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COMPATIBILITY OF MOLYBDENUM-BASE ALLOY TZM WITH $LiF-BeF_2-ThF_4-UF_4$ (68-20-11.7-0.3 mole %) at 1100°C

J. W. Koger and A. P. Litman

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ABSTRACT

The TZM alloy (Mo–0.5% Ti–0.08% Zr–0.02% C) showed a very small amount of attack by the fused fluoride salt ($LiF-BeF_2-ThF_4-UF_4$, 68-20-11.7-0.3 mole %) at 1100°C for 1011 hr. Corrosion manifested itself as leaching of titanium and possibly zirconium from the alloy. The TZM alloy exposed to the salt partially recrystallized, while that exposed to the vapor did not. This recrystallization was attributed to the removal of titanium and zirconium. On the basis of this single test the magnitude and mechanism of corrosion indicate no serious problems for long-term use of TZM in the vacuum distillation processing scheme for the Molten Salt Breeder Reactor. However, the strength properties of the TZM alloy would approach those of unalloyed molybdenum as salt exposure time increased; this is not considered a problem now.

INTRODUCTION

The current success of the Molten Salt Reactor Experiment at ORNL has stimulated work on a thermal Molten Salt Breeder Reactor (MSBR).[1] One of the requirements for a successful MSBR system will be the continuous reconditioning of the fuel salt to remove unwanted fission products. A possibility under study for one step of the salt reprocessing is vacuum distillation.[2] Uranium would be stripped from the fuel salt, and the remaining salt would be distilled at 1000°C and 2 torr. The diluents of the fuel salt, lithium and beryllium fluorides, would distil readily and leave behind the rare-earth and alkaline-earth fission products. This process has been demonstrated in laboratory experiments and with some radioactive salt from the MSRE.[2]

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[1]M. W. Rosenthal et al., MSR Program Semiann. Progr. Rept. Aug. 31, 1968, ORNL-4344, pp. 53–108.

[2]J. R. Hightower and L. E. McNeese, MSR Program Semiann. Progr. Rept. Aug. 31, 1968, ORNL-4344, pp. 306–308.

The strength and corrosion resistance required of a container material for the high-temperature vacuum distillation step eliminate most conventional alloys from consideration. Our preliminary survey disclosed that certain refractory alloys, particularly molybdenum-base materials, may be suitable for this special service. The alloy TZM (Mo—0.5% Ti—0.08% Zr—0.02% C) was selected for an initial experiment because it is stronger and usually more fabricable than pure molybdenum. Accordingly, the experiment reported here provides a preliminary test of the compatibility of TZM alloy with a typical fertile-fissile salt ($LiF-BeF_2-ThF_4-UF_4$, 68-20-11.7-0.3 mole %) at 1100°C. This salt is a strong candidate for the single-fluid MSBR now being designed. No tests were specifically conducted to determine the strength properties of TZM alloy, but conditions caused by the exposure to the salt that could affect the strength were noted.

EXPERIMENTAL PROCEDURE

The experimental system used for this study consisted of a simple capsule fabricated of cold-worked TZM alloy, containing specimens of the same alloy, and shown in Fig. 1. Note that the specimens were located in the salt, at the salt-vapor interface, and in the vapor. The purified salt (60 g) was supplied by the Fluoride Processing Group of the Reactor Chemistry Division. Purification involved sparging with an $HF-H_2$ mixture at 600°C to remove oxides and sulfides and stripping with H_2 at 700°C to remove metallic impurities. The loading operation, which consists of introducing the fluoride salt into the capsule, welding the test capsule, and sealing the outer Inconel protective container, was carried out in an inert-gas atmosphere chamber containing argon purer than 99.995%.

After being tested in the position shown in Fig. 1 for 1011 hr at 1100°C, the capsule was removed from the furnace, inverted to keep the specimens out of the salt, and quenched in liquid nitrogen to retain high-temperature corrosion products. After test, weight changes of the specimens were determined, the salt was analyzed for impurities, and the specimens and capsules were analyzed by x-ray fluorescence and examined metallographically.

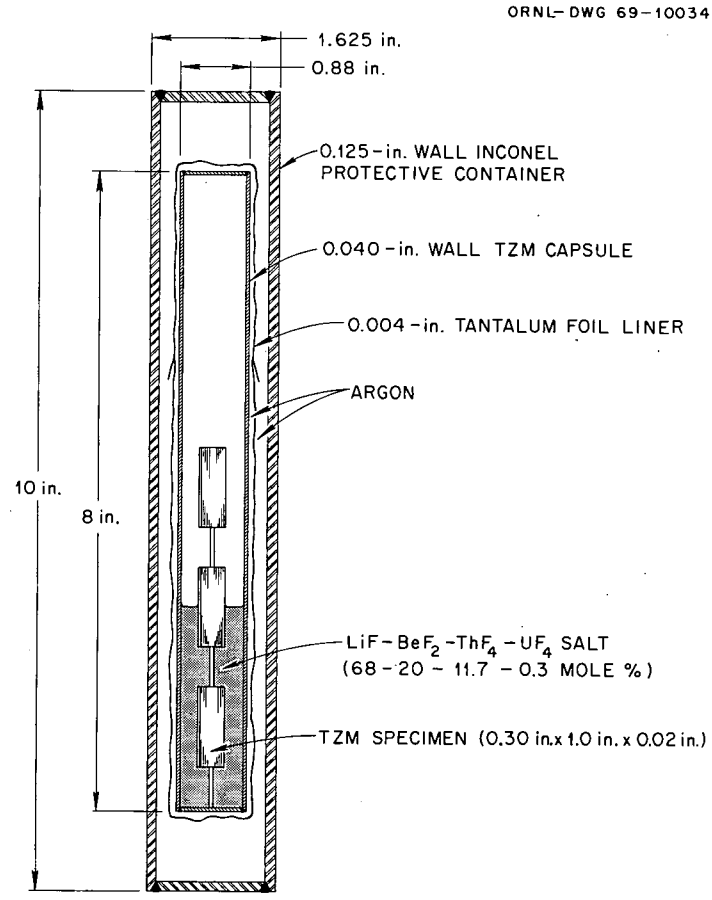

Fig. 1. Schematic Drawing of Corrosion Test Capsule Used to Study Compatibility of TZM Alloy with a Fused Fluoride Salt.

RESULTS AND DISCUSSION

Salt Analysis

Concentrations of the constituents of the salt and its impurities before and after test are given in Table 1. During the experiment titanium, zirconium, and chromium concentrations in the salt increased and that of iron decreased. The titanium and zirconium are intentional alloying additions, but chromium is an unwanted impurity.

Weight Changes

The specimens exposed to the salt showed small (0.5 mg/cm^2) weight gains, and the one exposed to the vapor did not change weight measurably.

Table 1. Chemical Analysis of Fertile-Fissile Salt Exposed to TZM Alloy Capsule for 1011 hr at 1100°C (2010°F)

Constituent	Content, ppm		Constituent	Content, wt %	
	Before	After		Before	After
Mo	< 5	< 10	Li	6.71	7.01
Zr	37	134	Be	2.65	2.55
Ti	74	151	Th	43.1	42.6
Fe	80	38	U	1.75	1.93
Cr	20	97	F	45.5	45.7
O	58	< 50			
H_2O	40	70			

X-Ray Fluorescence and Microprobe Analysis

Table 2 gives the concentrations of the major elements in the TZM alloy as determined by x-ray fluorescence before and after test. Iron was found on the surface and probably caused a major portion of the weight gains, but no quantitative value was obtained. Significantly, the quantitative analysis shows a decrease in titanium concentration, no significant change in zirconium concentration, and a corresponding increase in the concentration of molybdenum after exposure to the salt. Care must be taken in interpreting these results, since the sensitivity of the fluorescence analysis is questionable at these low concentrations and iron was deposited over the surface. The electron microprobe analysis showed 0.3% Ti on the surface and 0.5% Ti in the matrix. The zirconium content was about 0.1% in all portions of the specimen. Any changes at the level of 0.1% are beyond the limit of detection of the instrument. However, these results agree reasonably with the increase in concentration of certain alloying elements in the salt and are in accord with the proposed corrosion mechanism(s). (See Corrosion Reactions and Kinetics.)

Microstructural Changes

Figure 2(a) shows the typical cold-worked structure of the specimens and capsule before test. This figure is also typical of the specimen

Table 2. Concentration of Alloying Elements in TZM Alloy Specimen Before and After Exposure to a Fertile-Fissile Salt at 1100°C for 1011 hr, as Determined by X-Ray Fluorescence Analysis[a]

Sample Analyzed	Content, wt %		
	Mo	Zr	Ti
Untested alloy	99.4	0.08	0.5
Exposed specimens			
in vapor	99.8	0.08	0.1
at interface	99.87	0.09	0.04
in salt	99.90	0.08	0.013

[a]The analysis disclosed substantial iron on the alloy surface after test, but iron was not considered in determining the quantities above.

exposed to the vapor, where no microstructural change occurred. An unetched specimen, Fig. 2(b), exposed to the salt shows no attack at the surface. The same specimen etched, Fig. 2(c), shows recrystallization for a maximum depth of about 0.004 in. Examination of this specimen at a lower magnification, Fig. 2(d), shows that both surfaces recrystallized as the result of test. The inside capsule wall also recrystallized in the same manner.

Recrystallization

In view of the microstructural and chemical changes induced in the TZM alloy by this test, we compared reported recrystallization temperatures for molybdenum and TZM alloy (Table 3). It is clear from the above and from general metallurgical considerations that an increase in annealing time from 1 hr to several thousand hours should lower the recrystallization temperature of TZM alloy only 100 to 200°C. Moreover the presence of as little as 0.01% of a foreign element in solid solution can raise the recrystallization temperature as much as several hundred degrees.[3] Conversely, the removal of alloying constituents would free

[3]R. E. Reed-Hill, Physical Metallurgy Principles, Van Nostrand, Princeton, N. J., 1964, p. 198.

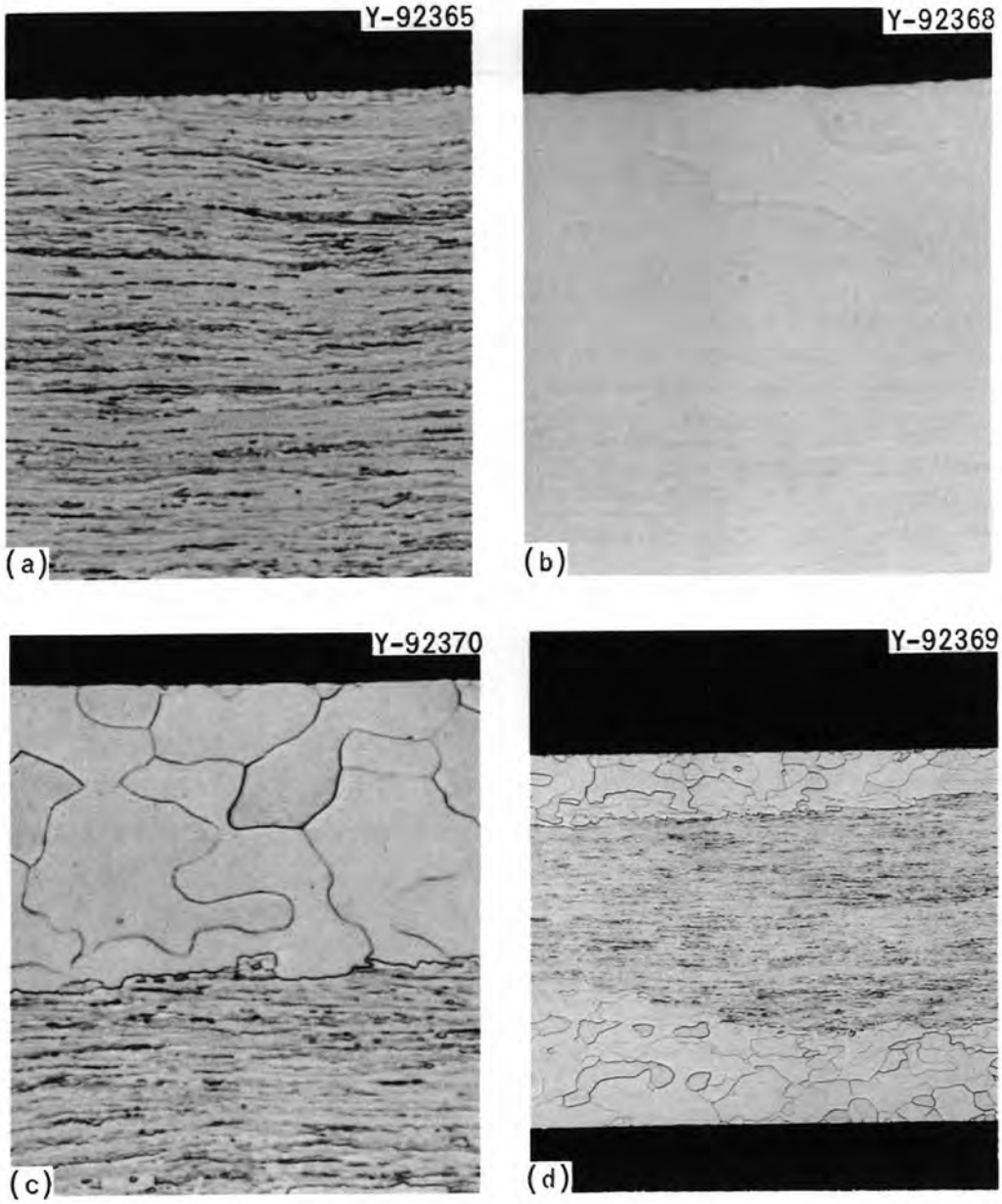

Fig. 2. TZM Alloy Exposed to Fertile-Fissile Salt, $LiF-BeF_2-ThF_4-UF_4$ (68-20-11.7-0.3 mole %) for 1011 hr at 1100°C. (a) Typical cold-worked structure of capsule and specimens before test; also the structure of the specimen exposed to the vapor during test. 500×. Etchant: H_2O, H_2O_2, H_2SO_4. (b) As-polished capsule and specimen exposed to salt. 500×. (c) Capsule and specimen exposed to salt. 500×. Etchant: H_2O, H_2O_2, H_2SO_4. (d) Specimen exposed to salt. 100×. Etchant: H_2O, H_2O_2, H_2SO_4.

Table 3. Recrystallization Behavior of Wrought, Stress-Relieved, Unalloyed Molybdenum and TZM Alloy

Alloy	Temperature (°C)	Time (hr)	Percent Recrystallization	Reference
Unalloyed Mo	1130	1	100	a
TZM	560	4400	0	b
TZM	1100	1	0	a
TZM	1160	4400	85	b
TZM	1250	4400	100	b
TZM	1390	1	100	a

[a]B. A. Wilcox, p. 26 in Refractory Metal Alloys, Metallurgy and Technology, ed. by I. Machlin, R. T. Begley, and E. D. Weisert, Plenum Press, New York, 1968.

[b]D. H. Jansen, Fuels and Materials Development Program Quart. Progr. Rept. Sept. 30, 1968, ORNL-4350, pp. 107–111, and private communication.

the grain boundaries and allow them to move to form new grains. Thus, the enhanced recrystallization (lower recrystallization temperature) in the samples and capsule of this experiment is due primarily to the removal of the titanium and possibly zirconium from the molybdenum matrix. This is further substantiated by the lack of recrystallization in the samples exposed to the vapor, where the composition changed much less.

The addition of carbon and one or more group IV-A elements to molybdenum greatly increases the recrystallization temperature.[4] Thus, carbon removal from the alloy should likewise change recrystallization behavior. However, carbon analyses show no difference (about 0.035% C in each) between exposed and unexposed TZM samples, so this effect is very small or absent. Although carbon mass transport is common in liquid metal systems, especially alkali metals, it is not considered a problem in fused fluoride systems.

[4]W. H. Chang, A Study of the Influence of Heat Treatment on Microstructure and Properties of Refractory Alloys, ASD-TDR-62-211 (April 1962).

Strength

The molybdenum-base TZM alloy is about the best documented refractory alloy in which base metal strength is improved by precipitation hardening. This alloy is strengthened by the formation of fine carbides of titanium and zirconium as well as by cold working, and its ultimate tensile strength is double or triple that of unalloyed molybdenum. The 100-hr rupture strength of TZM at 1100°C is also much greater than that of molybdenum.[5] Although TZM is much more difficult to fabricate than commercial alloys and many refractory alloys, it is usually much easier to work than unalloyed molybdenum. Thus, as an engineering material TZM has many advantages over molybdenum.

Comparing the strength and ductility of wrought, stress-relieved, and recrystallized TZM, Wilcox *et al.*[6] noted a significant increase in yield and ultimate strengths due to working at test temperatures of 1200 to 1300°C. At 1550°C after recrystallization of the wrought sample there was relatively little difference in the materials. However, at 1100°C, the temperature of our capsule test, Wilcox's recrystallized alloy had much lower strength than the wrought alloy. Thus, the use of a stress-relieved TZM alloy for conditions given in this experiment should also be considered.

Although TZM would generally be favored over molybdenum for the previous reasons, through the loss of its alloying elements (titanium and zirconium) during exposure to the fused fluoride salt the composition and the strength properties of the cold-worked TZM approach those of unalloyed recrystallized molybdenum. Although unalloyed molybdenum or recrystallized TZM is weaker than the initial cold-worked material, the strength of the exposed material would probably be ample for the loads proposed in the MSBR vacuum distillation system. However, before the depleted TZM is used, it should be tested to more carefully define

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[5]T. E. Tietz and J. W. Wilson, Behavior and Properties of Refractory Metals, Stanford University Press, California, 1965, pp. 156–205.

[6]B. A. Wilcox, A. Gilbert, and B. C. Allen, Intermediate Temperature Ductility and Strength of Tungsten and Molybdenum TZM, AFML-TR-66-89 (April 1966).

the strength properties of the recrystallized material. An advantageous trade-off with these mechanical property changes is, of course, that pure molybdenum is more resistant than TZM to the fluoride salts of interest to the MSRP. Thus, several benefits come from fabricating the system with TZM while others accrue from the "conversion" of TZM to molybdenum during the fluoride salt exposure.

Corrosion Reactions and Kinetics

In fluoride salt systems one of the major corrosion reactions is the oxidation of one of the constituents of the container alloy by the reduction of a less stable impurity metal fluoride initially in the salt,[7] for example

$$Cr + FeF_2 \rightarrow CrF_2 + Fe \ . \tag{1}$$

The reduced metal substitutes for the oxidized metal on the container material. This type of reaction apparently occurred in our experiment involving the strong reducing agents titanium and zirconium:

$$Ti + FeF_2 \rightarrow TiF_2 + Fe \ , \tag{2}$$

$$Zr + 2FeF_2 \rightarrow ZrF_4 + 2Fe \ . \tag{3}$$

The reported[8] free energy changes for the reactions shown by Eqs. (1), (2), and (3) are strongly negative, and Eqs. (2) and possibly (3) seem to be indicated by the results reported above. As noted earlier, the iron metal that formed in these reactions deposited in thin layers on the container and specimens.

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[7]W. R. Grimes, G. M. Watson, J. H. DeVan, and R. B. Evans, "Radio-Tracer Techniques in the Study of Corrosion by Molten Fluorides," pp. 559–574 in Conference on the Use of Radioisotopes in the Physical Sciences and Industry, September 6–17, 1960, Proceedings, Vol. III, International Atomic Energy Agency, Vienna, 1962.

[8]A. Glassner, The Thermochemical Properties of the Oxides, Fluorides, and Chlorides to 2500°K, ANL-5750 (1957).

Alternatively the fuel salt corrosion reactions in which UF_4 is reduced to UF_3 also may have occurred to remove titanium and zirconium from the alloy:

$$Ti + 2UF_4 \rightarrow TiF_2 + 2UF_3 \ , \tag{4}$$

$$Zr + 4UF_4 \rightarrow ZrF_4 + 4UF_3 \ . \tag{5}$$

However, no data with which to determine the extent of these reactions are available.

Assuming that the removal of the elements from the TZM was controlled by solid-state diffusion, one can calculate from the increase of the titanium and zirconium in the salt the apparent diffusion coefficients of titanium and zirconium in the TZM alloy. From these one can estimate the amount of those materials that would be removed at different times and temperatures. In regard to the zirconium removal, we feel that the salt analysis is correct and that the instruments involved in the fluorescence and microprobe analyses are not sufficiently sensitive to measure the movement of the zirconium.

The total amount of material, M_t, that diffuses from the alloy held under isothermal conditions with a zero surface concentration is given by[9]

$$M_t = 2C_0\sqrt{Dt/\pi} \ , \tag{6}$$

where

C_0 = the concentration of the diffusing element,

D = the diffusion coefficient, and

t = the time.

We calculated $D = 1.2 \times 10^{-12}$ cm^2/sec for titanium in TZM and 2.9×10^{-11} cm^2/sec for zirconium in TZM at 1100°C. We did not calculate for chromium removal, as its concentration fluctuated from sample to sample and we could not assume that it was distributed homogeneously through the alloy.

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[9]J. Crank, The Mathematics of Diffusion, Clarendon Press, Oxford, England, 1956, p. 11.

The expression

$$t \sim X^2/D \ , \tag{7}$$

where X is the distance of composition change, is very useful in calculating approximately whether the composition has changed appreciably by diffusion under a given set of circumstances. For example, we can calculate the time required for appreciable removal — concentration between the initial and ultimate concentrations — of the diffusing element at a certain distance from the surface.

From the calculated diffusion coefficients and the experimental time of 1011 hr, we find the depths of removal of titanium and zirconium, respectively, are 0.0008 and 0.0040 in. Since the microstructures show recrystallization for a distance of about 0.004 in., we may assume that the calculated diffusion coefficient for zirconium may be more accurate than that for titanium — that is, the salt analysis for the titanium may be somewhat in error. Extrapolation of the calculated values shows that it would require 4000 hr to recrystallize an additional 0.004 in. of material. This illustrates the decrease of the corrosion rate with time and the general usefulness of TZM alloy for MSR reprocessing service.

CONCLUSIONS

1. This test showed negligible corrosion of the TZM alloy by the fused fluoride salt (LiF-BeF_2-ThF_4-UF_4, 68-20-11.7-0.3 mole %) at 1100°C for over 1000 hr.

2. Corrosion manifested itself as leaching of titanium and possibly zirconium from the alloy. FeF_2 initially present in the salt oxidized the alloying elements to fluorides dissolved in the salt bath. The iron metal resulting from the reaction deposited in thin layers on the specimens and container. We found that D_{Ti} and D_{Zr} were 1.2×10^{-12} and 2.9×10^{-11} cm^2/sec, respectively, at 1100°C in the alloy.

3. The TZM alloy exposed to the salt partially recrystallized, while the TZM alloy simultaneously exposed to the vapor did not. This recrystallization was attributed to the removal of titanium and zirconium.

4. On the basis of this single test, the magnitude and mechanism of corrosion indicate no serious problems for long-term use of TZM alloy in the MSBR vacuum distillation processing scheme. However, the strength properties of the TZM alloy would approach those of unalloyed molybdenum as salt exposure time increased.

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