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Mechanical properties of low activation Cr–Mn austenitic steels changes in liquid lithium

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The mechanical properties of Fe–0.06C–12Cr–14Mn–4Ni–Al–Mo, Fe–0.10C–12Cr–20Mn–W, Fe–0.25C–12Cr–20Mn–2W, Fe–0.06C–17Cr–19Mn–3Ni–Nb–N, Fe–0.07–13Cr–20Mn–N steels attacked by liquid lithium were studied. Preexposure of steels was performed in static isothermal lithium at 723 and 873 K; in the hot leg of a convection loop at 723 K, and in inert atmosphere at 723 and 873 K for 2600 h. Lithium contained up to 400 ppm nitrogen and up to 1% hydrogen. The mechanical properties were determined by tensile test in lithium and in vacuum at a strain rate of 1×10^{-5}–1×10^{-3} s^{-1}.

It was shown that mechanical properties of tested steels after exposure in the lithium changed more than for Cr–Ni steels. The strong embrittlement of steels containing nitrogen is associated with intergranular penetration of lithium. The character of other steels mechanical properties changes is difficult to explain and may be associated with nonmetallic impurities redistribution and steel phase composition changes. The main mechanical properties change took place continually for the first 1000 h at 723 K exposure. Noticeable change in the mechanical properties of the steels exposed to lithium at 873 K occured even until 2600 h of exposure. The effect of strength and ductility reduction through absorption did not occur.

1. Introduction

Rapid decay of induced radioactivity is one of the attractive properties of Cr–Mn austenitic steels along with good operating properties, and this ranks these steels with desirable structural materials of fusion reactors [1]. Use of these steels in liquid-metal systems of a fusion reactor blanket as structural materials requires detailed study of their compatibility with liquid lithium or its alloys, which play the role of tritium breeder material or coolant, and the environmental effects on their mechanical properties.

At present, the influence of lithium on the mechanical properties of austenitic Cr–Ni steels is well-known. From the point of view of the mechanical properties' stability, the Cr–Ni steels are compatible with lithium to temperatures of 773–873 K [2]. Today's information on the effects of lithium on the mechanical properties of Cr–Mn steels is restricted and fragmentary [3,4].

The main purpose of the present work is to create systematic data on the corrosion and absorption effects of static lithium, flowing lithium and lithium with hydrogen impurity at temperatures of 723–873 K on the mechanical properties of Cr–Mn austenitic steels, and to reveal the mechanisms of this influence.

2. Experiment

2.1. Materials

The following austenitic steels, where nickel is completely or partially replaced by manganese, were chosen for the experiments: Fe–0.06C–12Cr–14Mn–4Ni–Al–Mo, Fe–0.10C–12Cr–20Mn–W, Fe–0.25C–12Cr–20Mn–2W, Fe–0.07C–13Cr–20Mn–N, Fe–0.06C–17Cr–19Mn–3Ni–Nb–N. Tensile specimens for examination with dimensions of 20 × 3 mm were produced by stamping of sheet with thickness of 1 mm (3 mm – for steel Fe–0.06C–17Cr–19Mn–3Ni–Nb–N). After manufacturing, the specimens were polished and an-

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Table 1
Mechanical properties in tension of Cr–Mn steels after exposure under various conditions: air, 293 K, $\dot{\epsilon} = 1 \times 10^{-3}\ s^{-1}$

Steel	Conditions of corrosion tests			Properties		
	T (K)	Time (h)	Medium	$\sigma_{0.2}$ (MPa)	σ_U (MPa)	σ (%)
Fe–0.10C–12Cr–20Mn–W	Without exposure			315	755	50
	723	1050	Ar	210	660	30
			Li, stat	225	615	24
			Li, dyn	155	615	55
		2600	Ar	265	825	30
			Li, stat	280	675	20
			Li, dyn	175	615	55
	873	1100	Ar	240	675	30
			Li, stat	320	630	23
		2600	Ar	425	700	30
			Li, stat	445	765	20
Fe–0.06C–12Cr–14Mn–4Ni–Al–Mo	Without exposure			355	610	50
	723	1050	Ar	355	610	34
			Li, stat	365	635	49
			Li, dyn	205	655	68
		2600	Ar	410	750	50
			Li, stat	400	730	38
	873	1100	Ar	295	600	50
			Li, stat	280	580	39
		2600	Ar	335	720	41
			Li, stat	320	675	34
Fe–0.25C–12Cr–20Mn–W	Without exposure			–	800	50
	723	1500	Ar	410	1035	43
			Li, stat	400	1030	46
			Li, dyn	395	1005	37
Fe–0.06C–17Cr–19Mn–3Ni–Nb–N	Without exposure			565	835	36
	723	1050	Ar	560	840	35
			Li, stat	515	850	33
	873	1035	Ar	–	835	33
			Li, stat	230	320	0
Fe–0.07C–13Cr–20Mn–N	Without exposure			475	820	52
	723	1050	Ar	480	795	52
			Li, stat	485	805	45
			Li, dyn	310	660	60
		2600	Ar	570	985	40
			Li, stat	565	950	40
	873	1050	Ar	440	790	34
			Li, stat	265	360	1
		2600	Ar	525	950	29
			Li, stat	320	450	0

nealed at 1373 K for 0.25 h. Electrolytic polishing in acid solutions was the final operation.

Lithium with a content of nitrogen additive of not more than 400 wppm was used as a test environment.

2.2. Testing technique

The main attention in this work was given to the examination of the steels' behaviour under the load against the background of corrosion and absorption effects of liquid lithium. The mechanical properties of steels in vacuum and lithium under chosen conditions were determined according to the results of tensile tests of not less than three specimens. Testing was carried out at temperatures of 293, 523, 723 and 823 K and strain rates of 1×10^{-5}–1×10^{-3} s^{-1}. Steels were tested both after initial heat treatment and after exposure without load in argon and liquid lithium. Accepted techniques allow us to determine the influence of thermal effects on the steels, the contribution of corrosion and absorption effects of lithium to variation of their mechanical properties, and also the resultant variation of the steels properties.

The contribution of thermal effect to variation of the steels' properties was determined from a comparison of results of the tensile tests of the specimens under initial conditions and after exposure in argon at temperatures of 723 and 873 K for up to 2600 h. The contribution of corrosion effects of liquid lithium to variation of the mechanical properties of the steels was determined from a comparison of results of the tensile tests in vacuum, on specimens exposed without load in argon and in liquid lithium environments. The contribution of absorption effects of lithium was determined from comparisons of results of the mechanical tests at 523 K in vacuum and in lithium of specimens exposed without load in argon. Variation of lithium's absorption effect contribution as a result of structural and compositional changes of the steels in the case of corrosion effect of lithium was determined from a comparison of results of the tensile tests at 523 K in both vacuum and lithium of specimens exposed in lithium without load at various conditions. The resultant variation of the mechanical properties of the steels in lithium environment was determined from a comparison of results of the tensile tests at 523 K both in vacuum of specimens exposed in argon without load and in lithium of specimens exposed in lithium environment.

Preliminary exposure of specimens was carried out under static isothermal conditions in ampoules at temperatures of 723 and 873 K, and under flowing conditions in the section of the convection loop at a temperature of 723 K for 1050–2600 h. The temperature difference between the hot and cold legs of the loop was 50 K and the flow rate of lithium was about 0.02 m/s. The material of the ampoules and loop was similar to the content of steels being examined. A controlled quantity of lithium hydride was added in lithium during preliminary exposure of specimens in order to reveal the influence of hydrogen additive in lithium on the mechanical properties of the Cr–Mn steels. The concentration of hydrogen in lithium was varied from approximately 0.02 to 1.1%.

The influence of liquid lithium on the mechanical properties of the steels was studied in connection with variation of their structure and analyses and phase content. Analyses were carried out by microscopic, micro X-ray and autoradiographic examinations. The modes of failure of specimens was determined by means of fractographic examinations on a scanning electron microscope.

3. Results and discussion

Data on the mechanical properties of Cr–Mn steels obtained in the present examination are specified in table 1. It is clear that thermal effects as a result of exposure in argon atmosphere at temperatures of 723 and 873 K led to ductility reduction. The steels Fe–0.06C–12Cr–14Mn–4Ni–Al–Mo and Fe–0.06C–17Cr–19Mn–3Ni–Nb–N alloyed with such carbide-forming elements as Nb and Mo have the greatest resistance to the thermal effect. Such behaviour of steels is connected with formation of carbide phases of the $M_{23}C_6$-type along the grain boundaries as described in the literature [3,4]. Failure of the Cr–Mn steels subjected to thermal effect had intergranular elastic patterns.

Corrosion effect of lithium on the Cr–Mn steels led to a complicated pattern of variation of their mechanical properties, which depended on temperature, on the pattern of liquid lithium effect, on lithium purity and on composition of the steels. When temperature of lithium exposure did not exceed 723 K, the examined steels tended to display increasing ductility. It was especially revealed in the case of flowing lithium effect on the steels. The mechanical properties of the steel Fe–0.25C–12Cr–20Mn–W were the most stable. Penetration of lithium and formation of a corrosion layer in the case of static isothermal lithium exposure was not practically observed. The failure had an elastic pattern. Flowing lithium led to the formation of a corrosion

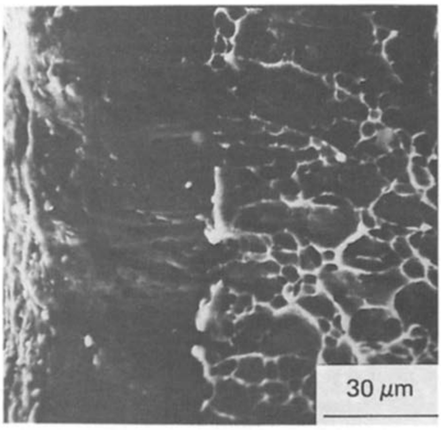

Fig. 1. Fracture surface of steel Fe–0.10C–12Cr–20Mn–W tensile-tested in vacuum, 293 K, $\dot{\epsilon} = 1 \times 10^{-3}$ s^{-1} after exposure in flowing lithium for 1050 h at 723 K.

Table 2
Mechanical properties of steel Fe–0.06C–12Cr–14Mn–4Ni–Al–Mo tensile tested in vacuum at $\dot{\epsilon} = 1 \times 10^{-3}$ s^{-1} at various temperatures after exposure under various conditions, 1500 h

Testing temperature		Medium	Properties		
Temperature of exposure (K)	Temperature of tensile tests (K)		$\sigma_{0.2}$ (MPa)	σ_u (MPa)	δ (%)
723	293	Ar	375	630	48
		Li, stat	350	600	37
	723	Ar	220	400	33
		Li, stat	250	385	28
873	293	Ar	330	630	54
		Li, stat	300	550	34
	873	Ar	180	350	30
		Li, stat	200	320	24

layer with a porous structure, which contained (according to data of micro X-ray analysis) Cr – 1%, Ni – 0.3%, Mn – 2.5% on the surface of all tested steels, independent of initial composition of the steels. This points to a process of selective corrosion in the case of flowing lithium. Autoradiographic analysis showed the presence of lithium in the corrosion layer. The corrosion layer formed on the surface of the steels cracked when the specimens were deformed to approximately 3%. Fractographic examinations (fig. 1) showed a brittle failure pattern of the corrosion layer and an elastic failure pattern in the reminder of the specimen. The corrosion layer was thin in comparison with the thickness of the specimens and it did not have an influence on the mechanical properties of the specimens on the whole. Obtained results and data of other work [5] allow us to concluded that the observed increase in plasticity of the steels examined is in connection with removal of interstitial impurities from steels by lithium.

Another picture was observed in case of mechanical testing of the steel specimens that were exposed in lithium at a temperature of 873 K. The steels Fe–0.10C–12Cr–20Mn–W, Fe–0.25C–12Cr–20Mn–2W, and Fe–0.06C–12Cr–14Mn–4Ni–Al–Mo showed considerable reduction of ductility and this was manifested in considerable degree by mechanical tests at a temperature of 293 K in comparison with tests at 723 and 873 K (table 2). The steels Fe–0.07C–13Cr–20Mn–N and Fe–0.06C–17Cr–19Mn–3Ni–Nb–N, which contain nitrogen, lost their plasticity completely. Autoradiographic examinations (fig. 2) showed deep (through thickness for 1 mm thick specimens) penetration of lithium into these steels. The failures had a brittle intergranular pattern (fig. 3). It may be connected with intergranular penetration of lithium in these steels and formation of a Li–Cr–N compound (possibly Li_9CrN_5).

It was shown in other works [5,6] that, at temperatures below 573 K, in the structure of Cr–Mn steels held in lithium at 873 K, α' (bcc) and ϵ (hexagonal) martensite phases are formed to a depth that exceeds the depth of penetration of lithium, and this was connected with the destabilization of austenite as a result of selective removal of Mn and C. From the other side,

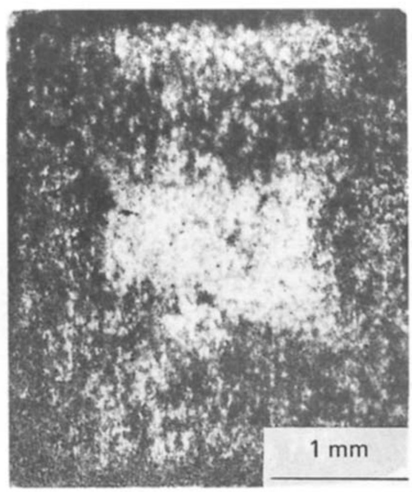

Fig. 2. Autoradiographic lithium trace of cross section of steel Fe–0.06C–17Cr–19Mn–3Ni–Nb–N after exposure in static lithium for 1050 h at 873 K.

the fact that the replacement of the mechanism of plastic deformation of metastable Cr–Mn steels by the sliding of dislocations in the lattice of austenite with deformation due to $\gamma \rightarrow \alpha'$, ϵ conversion at temperatures of 523 K with subsequent brittle failure of α'- and ϵ-phases, is stated in other work [7].

So, the observed behaviour of Cr–Mn steels under the load, which were exposed to lithium at a temperature of 873 K, can be assosiated with penetration of lithium along the grain boundaries (this is especially typical for steels with nitrogen). The austenite is then destabilized due to removal of C and Mn accompanied by formation of α'- and ϵ-phases, and the consequent changing of deformation mechanism at temperatures below 523 K.

In addition, the data showed that the basic variation of mechanical properties of Cr–Mn steels exposed in lithium at 723 K, took place in the first 1000 h. For Cr–Mn steels held in lithium at 873 K, stabilization of mechanical properties for 2600 h was not observed.

The influence of corrosion effect of lithium with the addition of hydrogen on mechanical properties of Cr–Mn steels was examined on the steel Fe–0.06C–12Cr–14Mn–4Ni–Al–Mo. As is clear from fig. 4, exposure of the steel at 873 K in lithium with hydrogen led to the reduction of ductility in the mechanical tests at 293 K. Plasticity of the steel was reduced along with an increase in hydrogen concentration in lithium. Therefore, the presence of hydrogen in lithium led to the amplification of variations of mechanical properties compared to the effect of pure lithium. According to data of other work [5], the presence of hydrogen in

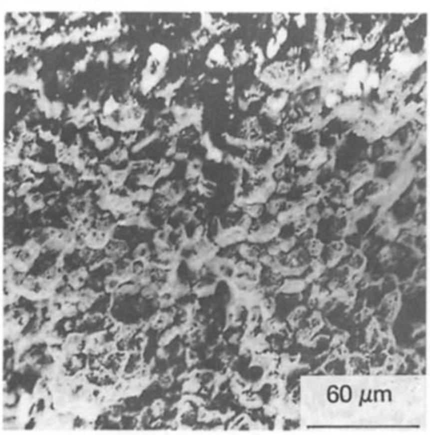

Fig. 3. Fracture surface of steel Fe–0.06C–17Cr–19Mn–3Ni–Nb–N tensile-tested in vacuum, 293 K, $\dot{\epsilon} = 1 \times 10^{-3}\ s^{-1}$ after exposure in static lithium for 1050 h at 873 K.

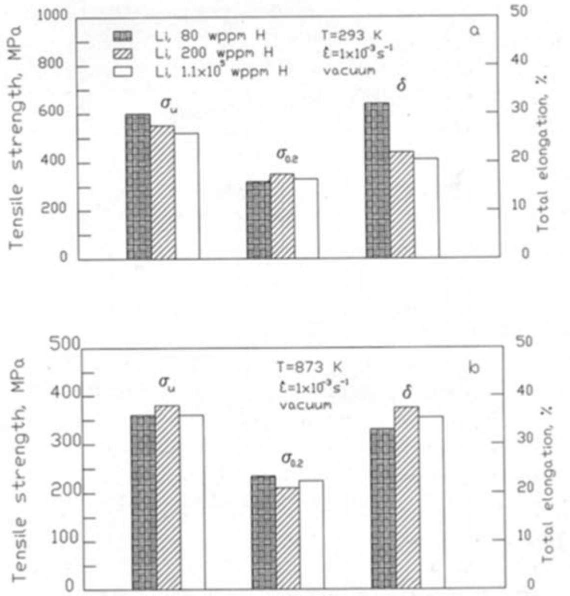

Fig. 4. Mechanical properties of steel Fe–0.06C–12Cr–14Mn–4Ni–Al–Mo tensile-tested after exposure in static lithium for 800 h at 873 K.

lithium intensifies the removal of carbon additive from steel into lithium, and this can cause destabilization of austenite and lead to reduction of steel plasticity according to the reasons stated above. The other reason for reduction of plasticity can be hydrogen embrittlement. The data obtained does not give a clear explanation of reduction of plasticity as a result of the effect of lithium with hydrogen addition.

Testing of Cr–Mn steels in vacuum and lithium at 523 K and $\dot{\epsilon} = 1 \times 10^{-5}\ s^{-1}$ showed no lithium absorp-

Table 3
Mechanical properties of Fe–0.06C–12Cr–14Mn–4Ni–Al–Mo steel tensile tested at 523 K and strain rate $1 \times 10^{-5}\ s^{-1}$ after exposure under various conditions

Conditions of exposure	Environment of tensile test	Properties		
		$\sigma_{0.2}$ (MPa)	σ_U (MPa)	δ (%)
Without exposure	Vacuum	260	495	25
	Lithium	255	490	23
823 K, 1100 h, Ar	Vacuum	245	460	23
	Lithium	250	455	21
823 K, 1100 h, Li static	Vacuum	200	440	21
	Lithium	205	450	18

tion effect influence on the mechanical properties and no variations of absorption effect contribution as a result of corrosion effect of lithium on the steels. This may be seen on an example of Fe–0.6C–12Cr–14Mn–4Ni–Al–Mo steel in table 3. In this investigation samples of the steel were exposed to lithium at 823 K for 0.08 h to wet them before tensile tests at 523 K. Samples tested in vacuum were exposed in vacuum at 823 K for 0.08 h before testing too.

4. Conclusions

Examination carried out in this work allows us to make the following conclusions.

(1) Cr–Mn austenitic steels are less stable and change their mechanical properties considerably due to influence of lithium in comparison with Cr–Ni austenitic steels.

(2) The corrosion effect of lithium gives the main contribution in variation of mechanical properties of Cr–Mn steels on exposure to lithium.

(3) At temperatures which do not exceed 723 K, removal of interstitial impurities is the main mechanism of influence of lithium on the mechanical properties of steels and this leads to an increase in the ductility of steels.

(4) At temperatures which are close to 873 K, the main mechanism of lithium influence is its intergranular penetration, destabilization of austenite due to removal of Mn and C, and this leads to variation of phase content of steels and to a charge of deformation mechanism.

(5) Alloying of Cr–Mn steels with nitrogen in order to stabilize austenite is not permissible because it leads (in contact with lithium) to complete loss of steel ductility due to complete intergranular penetration of lithium.

(6) The presence of hydrogen additive in lithium leads to amplification of trends in variation of mechanical properties of Cr–Mn steels observed under exposure to pure lithium.

(7) Absorption effect of lithium does not lead to variation of mechanical properties of Cr–Mn austenitic steels.

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