

Mechanical Properties of V-4Cr-4Ti after Exposure in Static Lithium at 650 °C*)

Pengfei ZHENG¹⁾, Takuya NAGASAKA^{1,2)}, Takeo MUROGA^{1,2)}, Masatoshi KONDO^{1,2)}
and Jiming CHEN³⁾

¹⁾The Graduate University for Advanced Studies (Sokendai), Toki 509-5292, Japan

²⁾National Institute for Fusion Science, Toki 509-5292, Japan

³⁾Southwestern Institute of Physics, P.O. Box 432, Chengdu 610041, China

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V-4Cr-4Ti is a leading candidate material for self-cooled liquid Li blanket in a fusion reactor. The interaction between Li and the alloy is of concern, because the mechanical properties of the alloy may be influenced by the mass transfer between liquid Li and the alloy. The present study focuses on the mechanical properties of V-4Cr-4Ti alloy after exposure in static Li at 650 °C for 248 hrs. Results show that the V-4Cr-4Ti alloys are strengthened by the Li exposure. The strengthening for the alloys varies with thermomechanical treatments before exposure. The behavior seems to be corresponding to the formation of precipitates due to trapping of C and N by Ti from the liquid Li.

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1. Introduction

In the existing concepts of blankets in fusion reactors, self-cooled liquid Li blanket is the simplest one. This system has many advantages from safety and economy points of view. Recently, V-4Cr-4Ti alloy has been identified as a leading candidate structural material for Li/V blanket, based on its low activation properties, high strength at elevated temperature and good compatibility with liquid Li [1, 2].

In the operation condition, mass transfer between V-4Cr-4Ti alloy and liquid Li, and long-term aging may change the microstructures of the alloy, thus possibly influence its mechanical properties. Possible mass transfer is mainly the re-distribution of impurities such as C, O, and N in the system including the alloy and liquid Li. Previous study pointed out that these impurities can strengthen the alloy by solid solution hardening and precipitation hardening [3, 4]. On the other hand, concomitant thermal aging can also influence the strength of V-4Cr-4Ti alloy, which usually increases to a peak with the aging time and then decreases [5].

Operation temperature for vanadium alloy is expected to range from 450 °C and 750 °C. T. Nagasaka and M. M. Li have investigated the mechanical properties of V-4Cr-4Ti alloy after Li exposure at 700 °C and 800 °C [6–8]. Results showed that C and N increased, while O decreased in the alloy, and that tensile strength at 700 °C and 800 °C was degraded [6]. Such impurity transfers are possible below

author's e-mail: zheng.pengfei@nifs.ac.jp

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700 °C, leading to the mechanical property change of V-4Cr-4Ti alloys. This study deals with mechanical property changes due to the impurity transfers between V-4Cr-4Ti alloy and liquid Li at 650 °C.

2. Experimental

The materials used in this study are pure V and a V-4Cr-4Ti alloy designated as NIFS-HEAT-2. The chemical compositions of them are shown in Table 1. For the V-4Cr-4Ti alloy, three different thermomechanical treatments are performed, which are called STD, SAA, and SACWA. The detailed treatment conditions are as follows:

STD-standard treatment for recrystallization: 1000 °C for 2 hrs.

SAA-solution annealing (SA) followed by aging (A), which can introduce high density of precipitates: 1100 °C for 1 hr + 600 °C for 20 hrs.

SACWA-solution annealing (SA) followed by cold working (CW) and then aging (A): 1100 °C for 1 hr + 20%

Table 1 Chemical compositions of pure V and NIFS-HEAT-2.

ID				
NIFS-HEAT-2	Bal.	4.02	3.98	(wt%)
C	O	N		
67	148	122	(wppm)	
ID				
Pure V	Bal.			
C	O	N		
90	60	120	(wppm)	

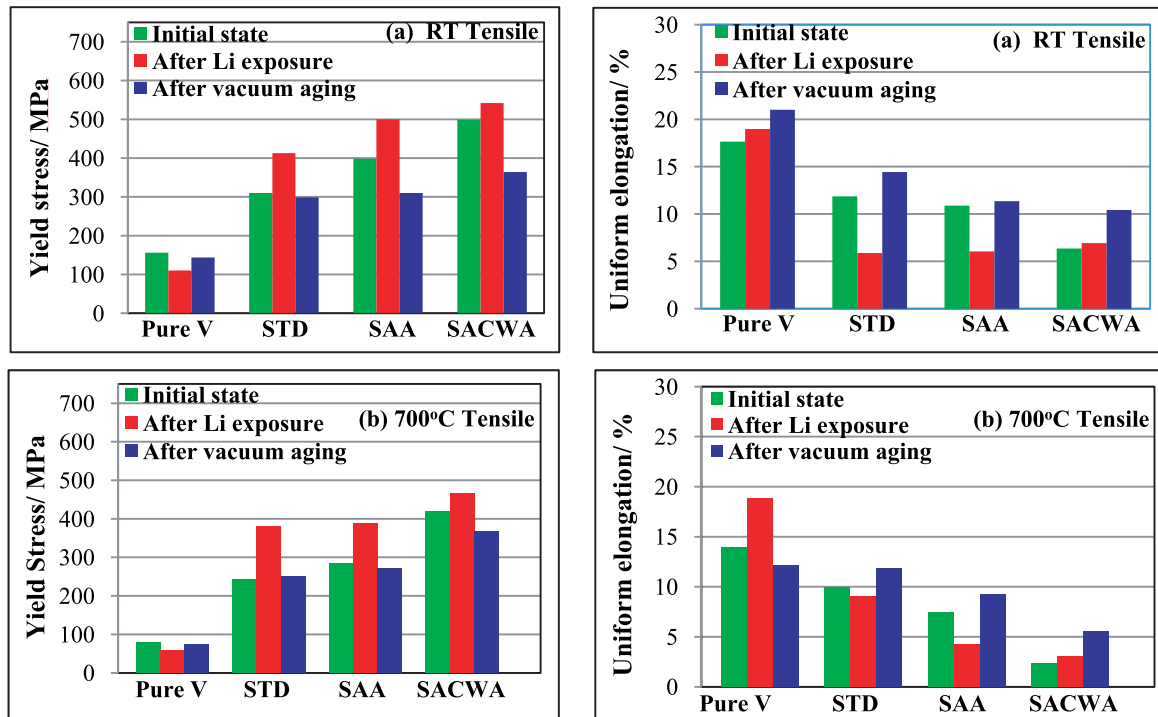


Fig. 1 Tensile properties of pure V and V-4Cr-4Ti in the initial state, after the Li exposure and vacuum aging tested at (a) room temperature and (b) 700 °C.

CW + 600 °C for 20 hrs. This treatment can introduce high density of dislocations and fine precipitates along the dislocations.

The purpose of these treatments is to investigate the effects of microstructure changes due to Li exposure on mechanical properties.

All the specimens for pure V, STD, SAA and SACWA are 0.25 mm in thickness. The gauge dimension of the tensile and uniaxial creep test specimens is 5 mm × 1.2 mm × 0.25 mm, which is designated as SSJ specimen. These specimens are exposed to liquid Li at 650 °C for 248 hrs. The Li contains 65 wppm N, but the concentrations of other impurities are not measured. The material of the crucible, cover and wire for specimen holder is molybdenum. After the Li exposure, all specimens are cleaned in ethanol.

An aging is carried out in vacuum for control specimens also at 650 °C for 248 hrs.

Cross section hardness tests, tensile tests at room temperature and 700 °C have been carried out. Uniaxial creep tests at 700 °C for SACWA after Li exposure have also been carried out, with a load of 100 MPa and 180 MPa. All the tests at 700 °C have been conducted in a vacuum better than 110^{-6} torr, with a Zr foil as an impurity getter. Data of similar creep tests for the initial state SACWA are introduced from previous results published elsewhere [9]. The microstructures are observed by a transmission electron microscope (TEM) to clarify their correlation to the mechanical properties.

3. Results

3.1 Mechanical properties

Figure 1 shows the tensile properties of pure V and V-4Cr-4Ti in the initial state, after the Li exposure and after the vacuum aging obtained at (a) room temperature and (b) 700 °C. According to the tensile tests at room temperature, pure V is softened after the Li exposure, while the V-4Cr-4Ti alloys are strengthened. The strengthening of STD and SAA, on the other hand, results in a decrease in the uniform elongation. However, the uniform elongation of SACWA after Li exposure seems not to decrease, corresponding to the smaller strengthening than STD and SAA get. The vacuum aging softens SAA and SACWA, and increases their uniform elongations. The trends of tensile properties of V-4Cr-4Ti alloy at 700 °C are similar to those at room temperature.

Figure 2 shows the distribution of cross section hardness of all the specimens in the initial state, after the Li exposure and after vacuum aging. Pure V doesn't have remarkable hardness change after Li exposure. STD is hardened remarkably after the Li exposure, especially near surfaces of both the sides. In contrast, SAA and SACWA get significant hardening near the surfaces of both the sides but softening at the center after Li exposure. Interestingly, the steep hardness change occurs in the area less than about 75 μm from the surfaces of all the V-4Cr-4Ti alloys after Li exposure in the present test. In the case of vacuum aging, only SAA and SACWA get remarkable softening.

The dependence of the minimum creep rate on applied

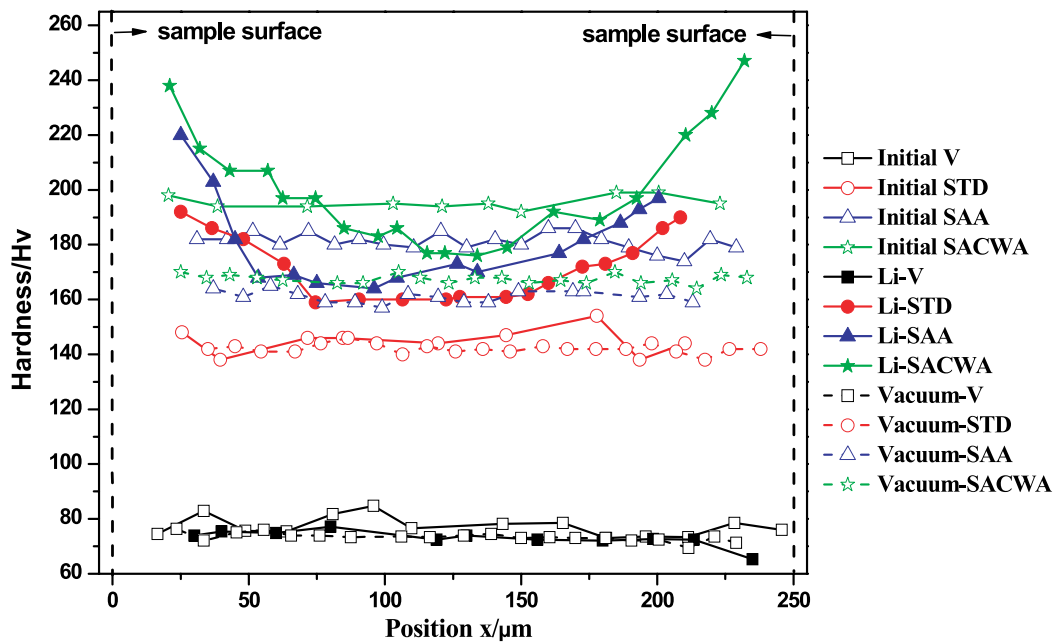


Fig. 2 Distribution of cross section hardness for pure V and V-4Cr-4Ti in initial state and after Li exposure. (Initial: initial state; Li-: after Li exposure; Vacuum-: after vacuum aging)

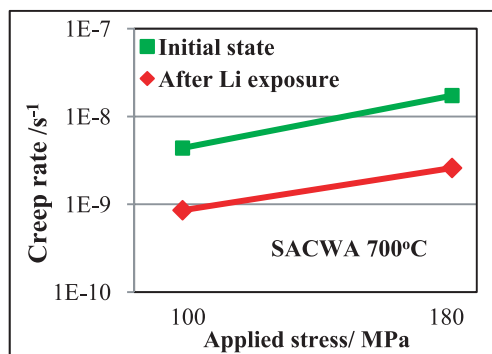


Fig. 3 The dependence of the minimum creep rate at 700 °C on applied stress for SACWA in the initial state and after the Li exposure.

stress for SACWA in the initial state and after the Li exposure is shown in Fig. 3.

All the creep data in Fig. 3 are fitted to the following equation:

$$\dot{\epsilon} = A \cdot \sigma^n \cdot \exp(-Q_a/RT), \quad (1)$$

where $\dot{\epsilon}$ is the minimum creep rate, A is a constant, σ is the applied stress, n is the stress exponent, R is the gas constant, T is the absolute temperature, and Q_a is the apparent activation energy [10]. According to the fitting, the stress exponent n for SACWA at 700 °C is 2.7 in the initial state and 1.9 after the Li exposure.

3.2 Microstructural observation

Figure 4 shows the microstructures in the initial state, after the Li exposure and after vacuum aging. It must

be noted that the microstructural observation was conducted at the center of the specimen, around the position of 125 μm in Fig. 2. For pure V, there seems to be no change in the microstructure after the Li exposure or the vacuum aging. High density of precipitates are formed in STD after the Li exposure, but not after the vacuum aging. Precipitates are formed in the initial state SAA [9], though too small to be observed in the present study. The existing precipitates in SAA have coarsened after both the Li exposure and the vacuum aging. In SACWA, precipitates are not observed under the present conditions, but they are expected to be formed along dislocations during the Li exposure and vacuum aging, according to the previous study [9]. Dislocation density of SACWA decreases during both the Li exposure and the vacuum aging. Li exposure, however, seems to keep more dislocations than that vacuum aging does.

4. Discussion

From the TEM images, the strengthening of STD after Li exposure seems to be due to the formation of precipitates. Previous study showed that N and C in Li solution can be trapped by Ti in the alloy matrix, hardening the alloy, while O diffuses from the alloy matrix into the molten Li, softening the alloy [6–8]. According to Ref. [11], the diffusion coefficient at 650 °C of O and C are $2.46 \times 10^{-13} \text{ m}^2/\text{s}$ and $2.29 \times 10^{-13} \text{ m}^2/\text{s}$, respectively. Based on the following equation [12],

$$d \approx \sqrt{D \times t}, \quad (2)$$

where d is the diffusion range, D is diffusion coefficient and t is diffusion time in seconds, diffusion ranges are

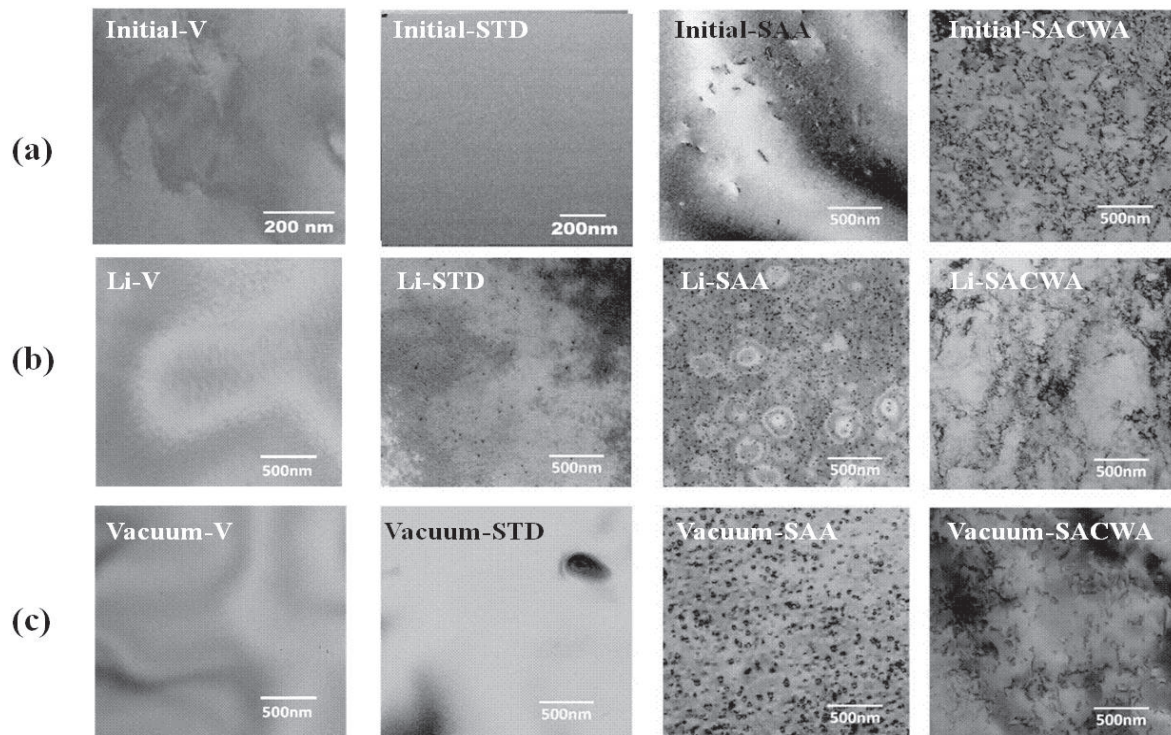


Fig. 4 TEM images for pure V and V-4Cr-4Ti (a) in initial state, (b) after Li exposure, (c) after aging in vacuum. Note: TEM images for pure V and STD in initial state are from the previous studies [13, 14].

475 μm for O and 452 μm for C, which penetrate the thickness of the specimens. While in the same condition, the diffusion coefficient of N in V is $6.67 \times 10^{-15} \text{ m}^2/\text{s}$, giving a diffusion range of 78 μm . This diffusion range is much smaller than those for O and C, and is not much different from the hardening range for V-4Cr-4Ti alloys in Fig. 2. Consequently, N diffusion is more suitable than C and O to explain the surface hardening.

In this case, the additional precipitates in the alloy observed by TEM are considered to be C-rich Ti (C, O, N) particles. The reason is that TEM observations in this study are performed on the center of the specimens, which is beyond the diffusion range of N in the present Li exposure, and O is gone into Li. These C-rich Ti (C, O, N) precipitates contribute the hardening at all the positions in STD. While, N mainly contributes hardening within the diffusion range, 75 μm from the surfaces. In the hardened region of V-4Cr-4Ti alloys, the hardening due to C and N was larger than the softening due to decrease in O concentration.

Li-exposed SAA and SACWA only gain hardening within 75 μm from the surfaces. For SACWA, the dislocations can supply nucleation sites for the precipitates and suppress the coarsening of precipitates [9], enhancing the hardening. Then the stronger interaction of dislocations and precipitates increases the flowing stress in SACWA, reducing the creep rates as shown in Fig. 3. However, SAA and SACWA are softened in the center part after Li exposure. The softening of SAA is considered to be caused by the decrease in the number density of precipitates due to

their growth. This might be more effective than the hardening due to additionally formed C-rich Ti (C, O, N) precipitates. And the softening of SAWCA in the center part is probably due to the decrease in dislocation density.

For pure V, there seems to be no change in the microstructures after Li exposure. However, O has diffused out from the V matrix, thus the matrix was softened according to the results of tensile tests in Fig. 1.

5. Conclusions

Mechanical properties of pure V and V-4Cr-4Ti alloys after Li exposure at 650 $^{\circ}\text{C}$ for 248 hrs are investigated at room temperature and 700 $^{\circ}\text{C}$ in the present study. The conclusions are as follows:

- (1) Li exposure at 650 $^{\circ}\text{C}$ for 248 hrs leads to strengthening for V-4Cr-4Ti alloys with various thermomechanical treatments, while not for pure V.
- (2) The Li exposure strengthening for V-4Cr-4Ti alloys is likely attributed to the increase in number density of Ti (C, O, N) precipitates, due to mass transfer of C and N from liquid Li. Softening by transfer of O from pure V and V-4Cr-4Ti alloys to liquid Li is expected. In the present condition, the precipitation hardening contributes more than the decrease in solid solution hardening due to loss of O.

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