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**Environmental effects on irradiation creep behavior of highly purified V-4Cr-4Ti alloys (NIFS-Heats) irradiated by neutrons**

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## **Environmental effects on irradiation creep behavior of highly purified**

### **V-4Cr-4Ti alloys (NIFS-Heats) irradiated by neutrons**

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#### **Abstract**

In order to investigate the effect of the environment on the irradiation creep properties of highly purified V-4Cr-4Ti alloys, neutron irradiation experiments with sodium-enclosed irradiation capsules in Joyo and lithium-enclosed irradiation capsules in HFIR-17J were carried out using pressurized creep tubes (PCTs).

It was found that the creep strain rate exhibited a linear relationship with the effective stress up to 150 MPa at 458°C and 598°C in the Joyo irradiation experiments. For HFIR-17J irradiation at 425°C, the creep strain rate also exhibited a linear relationship with the effective stress up to 150 MPa. The activation energy of the irradiation creep and irradiation creep stress factor were estimated to be 46 kJ/mol·K and 1 to 2, respectively. No significant difference in the irradiation creep behavior between liquid-sodium and liquid-lithium environments could be seen.

## Introduction

Vanadium alloys are candidate materials for use in the blanket structure of fusion reactors because of their potentially high operating temperatures [1]. However, the knowledge about their creep properties under neutron irradiation at fusion-relevant high temperatures is limited, and there are uncertainties that may have influenced the existing results, such as mass transfer of interstitial impurities from the liquid-metal coolant. In addition, little is known about the mechanical properties of vanadium alloys during neutron irradiation at high temperatures. Recently, material irradiation technology in liquid-metal environments has been developed and irradiation creep experiments on vanadium alloys in nuclear piles have been carried out [2,3]. Environmental and irradiation effects on creep deformation in irradiation creep experiments under neutron irradiation should be distinguished independently in order to understand the essential irradiation creep process in a nuclear pile.

The objective of this study was to investigate the effect of the environment on the irradiation creep properties of NIFS-Heat2 highly purified V-4Cr-4Ti alloys NIFS-Heat irradiated by neutrons. To carry out the irradiation creep tests in a nuclear pile, a Na-enclosed irradiation rig in Joyo and a Li-enclosed irradiation rig in HFIR-17J were used for pressurized creep tubes (PCTs) made of NIFS-Heat alloys with their impurity contents suppressed during manufacture.

## Experimental Procedure

The V-4Cr-4Ti alloy used in this study was produced by NIFS and Taiyo Koko Co. and is designated NIFS-Heat2 [4]. Details of the tubing and fabrication processes of the PCTs have been reported elsewhere [5-7].

The neutron irradiations for the irradiation creep experiments were performed in HFIR and Joyo. Detailed information about the experimental procedure for Joyo irradiation is given in ref. [2]. The MNTR-01 and -02 irradiations in Joyo were conducted at reflector positions (6<sup>th</sup> row position) for material irradiation examination in the MK-III core configuration of the Joyo reactor. The irradiation period was two Joyo irradiation cycles, equivalent to 116.7 days (2802 h). The irradiation temperatures were calculated to be 458°C and 598°C. The neutron doses ( $E > 0.1 \text{ MeV}$ ) were  $2.4 \times 10^{25} \text{ n/m}^2$  for irradiation at 458°C and  $6.7 \times 10^{25} \text{ n/m}^2$  for 598°C irradiation. The damage levels corresponding to pure vanadium were estimated to be 1.8 dpa at 458°C and 5.0 dpa at 598°C.

After removing the sodium remnant on the PCTs and cleaning their surface [2], their dimensional changes were measured with a KEYENCE LM-7030MT precision laser profilometer to an accuracy of 1  $\mu\text{m}$  for the outer-diameter measurement.

A plan of the irradiation creep experiment in a lithium environment was designed and

executed as part of the program of the JUPITER-II project. The HFIR-17J irradiations were carried out mainly with vanadium alloy specimens in direct contact with lithium at 450°C, 600°C, and 700°C in a europium-shielded RB position with five cycles for a total of 9930 MWD [3]. Irradiation creep experiments in the HFIR-17J were performed at 425 and 600°C with PCTs in lithium-containing capsules irradiated to 3.7 dpa.

After neutron irradiation, the lithium-filled capsules were disassembled and the specimens were rinsed with ammonia solution in order to remove completely the molten lithium remnant. Dimensional changes of the PCTs were measured at ORNL with a Z-mike precision laser profilometer. Detailed additional information about the laser profilometry is given elsewhere [8]

## Results

### Irradiation creep properties in a sodium environment in Joyo

Fig.1

Fig. 1 shows plots of the effective irradiation creep strain as a function of applied stress for highly purified V-4Cr-4Ti alloys irradiated in Joyo in a sodium environment [2]. It is apparent that the irradiation creep strain of the annealed alloys increases in proportion to the applied stress. It is assumed that the dependence of the applied stress on irradiation creep strain obeys the power-law equation  $\dot{\epsilon} \propto \sigma^n$ , where  $n$  is the creep stress factor. The values of  $n$  were estimated to be  $1.7 \pm 0.3$  for irradiation at 458°C and  $1.1 \pm 0.2$  for the 598°C irradiation of the annealed alloys.

Cold-worked V-4Cr-4Ti alloys had a larger irradiation creep strain than annealed ones. In previous studies of thermal creep in V-4Cr-4Ti alloys, the creep strain rate of cold-worked V-4Cr-4Ti alloys was much smaller than that of annealed V-4Cr-4Ti alloys in the temperature range 700 to 800°C [6-10]. The tendency of the behavior of irradiation creep deformation in V-4Cr-4Ti alloys was different from that of thermal creep deformation at the point of deformation processing.

#### **Irradiation creep properties in a lithium environment in HFIR-17J**

Fig. 2 shows plots of the effective irradiation creep strain as a function of applied stress for highly purified V-4Cr-4Ti alloys irradiated at 425°C in HFIR-17J in a lithium environment. The irradiation creep strain of the V-4Cr-4Ti alloys increases with the applied stress. The values of  $n$  for the NIFS-Heat2 alloys were about 1 for the 425°C irradiation. The data of creep strain for #832662USA V-4Cr-4Ti alloys and NIFS-Heat alloy irradiated in Joyo at 458°C are superimposed in Fig.2. The #832665USA V-4Cr-4Ti alloys were irradiated under the same conditions as the NIFS-Heat alloys and the damage level was almost the same as for NIFS-Heat alloys irradiated in HFIR-17J. From Fig. 2, at 425°C  $n$  was about 1 below a stress level of 150 MPa. A small transition in the stress dependence of irradiation creep strain can be seen at 150–160 MPa.

Fig.2

Fig. 3 shows plots of the effective irradiation creep strain as a function of applied stress for

Fig.3

highly purified V-4Cr-4Ti alloys irradiated at 600°C in HFIR-17J in a lithium environment. The creep strain data of NIFS-Heat alloy irradiated at 600°C were scattered. When the data were converted into the creep strain rate per damage level (dpa) and were plotted with the data of NIFS-Heat alloys irradiated in Joyo at 600°C, the data of longitudinal axis of Fig. 3 were obtained. From the data for 600°C irradiation in Fig. 3, the higher value of the irradiation creep strain rate per 2 dpa for NIFS-Heat alloy irradiated in HFIR-17J shows the same tendency of stress dependence of the irradiation creep strain rate as for NIFS-Heat alloys irradiated in Joyo. The lower value of the irradiation creep strain for NIFS-Heat irradiated in HFIR-17J may be caused by defects and malfunction of the creep tube during manufacture, such as helium leakage at pinholes on the surface of the PCTs and unexpected uptake of interstitial impurities during the tubing process. It was considered that data at 30 MPa, 60 MPa and 170 MPa were essential for studying the irradiation creep behavior of NIFS-Heat alloys in a lithium environment.

## **Discussion**

No apparent differences of the stress dependence of irradiation creep and the value of irradiation creep strain rate for NIFS-Heat alloys between Joyo irradiation and HFIR-17J irradiation could be seen. In addition, corrosive roughness on the specimen surface and scale due to the formation of corrosive products could not be observed directly after neutron irradiation. That



is, surface corrosion due to chemical reaction between bulk vanadium and liquid metal did not occur during irradiation and had no influence on the creep behavior during neutron irradiation.

The most important point for creep behavior in a liquid-metal environment is mass transfer of interstitial impurities between the bulk matrix and the liquid metal. In the case of vanadium in liquid lithium, oxygen atoms move from the vanadium bulk into liquid lithium and nitrogen atoms move from the liquid lithium into the vanadium bulk. On the contrary, in the case of vanadium in liquid sodium, the oxygen and nitrogen atoms move in the opposite directions. A thermal creep measurement was carried out for NIFS-Heat alloys with the same temperature and creep-stress conditions as for Joyo irradiation and chemical analyses were performed after creep irradiation for thermal creep tests at 450°C and 600°C. The results of these chemical analyses showed that the number of oxygen impurities in NIFS-Heat alloy was about doubled, and the nitrogen concentration did not change in a sodium environment. It is considered that mass transfer of interstitial impurities does not significantly influence the creep behavior, but there are no chemical analysis data for interstitial impurities in irradiated specimens. However, from a comparison of the irradiation creep behavior between HFIR-17J and Joyo irradiations, no typical differences in the creep strain rate can be seen for the NIFS-Heat alloys in this study. Therefore, the effect of the environment on creep behavior due to mass transfer of interstitial impurities is assumed to be quite small or negligible.

The creep stress factor,  $n$ , showing the stress dependence of the creep strain rate was 1– 2 for NIFS-Heat alloys in sodium and lithium environments in the temperature range 425 to 600°C. The behavior of  $n$  is very similar to that in a previous study of irradiation creep in austenitic steels and ferrite–martensitic steels [11]. It is suggested that the irradiation creep mechanism for NIFS-Heat alloy is a type of irradiation- induced creep where point defects are absorbed at a dislocation core, and that glide dislocation movement assisted by climbing motion contributes to the creep deformation.

Fig. 4 shows an Arrhenius plot of the creep strain rate. In this Figure, the values of creep strain rate were obtained using the ratio of total creep strain to irradiation period and the differences of the amplitude of the damage rate were within one order for all irradiation conditions. The stress level of the creep condition for all data was fixed at 150 MPa [12,13]. From Fig. 4, the activation energy of irradiation creep was estimated to be 46 kJ/mol·K. The activation energy of vacancy diffusion for NIFS-Heat alloy was 210 kJ/mol·K, obtained from the thermal creep examination in our previous study. The irradiation creep behavior somehow contains a thermal process of creep deformation or an irradiation-induced diffusion process due to excess concentration of point defects. It is still not clear which factor, such as glide dislocation motion, climbing motion, preferential absorption of point defects into the dislocation core, or intergranular vacancy diffusion, is dominant for rate-limiting irradiation creep deformation .

Fig.4

Consequently, a set of essential physical data on the irradiation creep property was obtained in this study in order to predict the creep behavior of NIFS-Heat alloys during neutron irradiation in a liquid-metal environment, focusing on fusion reactor operation.

## **Summary**

Irradiation experiments with sodium-enclosed irradiation capsules in Joyo and lithium-enclosed irradiation capsules in HFIR-17J were carried out using PCTs.

It was found that the creep strain rate exhibited a linear relationship with the effective stress up to 150 MPa at 458°C and 598°C in the Joyo irradiation experiments. The creep strain rate also exhibited a linear relationship with the effective stress up to 150 MPa for HFIR-17J irradiation at 458°C. The activation energy and creep stress factor of irradiation creep were estimated to be 46 kJ/mol·K and 1 to 2, respectively. No significant difference for irradiation creep behavior between liquid-sodium and liquid-lithium environments could be seen.

## **Acknowledgements**

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operation. Also this work has been performed with the cooperation of INITIC, JAEA in Tsuruga.

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### Figure captions

Fig.1. Stress dependence of the effective mid-wall creep strain of a highly purified V-4Cr-4Ti alloy (NIFS-Heat2) irradiated at 458°C and 598°C in Joyo in a sodium environment. Open circles show the data for cold-worked samples and closed circles for annealed samples.

Fig.2. Stress dependence of the effective mid-wall creep strain of a highly purified V-4Cr-4Ti alloy (NIFS-Heat2) irradiated at 425°C in HFIR-17J in a Li environment. Closed circles, #832665-USA V-4Cr-4Ti alloy; open circles, NIFS-Heat alloy; closed triangles, NIFS-Heat alloy irradiated in Joyo.

Fig.3. Stress dependence of the effective mid-wall creep strain of a highly purified V-4Cr-4Ti alloy (NIFS-Heat2) irradiated at 425°C in HFIR-17J in a Li environment. Open circles, NIFS-Heat alloy irradiated in HFIR; closed circles, NIFS-Heat alloy irradiated in Joyo.

Fig.4. Arrhenius plot of creep strain rate for NIFS-Heat alloys. Closed squares show the data for thermal creep at 150 MPa. Other symbols show the data for irradiation creep around 150 MPa.

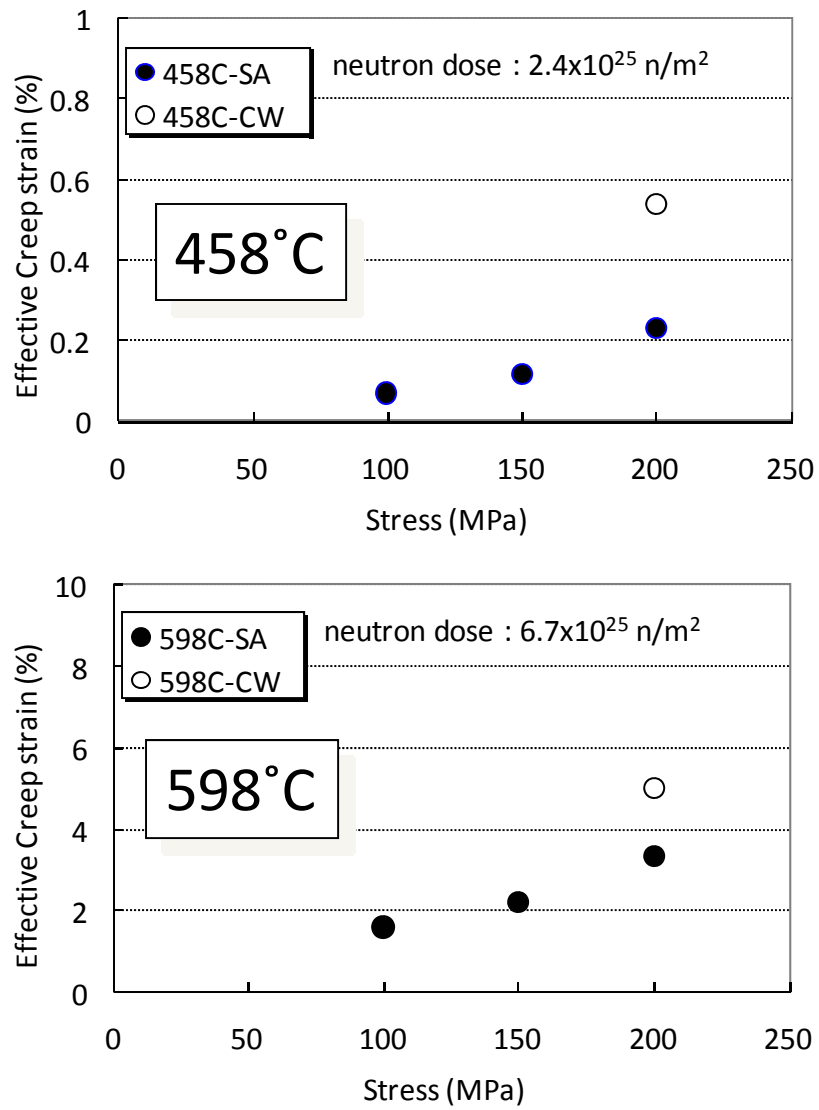


Fig.1 one column

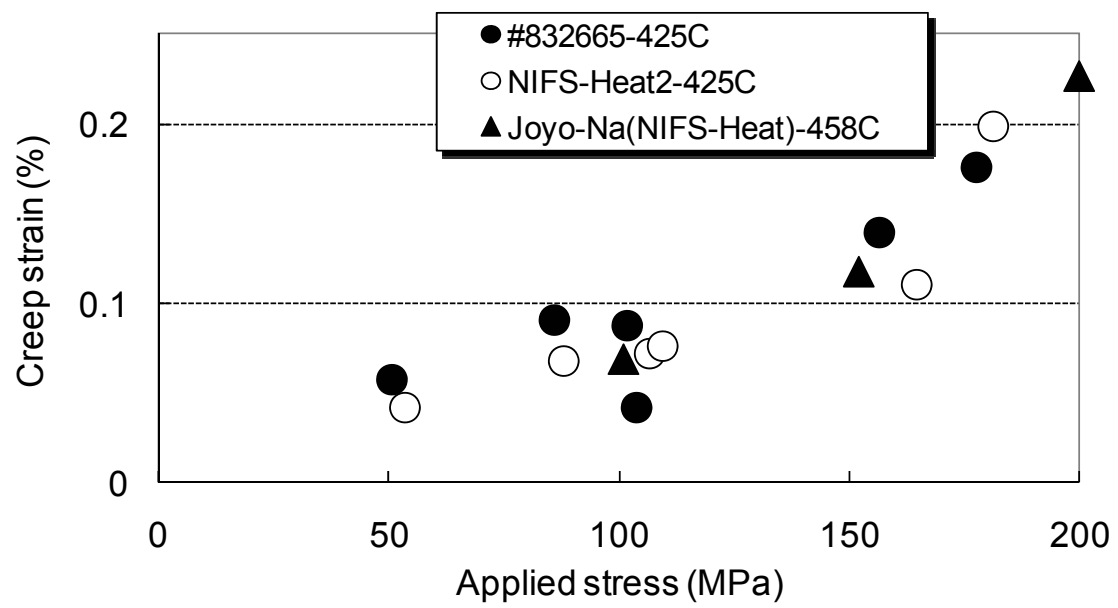


Fig.2. one column



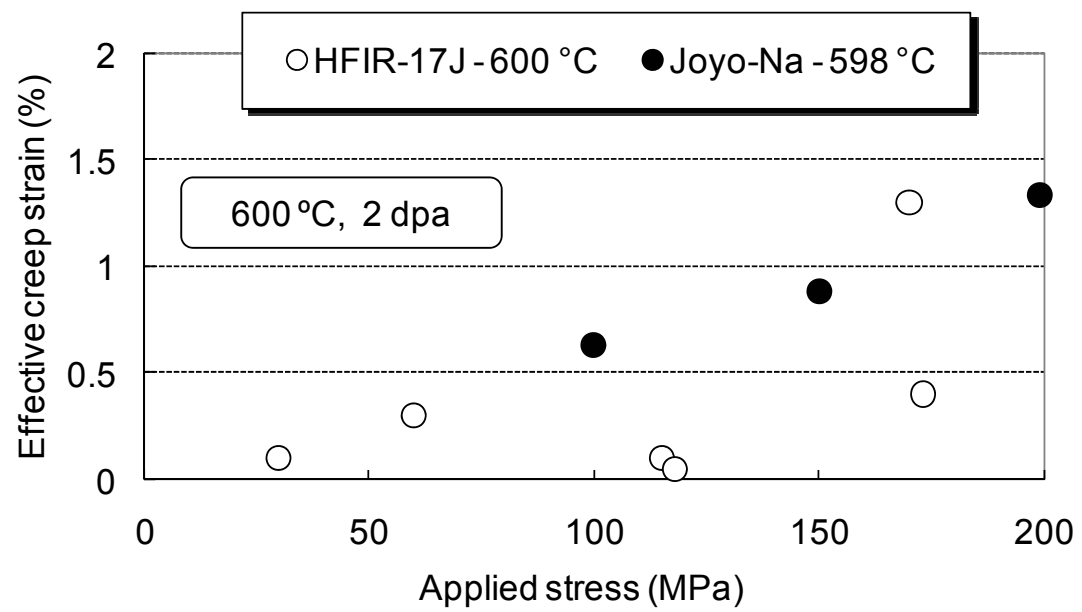


Fig.3. one column

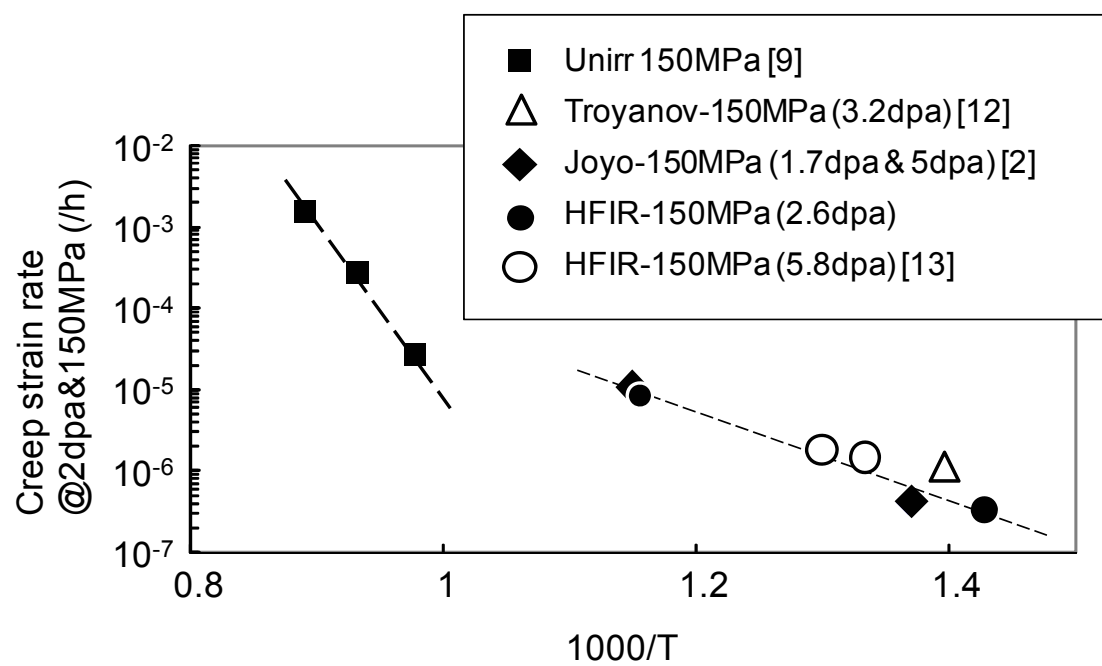


Fig.4. one column