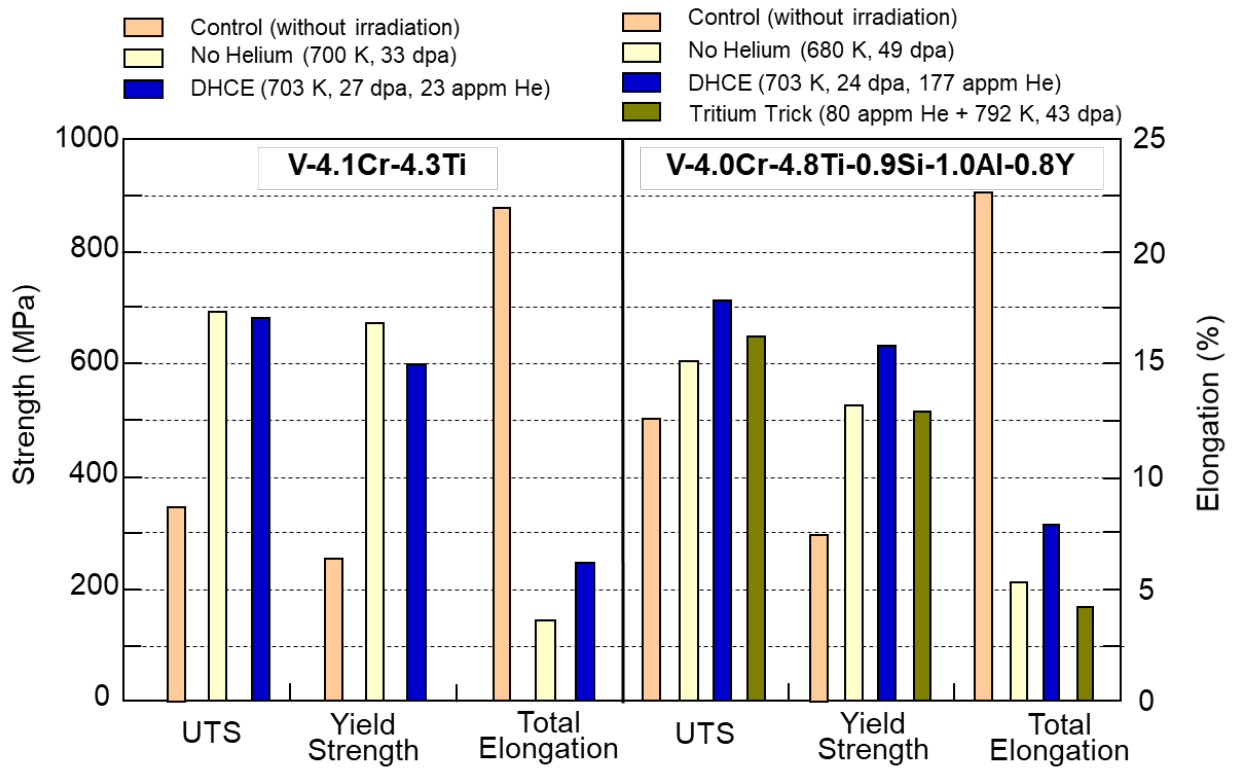


Fig. 5. Tensile properties of V-4.1Cr-4.3Ti alloy [32] and V-4.0Cr-4.8Ti-0.9Si-1.0Al-0.8Y alloy [33] irradiated in FFTF/MOTA without and with dynamic helium charging (DHCE). V-4.0Cr-4.8Ti-0.9Si-1.0Al-0.8Y alloy includes Tritium Trick experiment data, in which helium was charged before irradiation. The test temperatures were 693~698 K for V-4.1Cr-4.3Ti alloy, and 723 K for the control and 673 K for the others for V-4.0Cr-4.8Ti-0.9Si-1.0Al-0.8Y alloy.

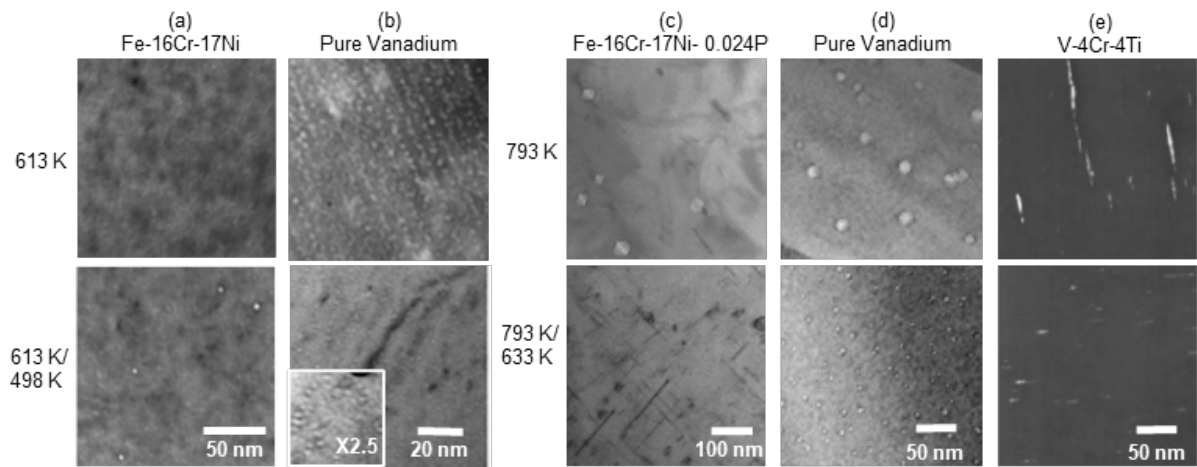


Fig. 6. Examples of the comparison of steady and varying temperature irradiations for Fe-16Cr-17Ni and Fe-16Cr-17Ni-0.024P austenitic alloys [40], pure vanadium [41,42], and V-4Cr-4Ti [43]. In this experiment, symmetrical comparisons of the steady and the varying temperature irradiation was carried out in HFIR for 613 K (steady) /613 K (90%)-498 K (10%) (varying), and 793 K (steady) /793 K (90%)-633 K (10%) (varying). The negative temperature excursion took place 8 times during the 8 irradiation cycles of HFIR [39]. The damage level was  $\sim 4$  dpa for the stainless steel.