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COMPATIBILITY OF LOW ACTIVATION FERRITIC STEELS WITH LIQUID LITHIUM

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Fe-Cr-W based low activation ferritic steels are widely regarded as promising blanket structural materials for fusion reactors, while liquid lithium breeder/coolant concept provides an attractive option for high efficiency and simplicity of blanket system. However, past compatibility tests of ferritic steels with liquid lithium were almost limited to conventional Fe-Cr-Mo steels. In this study, the corrosion behavior of the candidate reduced activation ferritic steel, JLF-1(Fe-9Cr-2W-0.1C) in lithium was investigated. Static immersion tests were carried out using coupon specimens (16×4×0.25 mm) at 873K and 973K for 100hr. At 973K, the phase transformation from martensite to ferrite resulted in decrease in hardness from 250 to 140Hv. This seemed to be caused by depletion of C. Examinations of binary Fe-Cr and pure iron were also carried out for comparison with JLF-1.

I. INTRODUCTION

Development of breeding and structural materials for blankets is the key issue of fusion reactors. Liquid lithium is regarded as an attractive breeding material because of its high tritium-breeding rate, high thermal conductivity and so on. Vanadium alloys have mainly been proposed as structural materials for lithium blanket.¹ However, use of Fe-Cr-W based reduced activation ferritic-martensitic steels (RAFM) was proposed recently as an alternative concept for lithium blanket.² This is a low risk near-term option because of the industrial maturity of RAFM relative to vanadium alloys, although maximum operation temperature is limited because of the strength of RAFM at high temperature.

Several studies were reported on compatibility of ferritic steels with liquid lithium in thermal-convection loops.^{3,4} But most of the past researches were on conventional Fe-Cr-Mo based steels, instead of candidate Fe-Cr-W based RAFM. In this study, the compatibility of RAFM with lithium was investigated including comparative examinations using model binary Fe-Cr alloy and pure iron.

II. EXPERIMENT

JLF-1(Fe-9Cr-2W-0.1C), a pure iron and a Fe-9Cr binary alloy were used for the experiment. JLF-1 specimens were heat-treated at 1323 K/3.6 ks/air cooled (normalizing) and 1052 K/3.6 ks/air cooled (tempering). The coupon specimens with the size of 26×5×0.25 mm were prepared from sheet. Pure iron and Fe-9Cr specimen was cut to 0.25×20×4 mm coupon specimens after rolling to 0.25mm sheet from rod and plate, followed by annealing at 973K for 1 hour. The surfaces of specimens were mechanically polished before experiment.

Static exposure experiments to liquid lithium were carried out in molybdenum crucible, which were placed in stainless steel autoclave. In order to suppress the effect of impurities which was reported previously,^{5,6,7} high purity lithium (99.9%wt with the nominal N impurity level of 40ppm) was used and the preparation was carried out in a glove box filled with argon in which content of nitrogen is less than 1ppm. The content of N in lithium during the experiment was estimated to be <100 ppm. For each experiment, 10 specimens were exposed in 100ml lithium and the volume ratio of Li to specimens was about 300:1. The lithium was refreshed in each experiment.

Pure iron, Fe-9Cr and JLF-1 were exposed to lithium at 973K for 100h (test A) to study the influence of alloy elements. Another group of JLF-1 specimens were exposed in lithium for 100h at 873K and 973K (test B) to study the influence of temperature. The pot and the

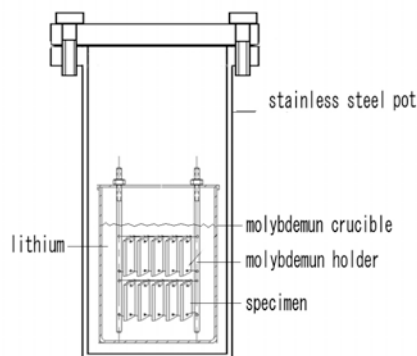


Fig.1 The pot, molybdenum crucible and the interior

interior structure are shown in Fig. 1. The specimens were cleaned by ammonia after the exposure experiment followed by surface analysis. After that, the specimens were cleaned by water and analyzed again. In addition, JLF-1 as received was annealed in vacuum for 100 h at 873 and 973K for examining the thermal history effects.

The examinations of the specimens were carried out for weight loss measurements by electro-balance, surface morphology and composition by Scanning Electron microscope (SEM) equipped with Energy Dispersive X-ray Spectrometer (EDS), microstructure by Transmission Electron Microscope (TEM), hardness by Vickers Hardness Testing Machine with the weight of 10 g, as well as chemical analysis.

III. RESULTS AND DISCUSSION

III.A. Weight Loss and Cr and W Depletion

The weight changes during 100h test at 873K and 973K are shown in Table I. After exposure for 100h, pure iron and Fe-9Cr gained weight, while JLF-1 lost weight. This indicates that faster corrosion took place at JLF-1 during lithium exposure. Small corrosion occurred on JLF-1 specimen at 873K. The corrosion rate after water cleaning is 0.0046mm/a. (Note that constant dissolution rate is assumed for the estimate). In the present experiment, water cleaned specimen had higher weight loss than ammonia cleaned ones, due to removal of the surface corrosion products by water.

The surface composition changederived by EDS measurement from the normal direction to surface is listed in Table I. As shown in Table I, depletion of chromium and tungsten was observed on the surface of all specimens exposed in liquid lithium except pure iron. Increase in Cr on the surface of the pure iron seems to be caused by the deposition from lithium during the cooling period. The depth of Cr depletion was about 5μm according to EDS line scan as shown in Fig. 2. The selective dissolution of both Cr and W is thought to

enhance the weight loss of JLF-1 relative to that of Fe-9Cr and pure iron.

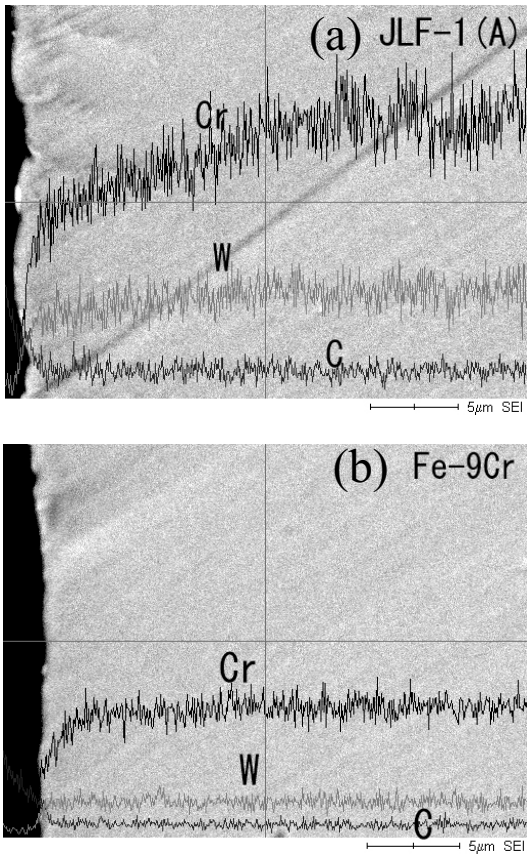


Fig.2 Cross section EDS line scan on JLF-1 (Test A) and Fe-9Cr after exposure to Li at 973K for 100 h

III.B. Phase Transformation and Hardness Reduction

The surface morphology of JLF-1 (ammonia clean) exposed at 973K is shown in Fig. 3. Clear grain

TABLE I. Weight loss and the surface composition of specimens exposed to Li for 100h

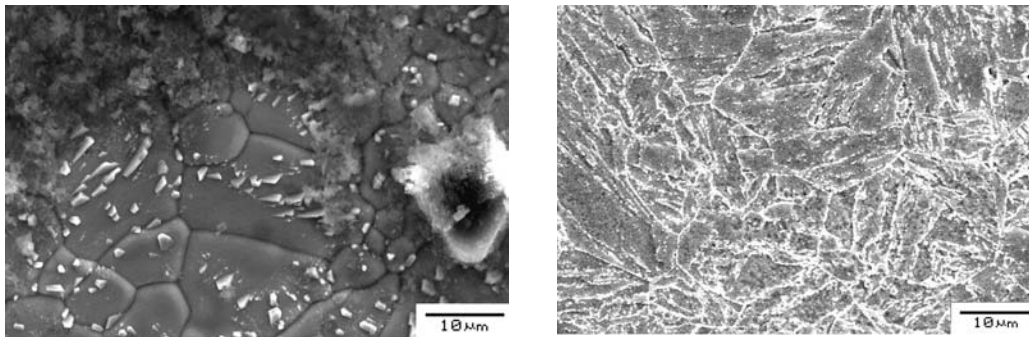
Materials/exposure temp.	Ammonia cleaning	Water cleaning	Surface composition (EDS result)			
	Weight loss (g/m ² h)*	Weight loss (g/m ² h)*	Fe	Cr	W	C
JLF-1/ bef. experiment			89.0	8.9	2.0	0.1
Fe/ 973K	-0.021	Not available	96.4	1.7**	—	—
Fe-9Cr/ 973K	-0.0075	Not available	92.1	6.3	—	—
JLF-1/ 973K(A)	0.0075	Not available	93.5	5.4	1.1	—
JLF-1/ 973K(B)	0.0327	0.1667	90.7	7.5	0.8	—
JLF-1/ 873K(B)	-0.065	0.0041	92.1	6.8	0.7	—

* Constant weight change rate during the immersion was assumed
 ** This seems to be the result of Cr deposition from Li during the cooling period
 (A) JLF-1, pure iron and 9Cr were exposed in the same lithium pot
 (B) Only JLF-1 was exposed in lithium pot

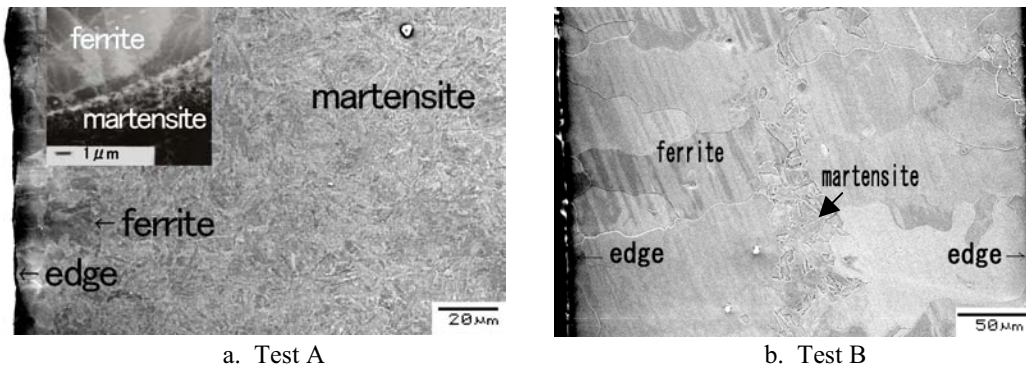
structure was observed on the surface of the specimen exposed in Li for 100 h at 973K. Spongy corrosion product layer covered the surface of the specimen after ammonia cleaning. These corrosion products disappeared during water cleaning. It is clear that phase transformation occurred at the surface of JLF-1 with martensite structure changing into a ferritic phase. This phenomenon was not found on the specimen that was annealed in vacuum at the same temperature for 100h, as shown in Fig. 3. No phase transformation was observed on JLF-1 after exposure at 873K.

The phase transformation has also been observed inside the specimen. Figure 4 (a) and (b) are the cross section SEM pictures of JLF-1 exposed at 973K in test A and test B respectively. The phase change from martensite

to ferrite was also proved by TEM as shown in Fig. 4. In test B the phase transformation depth reached 100 μm at 973K. The martensite structure only remained in the center of specimens as seen in Fig. 4. In contrast, the depth of phase change was only $\sim 20\text{ }\mu\text{m}$ in Test A. Figure 5 shows hardness change with depth before and after exposure to lithium. The hardness reduction occurred on JLF-1 and Fe-9Cr at 973K. For Fe-9Cr, the softening seems to be caused by Cr depletion because the softening depth is close to Cr depletion range of $5\mu\text{m}$. In JLF-1, phase transformation played a dominant role for the hardness. Softening depth is consistent with the phase transformation depth. By the carbon dissolution and phase change, the hardness decreased from 250 to 140Hv. In the case of test B (973K), the hardness of JLF-1 at the area



a. JLF-1 exposed in Li at 973K for 100h b. JLF-1 annealed in vacuum at 973K for 100h
Fig.3. SEM micrographs of JLF-1



a. Test A b. Test B
Fig.4. Cross section SEM and TEM images of JLF-1 exposed Li at 973K for 100h for the two tests.

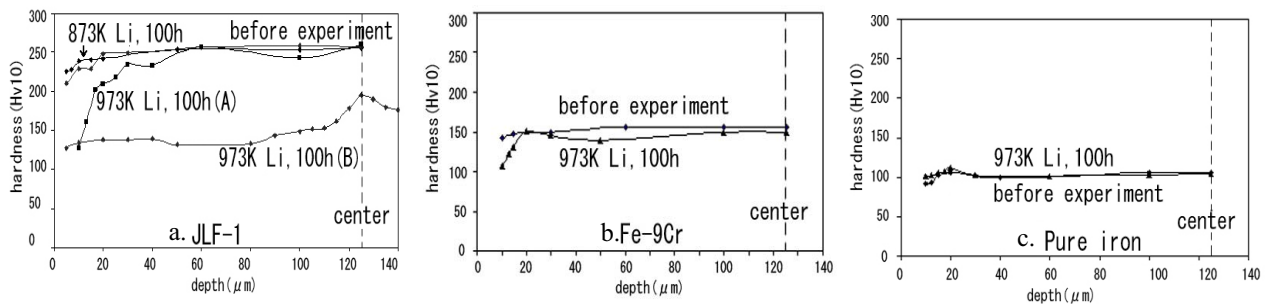


Fig.5. Depth dependence of hardness

where the phase change occurred was close to that of Fe-9Cr. This implies that W has small impact on hardness in single ferritic phase. The fact that the hardness at the center of the specimen in test B is between that of the martensitic and ferritic phase implies that the phase at the center is mixture of the two phases. For pure iron, hardness was kept constant because neither phase transformation nor selective dissolution of alloying element occurred. Almost no hardness change was detected on JLF-1 exposed at 873K.

The chemical analysis of JLF-1 was carried out and the result is listed in Table II. Two third of carbon in JLF-1 (test B) dissolved into liquid Li at 973K for 100h. Martensite can be converted to ferrite when carbon is removed. Thus, the loss of carbon is expected to be the major reason for the phase change in this experiment.

According to thermodynamics graph in Fig. 6 (Ref. 8), the order of stability of these compounds is at 973K is $V_2C > VN > Li_3N > Li_2C_2 > Fe_3C$. It indicates the tendency that V should get C and N, while Fe should lose C during lithium exposure.

TABLE II. Chemical analysis result (wt %, JLF-1 specimens in test B)

Materials	C	Cr	W
Before exposure	0.09	8.92	2.00
Li 973K 100hr	0.03	8.88	1.95
Li 873K 100hr	0.10	8.89	1.99

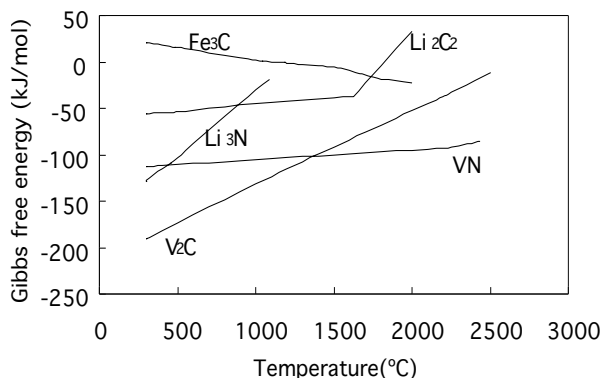


Fig.6 Thermodynamic data of several compounds in Li (Ref.8)

The extreme difference in loss of carbon and the resulting phase change between 873K and 973K seems to be caused by the difference in the mobility of carbon. In the present study JLF-1 was tempered before the experiment. This means carbon was trapped at stable positions in the tempered martensitic phase. According to the tests of tempered F82H (Fe-8Cr-2W), decrease in yield stress was observed by aging at 923K but not at

873K (Ref. 9). This suggests that the de-trapping of carbon in the tempered martensitic phase should begin above 873K. Thus decarburization by lithium is thought to become prominent above 873K in the present experiment.

IV. SUMMARY

At 873K, relatively small corrosion occurred on JLF-1 exposed to lithium. Neither phase change, nor carbon depletion was found. On the other hand, JLF-1 was largely influenced by the exposure at 973K, including depletion of carbon and chromium and resulting phase transformation from martensite to ferrite. Hardness reduction was caused by the phase transformation and element dissolution

The corrosion of JLF-1 is more significant than that of Fe-9Cr and pure iron at 973K. The selective dissolution of Cr, W and C into lithium seems to enhance the corrosion.

Based on the data obtained, corrosion in Li seems not to be a serious issue for low activation ferritic steel below 873K. However, corrosion could be a problem by exposure to Li for longer time or in flowing conditions. In these cases, possible influence of Li may be the phase change and Cr and W depletion.

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