JP0550298

COMPATIBILITY OF REDUCED ACTIVATION FERRITIC/MARTENSITIC STEEL

SPECIMENS WITH LIQUID Na AND NaK IN IRRADIATION RIG OF IFMIF

June 2005

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Compatibility of Reduced Activation Ferritic/Martensitic Steel Specimens
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In the high flux region of the International Fusion Materials Irradiation Facility (IFMIF), the neutron irradiation damage for iron-based alloys will exceed 20 dpa/ year. An accurate specimen temperature measurement under a large amount of nuclear heating is a key issue but the change of heat transfer of gap between irradiation specimens and specimen holder during irradiation test is inevitable, if gap is filled with an inert gas and temperature is monitored by a thermocouple buried in the specimen holder. A solution to make heat transfer predictable is to fill the gap with a liquid metal (sodium or sodium-potassium alloy). An issue of compatibility between Reduced Activation Ferritic/Martensitic steels and the liquid metalsis addressed in this paper, and some recommendations for designing irradiation rig are presented, such as a purification control before filling liquid metals, or a careful selection of material of rig to avoid carbon mass transfer.

※ Cooperative Staff

IFMIF照射リグにおける
液体Na、NaKと低放射化フェライト鋼照射試料との両立性

日本原子力研究所那珂研究所核融合工学部
湯谷　順明※・中村　博雄・杉本　昌義

　材料の照射特性は温度依存性が強いため、照射中の試料温度測定は重要である。しかしながら、中性子照射損傷が20 dpa/年以上となるIFMIFの高中性子束領域において、鉄ベースの合金を照射する場合、大量の核発熱が生じるため、試料温度を正確に計測することは、不活性ガスを照射試料と試料ホルダーとの間のギャップに充てんして試料温度を試料ホルダーに埋め込んだ熱電対によって測定する従来の方式では、照射中にギャップの熱伝達率が変化するため極めて困難である。その対策としてギャップの熱伝達率がより安定と期待される液体金属（ナトリウム又はナトリウム－カリウム合金）をギャップに充てんする方法が提案（FZKが提案）されているが、液体金属と低放射化フェライト鋼との両立性に懸念があった。本報告は、このような液体金属と低放射化フェライト鋼との両立性について検討し、照射リグ設計への提案として、充てん前の液体金属の純度管理や炭素原子移行を予防する材質選択上の注意を述べる。

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1. Introduction

In the conceptual design of the International Fusion Materials Irradiation Facility (IFMIF) [1], a helium-cooled High Flux Test Module (HFTM) is used to irradiate the specimens for the first wall and blanket structural materials up to a damage level of 20-50 dpa/fpy (full power year) in the case of reduced activation ferritic-martensitic (RAFM) steels. The HFTM includes a number of irradiation rigs containing the encapsulated specimens controlled at the desired irradiation temperature between 250 and 1100 °C with the specified temperature uniformity. A method to fill the capsule that houses specimens with an inert gas such as helium and to monitor the specimen temperature by using thermocouple buried in the capsule wall is usually used in the irradiation with the fission reactors. This method may have a large uncertainty of specimen temperature prediction due to the change of heat transfer property of gas gap between capsule wall and specimens during irradiation test period. To improve the predictability of the heat transfer, a liquid metal (sodium or, if possible, sodium-potassium alloy) has been proposed as the gap filler to increase thermal contact, as shown in Fig. 1. Both Na (Boiling Point 883°C) and NaK (B.P. 784°C for Na-78 wt% K alloy) can be used with RAFM steels, because the requirement for maximum irradiation temperature is 650℃.
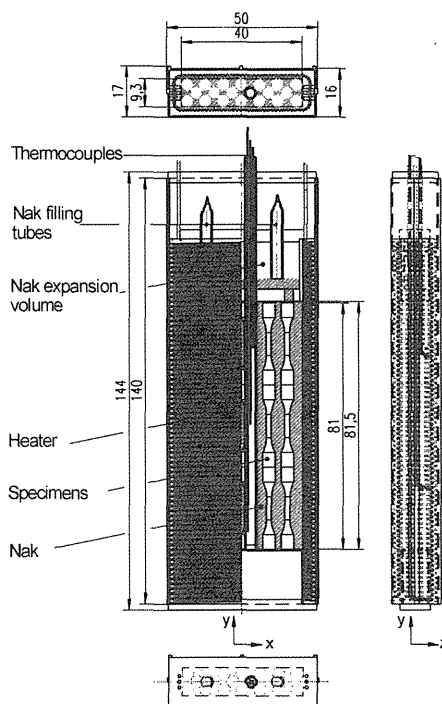

Fig. 1   Configuration of the irradiation rig (dimensions in mm) [Fig. 3.1-5 in Ref. 1].

To realize this concept, the compatibility issues are main concern and key performance variables were identified as 1) corrosion due to mass transfer of metallic elements, 2) mass transfer of

non-metallic elements (O, C, and N), and 3) influence of corrosion on mechanical properties [2]. In Na, corrosion due to mass transfer of metallic elements is accelerated when the concentration of oxygen in sodium is high. Also, decarburization or carburization occurs in the specimens. In the RAFM steel the influences of decarburization and carburization on mechanical properties are large. The performance of NaK compatibility seems to be similar to that of sodium, because the NaK compatibility is fairly similar to that for sodium [2]. In this work, the data of sodium except for oxygen solubility were used to evaluate NaK compatibility.

2. Compatibility Issues
2.1. Mass transfer of metallic elements

The irradiation in the temperature range from 250 to 650 °C is required for RAFM steel specimens. During the irradiation under this condition, the mass transfer of metallic element between the specimens and the Na (or NaK) is occurred. Its level is determined by the solubility of the constituents of specimens in Na (or NaK) and the temperature dependence of the solubility.

The solubility data excerpted from the literatures for several metals in sodium are shown in Fig. 2 and table 1 ([2, 3, 4]). Figure shows that the solubility values are small for the primary constituents (Fe, Cr) of RAFM steel. Vanadium has a small solubility and tantalum has a little larger solubility. It is considered that the corrosion depth of steel is dominated by the dissolution of primary constituents, because corrosion morphology of the steel in sodium is known to be a uniform corrosion. The corrosion depth of RAFM steel, F82H (8Cr-2W steel), specimen in NaK by the mass transfer of metallic elements is evaluated conservatively under the following assumptions.

- 1) NaK and F82H specimen occupy the equal portions in the irradiation volume of 500 cc. Therefore the weights of NaK and F82H are calculated as 388 g and 3935 g (Fe: 3541 g, Cr: 315 g, W: 79 g), respectively. (The densities of NaK and F82H steel are 0.775 and 7.87 g /cm^3 respectively.)
- 2) Temperature of NaK during irradiation keeps at 700 °C. From the solubility data, the mass transfers of Fe and Cr to NaK are estimated to be 1.20×10^{-3} and 4.57×10^{-4} g, respectively.
- 3) The thickness of specimen is 1 mm, so the surface area is 2,500 cm^2.

Under above assumptions the calculated weight loss ratio of Fe to Cr is about 2.6, and it dose not exceed the composition ratio of Fe to Cr (Fe/Cr = 11). Then, the corrosion depth is dominantly determined by the weight loss of Cr, namely F82H specimen corrodes by 12.5 (=100/8) times of weight loss of Cr. The corrosion depth due to mass transfer of metallic elements is evaluated to be:

$$4.57 \times 10^{-4} \times 12.5 / (2500 \times 7.87) = 2.90 \times 10^{-7} \text{ cm}$$

This value is too small to be of concern.

Table 1 Solubility data for metals in sodium

Metal	Solubility, log S (wppm) = A + B/T		Ref.
	A	B	
Fe	4.72	− 4116	3
Cr	9.33	− 9010	2
V*	–	–	4
Ta**	–	–	4

* Solubility value is 2 wppm at 600 °C.

** Solubility value is 3 wppm at 525 °C.
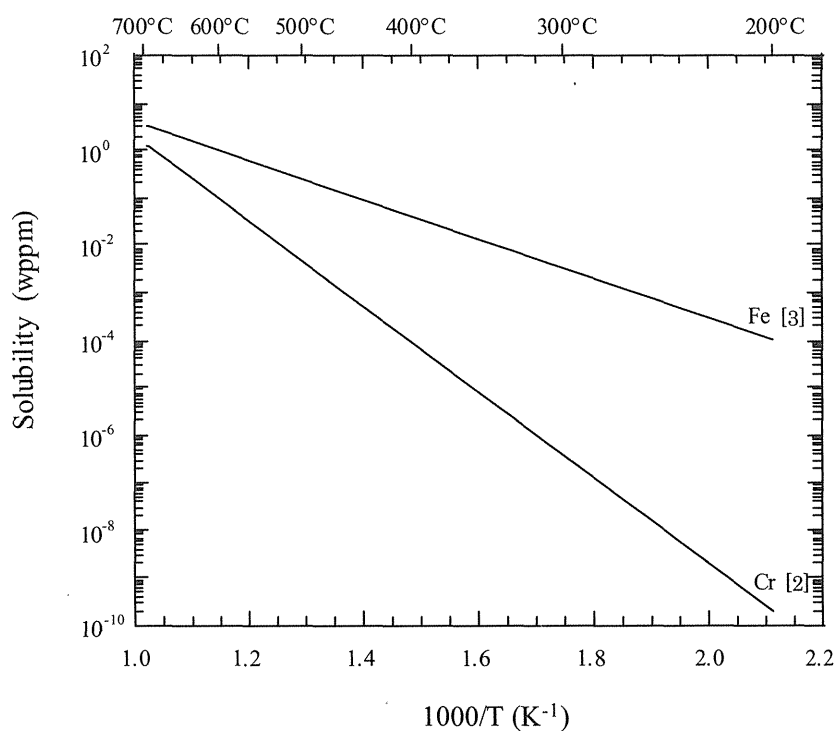
Fig. 2 Solubility of metal in sodium.

2. 2. Mass transfer of non-metallic elements

Non-metallic elements such as oxygen, carbon and nitrogen in liquid metal are known to migrate across the boundaries of structural-materials and liquid-metal. The RAFM steel constituents may react with such non-metallic elements in Na or NaK to form some stable compounds such as $NaCrO_2$.

Solubility data for non-metallic elements in Na and NaK are shown in Fig. 3 and table 2. Solubility data of oxygen in Na [5] seems to be most reliable. The oxygen solubility data for NaK have large discrepancy between the published data ([6,7]), especially at the low temperature region.

Recently, H. U. Borgstedt et al. [8] broadly surveyed the published data for the solubility of non-metals in liquid alkali metals. However, the data for NaK were poor except the solubility of oxygen. So the solubility values for nitrogen and carbon in Na are applied to the case of NaK. This procedure might be sensible because the primary impurity in Na or NaK is oxygen, while nitrogen and carbon concentrations are usually low.
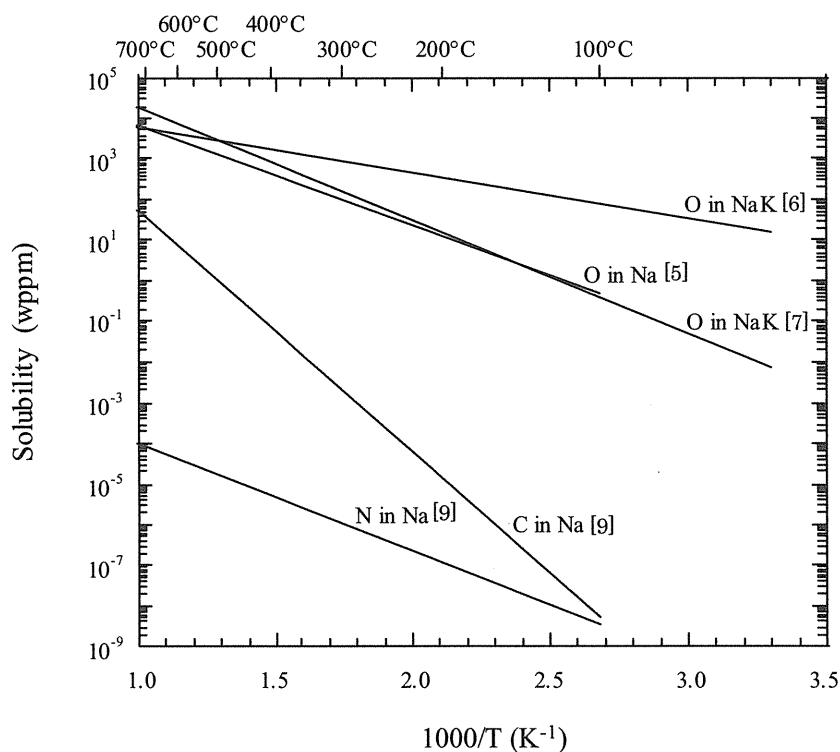
Fig. 3   Solubility of non-metallic elements in Na and NaK.

Table 2   Solubility data for non-metallic elements in Na and NaK

Non-metallic Element	Liquid Metal	Solubility, log S (wppm) = A + B/T		Ref.
		A	B	
O	Na	6.2571	-2444.5	5
O	NaK	4.848	-1101	6
O	NaK	7.09	-2795	7
N	Na	-1.336	-2660	9
C	Na	7.700	-5967	9

2. 2. 1. Oxygen

It is known that the metals form ternary oxides such as $NaCrO_2$ and Na_4FeO_3 as well as binary

oxides when immersed in liquid sodium containing oxygen at high temperatures. Standard free energy of formation and oxygen potential of formation for oxides of liquid metal and primary constituent of RAFM steel are shown in table 3 and Fig. 4, respectively. The oxygen potential of formation of ternary oxide for each metal is lower than that of binary oxide, namely the ternary oxide is formed at the lower oxygen concentration in Na or NaK than the binary oxide.

Table 3   Standard free energy of formation for oxide

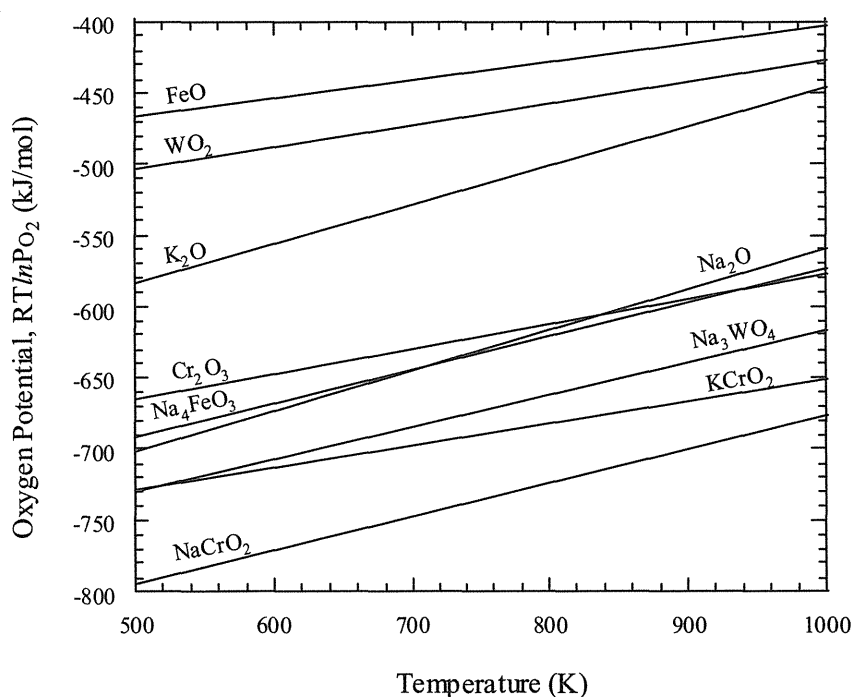| Oxide	ΔG_f° (J/mol) = A + BT		Ref.
	A	B	
Na_2O	-421530.1	141.41	10
K_2O	-360391.4	137.4	10
FeO	-264900	63.4	11
Cr_2O_3	-1125150	257.5	11
WO_2	-579500	153.0	11
Na_4FeO_3	-1212202	351.1	12
$NaCrO_2$	-913762.8	237.24	13
Na_3WO_4	-1680970	446.5	14
$KCrO_2$	-803800	152.53	15

Fig. 4   Oxygen potential of formation for oxide

Oxygen potential of oxygen dissolved in sodium is given by the following relation [16].

$$\Delta G_{O_2}[O]_{Na} \ (J/mol) = 2\Delta G_f^{\circ} < Na_2O > +38.29T\left(\log C_O - \log S_O\right) \tag{1}$$

Where $\Delta G_{O_2}[O]_{Na}$ is oxygen potential dissolved in Na, $\Delta G_f^{\circ} < Na_2O >$ is standard free energy of formation of Na2O shown in table 3, T is temperature (K), C_O is oxygen concentration (wppm) in Na, and S_O is oxygen solubility in Na shown in table 2. From equation (1) and data shown in tables 2 and 3, following relation is obtained.

$$\Delta G_{O_2}[O]_{Na} = -749500 + 43.24T + 38.29T \log C_O \tag{2}$$

Similarly, following relation for oxygen potential of oxygen dissolved in potassium is obtained.

$$\Delta G_{O_2}[O]_K = -704700 + 122.8T + 38.29T \log C_O \tag{3}$$

Oxygen potentials of ternary oxide and dissolved oxygen derived by equations (2) and (3) are shown in Fig. 5. It is found that the minimum oxygen concentration to form $NaCrO_2$ is lowest among 3 ternary oxides, for example $NaCrO_2$ is formed at 1 wppm at 900 K but Na_3WO_4 is not formed. The difference of free energy of formation between $NaCrO_2$ and Na_3WO_4 is large. Then, $NaCrO_2$ will be preferentially formed when RAFM steel is immersed in liquid sodium. In liquid potassium, the ternary oxide of chromium is formed at lower oxygen concentration. Also chromium may preferentially oxidize, although data for ternary oxides of Fe and W are absent. Therefore, chromium in RAFM steel will preferentially oxidize in NaK same as in Na. However, the minimum oxygen concentration to form oxide in NaK may be lower than that in Na.

When Na or NaK is used in irradiation rig of HFTM, they must be purified, because they may contain much oxygen and oxide. Cold trapping is the purification method available to achieve the low oxygen concentration in Na or NaK. The lower operating temperature limits established for Na and NaK on the basis of experience in dynamic alkali metal loops are ~120 °C and ~30 °C, respectively [9]. The oxygen level that can be achieved at the lower operating temperature limit is ~1 wppm in Na and ~16 wppm in NaK from the values in Fig. 3. However, in this report, the oxygen concentration in Na or NaK is assumed to be 100ppm to evaluate conservatively the corrosion depth. Under the assumptions 1) to 3) mentioned in section 2.1, the corrosion depth due to oxygen dissolved in Na or NaK is 4.00×10^{-5} cm = 0.4 μm, if all of the oxygen is consumed by the oxidation and only $NaCrO_2$ is formed. This value may cause insignificant effect for the mechanical properties of irradiated RAFM steel specimens.
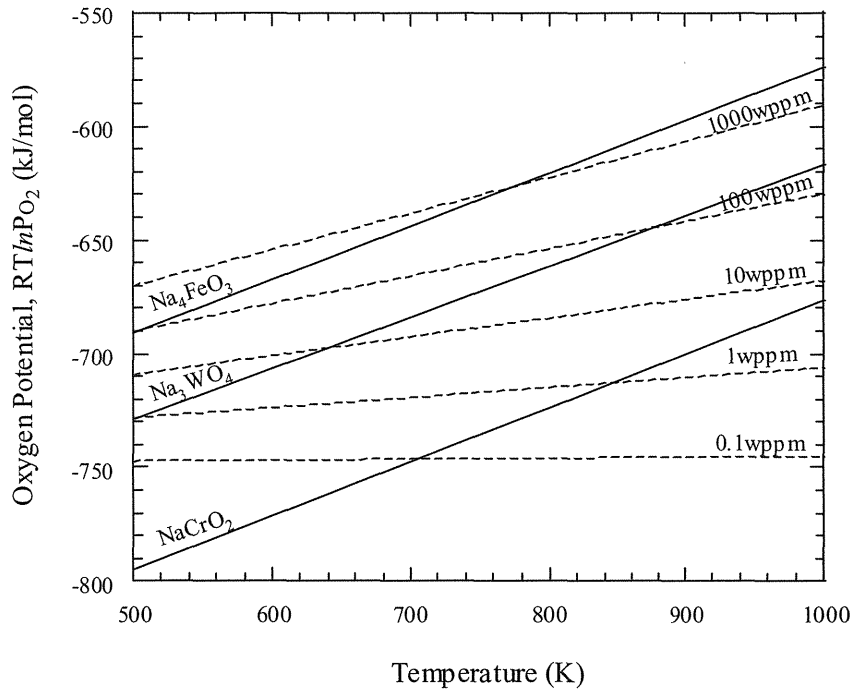
Fig. 5 Oxygen potential of ternary oxide (solid lines) and dissolved oxygen (broken lines) in liquid sodium.
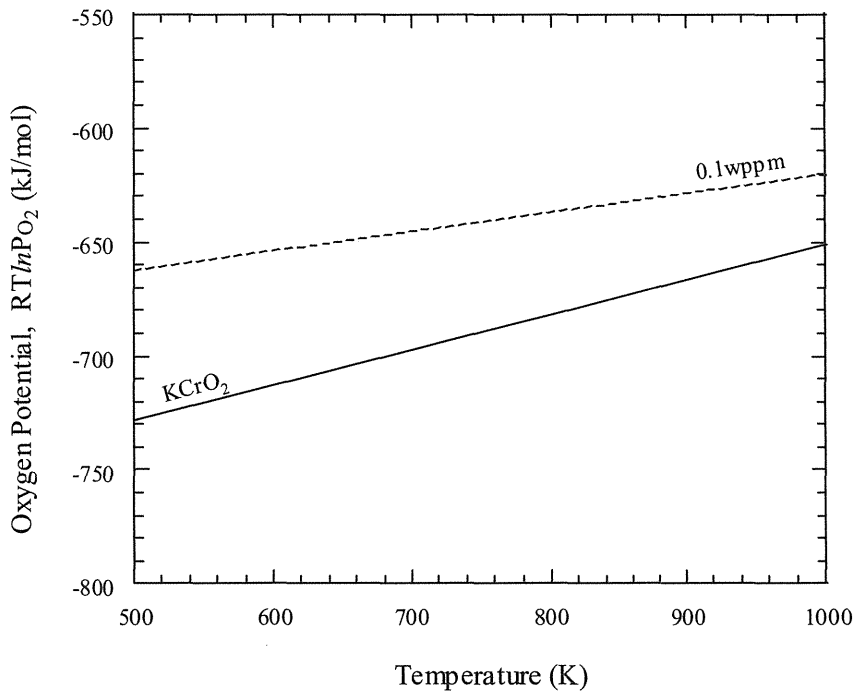

Fig. 6 Oxygen potential of ternary oxide (solid line) and dissolved oxygen (broken line) in liquid potassium.

2. 2. 2. Nitrogen

As above mentioned, the solubility of nitrogen in sodium is extremely small, less than 1 ppb even at 700 °C. Nitride, Na_3N is unstable and nitrogen in usual form dose not react with potassium [17]. Then, the reactions of RAFM steel constituents with nitrogen dissolved in Na or NaK will be insignificant.

For the transport of nitrogen into and out of 9Cr-(1 to 2.5) Mo ferritic steels in sodium, nitrogen loss from these steels was negligible below 823 K, but it increased with the increase in either the sodium-exposure temperature or time at higher temperature [18]. In the other literature nitrogen loss of several 9Cr-2Mo ferritic steels exposed in sodium at 650 °C for 3,000 h, the nitrogen contents were reduced to about 1/5 of initial values of ~0.04 wt% [19]. The mechanism was explained that the nitrogen in the steels diffused to the grain boundaries and dissolved into sodium, and the dissolved nitrogen was transported to the free surface of the sodium adjacent to the argon cover gas and was released into the cover gas. The similar phenomenon might occur in the irradiation rig of HFTM, namely the dissolved nitrogen is released into the filling gas in the NaK expansion volume shown in Fig. 1. Six kinds of (5 to 15) Cr-1Mo ferritic steels with nitrogen content of 0 to 0.002 wt% were also exposed to the flowing sodium at 600 and 650 °C for up to 3,000 h [20]. However, noticeable changes in nitrogen contents of the ferritic steels were not observed although some of them showed slight decrease of nitrogen contents. It should be noticed that nitrogen contents as well as the contents of Cr and Mo of materials differed in these two reports [19, 20]. As the nitrogen content in RAFM steel is specified to be small to achieve the low activation, the nitrogen loss in the specimens will not be noticeable during the irradiation.

2. 2. 3. Carbon

It is well known that the decarburization or carburization of steels occurs when exposed to flowing sodium at high temperatures. The decarburization that the carbon in the steels dissolves into the liquid metal often occurs in the chromium molybdenum steels with high carbon contents, while the carburization occurs in the stainless steels with low carbon contents. These phenomena affect the strength and ductility of materials.

The solubility of carbon in sodium is not large, 37 wppm even at 700 °C. However, the carbon concentration in sodium is affected by the ingress of carbonaceous materials into sodium, and a dynamic equilibrium for carbon in sodium is generally established as a balance between the carbon sources and sinks in the system [9]. In bimetallic sodium systems consisting of austenitic and ferritic steels, decarburization of the ferritic material leads to carburization of the austenitic stainless steels [18]. Six kinds of (5 to 15) Cr-1Mo ferritic steels and type 316 stainless steel were exposed to the flowing sodium in the sodium loop and the concentration or activity in sodium was neither monitored nor controlled, but the carbon activity seemed to be almost determined by the

construction material of specimen holders and loop system, type 316 stainless steel [20, 21]. Carbon contents of steel specimens in those works are summarized in table 4. Type 316 stainless steel specimens hardly decarburized or carburized at any temperatures. However, the decarburization was observed for the 5Cr, 7Cr and 9Cr ferritic steels. The lower the chromium content and higher the temperature, the more the ferritic steels decarburized. The specimens for 5Cr, 7Cr and 9Cr ferritic steels apparently acted as the carbon source and the loop system as the carbon sink.

Table 4   Carbon content of steel specimens before and after exposure in sodium [20,21]

Unit: wt%

Steel	600 °C, 2990 h			650 °C, 3156.5 h			700 °C, ~3350 h		
	Before	After	After/ Before	Before	After	After/ Before	Before	After	After/ Before
5Cr	0.096	0.066	0.69	0.098	0.007	0.07	0.100	0.004	0.04
7Cr				0.101	0.058	0.57	0.100	0.004	0.04
9Cr	0.097	0.094	0.97	0.100	0.084	0.84	0.100	0.004	0.04
11Cr				0.108	0.102	0.94			
13Cr	0.102	0.104	1.02	0.111	0.104	0.94			
15Cr				0.100	0.098	0.98			
316 SS	0.042	0.042	1.00	0.043	0.047	1.09	0.044	0.046	1.05

The carbon transfer between the carbon source and sink through Na (or NaK) may occur in the irradiation rig of HFTM. If the alloy other than the RAFM steel specimens contacts with Na (or NaK) of the irradiation rig, it will act as the carbon source or sink to decarburize or carburize the specimens. It is ambiguous to judge which process will be promoted because the differences of the effect on the decarburization or carburization between molybdenum and tungsten are still unknown.

The change of carbon content in RAFM steel specimens is estimated when no other kind of materials is used for specimens and the specimen holder. Since the data such as activities of Fe, Cr and W in RAFM steel are not known, it is assumed that the carbon dissolves into Na (or NaK) from the specimens until the carbon concentration in Na (or NaK) reaches to the solubility (e.g. 37 wppm at 700 °C) from the initial value of zero. The change of carbon content in RAFM steel results in 4×10^{-4} wt% even at 700 °C under the assumption 1) mentioned in section 2.1, and it is much smaller than the original carbon content, 0.1 wt%.

3. Conclusion

The compatibility of RAF with Na (or NaK) in the irradiation rig of HFTM was addressed. The

corrosion depth of RAFM steel specimens, due to the dissolution of metallic elements into the liquid metal and the oxide formation with oxygen dissolved in the liquid metal, will be less than $1\mu m$ if the liquid metal is purified with cold trap. It is insignificant for changing the performances of RAFM steel. When specimens in the irradiation rig and the specimen holder of rig are made of RAFM steel, the specimens will hardly decarburize or carburize. The nitrogen content in RAFM steel specimens will not noticeably decrease.

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国際単位系 (SI) と換算表

表1　SI基本単位および補助単位

量	名　称	記号
長　　さ	メ ー ト ル	m
質　　量	キログラム	kg
時　　間	秒	s
電　　流	アンペア	A
熱力学温度	ケ ル ビ ン	K
物　質　量	モ ル	mol
光　　度	カ ン デ ラ	cd
平　面　角	ラ ジ ア ン	rad
立　体　角	ステラジアン	sr

表3　固有の名称をもつSI組立単位

量	名　称	記号	他のSI単位による表現
周　波　数	ヘ ル ツ	Hz	s^{-1}
力	ニュートン	N	$m\cdot kg/s^2$
圧力，応力	パスカル	Pa	N/m^2
エネルギー,仕事,熱量	ジュール	J	$N\cdot m$
工率，放射束	ワ ッ ト	W	J/s
電気量，電荷	クーロン	C	$A\cdot s$
電位,電圧,起電力	ボ ル ト	V	W/A
静　電　容　量	ファラド	F	C/V
電　気　抵　抗	オ ー ム	Ω	V/A
コンダクタンス	ジーメンス	S	A/V
磁　　　束	ウェーバ	Wb	$V\cdot s$
磁　束　密　度	テ ス ラ	T	Wb/m^2
インダクタンス	ヘンリー	H	Wb/A
セルシウス温度	セルシウス度	℃	
光　　束	ル ー メ ン	lm	$cd\cdot sr$
照　　度	ル ク ス	lx	lm/m^2
放　射　能	ベクレル	Bq	s^{-1}
吸　収　線　量	グ レ イ	Gy	J/kg
線　量　当　量	シーベルト	Sv	J/kg

表2　SIと併用される単位

名　称	記号
分，時，日	min, h, d
度，分，秒	°, ′, ″
リットル	l, L
トン	t
電子ボルト	eV
原子質量単位	u

$1\,eV=1.60218\times10^{-19}J$

$1\,u=1.66054\times10^{-27}kg$

表4　SIと共に暫定的に維持される単位

名　称	記号
オングストローム	Å
バ ー ン	b
バ ー ル	bar
ガ ル	Gal
キ ュ リ ー	Ci
レ ン ト ゲ ン	R
ラ ド	rad
レ ム	rem

$1\,Å=0.1\,nm=10^{-10}\,m$

$1\,b=100\,fm^2=10^{-28}\,m^2$

$1\,bar=0.1\,MPa=10^5\,Pa$

$1\,Gal=1\,cm/s^2=10^{-2}\,m/s^2$

$1\,Ci=3.7\times10^{10}\,Bq$

$1\,R=2.58\times10^{-4}\,C/kg$

$1\,rad=1\,cGy=10^{-2}\,Gy$

$1\,rem=1\,cSv=10^{-2}\,Sv$

表5　SI接頭語

倍数	接頭語	記号
10^{18}	エ ク サ	E
10^{15}	ペ タ	P
10^{12}	テ ラ	T
10^9	ギ ガ	G
10^6	メ ガ	M
10^3	キ ロ	k
10^2	ヘ ク ト	h
10^1	デ カ	da
10^{-1}	デ シ	d
10^{-2}	センチ	c
10^{-3}	ミ リ	m
10^{-6}	マイクロ	μ
10^{-9}	ナ ノ	n
10^{-12}	ピ コ	p
10^{-15}	フェムト	f
10^{-18}	ア ト	a

(注)

1. 表1−5は「国際単位系」第5版，国際度量衡局 1985年刊行による。ただし，1 eV および1 uの値はCODATAの1986年推奨値によった。
2. 表4には海里，ノット，アール，ヘクタールも含まれているが日常の単位なのでここでは省略した。
3. bar は，JISでは流体の圧力を表わす場合に限り表2のカテゴリーに分類されている。
4. EC閣僚理事会指令では bar，barn および「血圧の単位」mmHgを表2のカテゴリーに入れている。

換　算　表

力	N(=10^5dyn)	kgf	lbf
	1	0.101972	0.224809
	9.80665	1	2.20462
	4.44822	0.453592	1

粘　度　$1\,Pa\cdot s(N\cdot s/m^2)=10\,P(ボアズ)(g/(cm\cdot s))$

動粘度　$1\,m^2/s=10^4\,St(ストークス)(cm^2/s)$

圧力	MPa(=10 bar)	kgf/cm²	atm	mmHg(Torr)	lbf/in²(psi)
	1	10.1972	9.86923	7.50062×10^3	145.038
	0.0980665	1	0.967841	735.559	14.2233
	0.101325	1.03323	1	760	14.6959
	1.33322×10^{-4}	1.35951×10^{-3}	1.31579×10^{-3}	1	1.93368×10^{-2}
	6.89476×10^{-3}	7.03070×10^{-2}	6.80460×10^{-2}	51.7149	1

エネルギー・仕事・熱量	J(=10^7erg)	kgf・m	kW・h	cal（計量法）	Btu	ft・lbf	eV
	1	0.101972	2.77778×10^{-7}	0.238889	9.47813×10^{-4}	0.737562	6.24150×10^{18}
	9.80665	1	2.72407×10^{-6}	2.34270	9.29487×10^{-3}	7.23301	6.12082×10^{19}
	3.6×10^6	3.67098×10^5	1	8.59999×10^5	3412.13	2.65522×10^6	2.24694×10^{25}
	4.18605	0.426858	1.16279×10^{-6}	1	3.96759×10^{-3}	3.08747	2.61272×10^{19}
	1055.06	107.586	2.93072×10^{-4}	252.042	1	778.172	6.58515×10^{21}
	1.35582	0.138255	3.76616×10^{-7}	0.323890	1.28506×10^{-3}	1	8.46233×10^{18}
	1.60218×10^{-19}	1.63377×10^{-20}	4.45050×10^{-26}	3.82743×10^{-20}	1.51857×10^{-22}	1.18171×10^{-19}	1

$1\,cal=4.18605\,J（計量法）$

$=4.184\,J　（熱化学）$

$=4.1855\,J　（15\,℃）$

$=4.1868\,J（国際蒸気表）$

仕事率　1 PS (仏馬力)

$=75\,kgf\cdot m/s$

$=735.499\,W$

放射能	Bq	Ci
	1	2.70270×10^{-11}
	3.7×10^{10}	1

吸収線量	Gy	rad
	1	100
	0.01	1

照射線量	C/kg	R
	1	3876
	2.58×10^{-4}	1

線量当量	Sv	rem
	1	100
	0.01	1

Compatibility of Reduced Activation Ferritic/Martensitic Steel Specimens with Liquid Na and NaK in Irradiation Rig of IFMIF