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IDENTIFICATION OF A NEW LITHIUM OXIDE, Li_3O_2

by Stephan Stecura

Lewis Research Center
Cleveland, Ohio 44135

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16. Abstract

Lithium oxide (Li_3O_2) was prepared by decomposing anhydrous lithium hydroxide at 360° C in 48 hours and between 640° and 730° C in times up to 1.3 hours and pressures of about 10^{-5} torr. Decompositions were done in nickel, molybdenum, niobium, tantalum, and the T-111 tantalum alloy (Ta - 8-wt.% W - 2 wt.% Hf) containers. This oxide probably belongs to the simple orthorhombic system with the following lattice parameters: $a = 108.4 \pm 0.4$ nm (10.84 ± 0.04 Å), $b = 128.4 \pm 0.5$ nm (12.84 ± 0.05 Å), and $c = 103.6 \pm 0.4$ nm (10.36 ± 0.04 Å).

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SUMMARY

Decomposition of anhydrous lithium hydroxide at 360° C in 48 hours and between 640° and 730° C in times up to 1.3 hours and pressures of 10^{-5} torr in nickel, molybdenum, niobium, tantalum, and the T-111 tantalum alloy (Ta - 8 wt. % W - 2 wt. % Hf) containers results in the formation of a lithium oxygen residue whose composition according to chemical analysis corresponds to Li_3O_2. It is believed that the products obtained by decompositions of LiOH probably consisted of a single compound. This compound probably belongs to the simple orthorhombic system with the following lattice parameters: a = 108.4±0.4 nanometers (10.84±0.04 Å), b = 128.4±0.5 nanometers (12.84±0.05 Å), and c = 103.6±0.4 nanometers (10.36±0.04 Å).

In the determination of oxygen concentration in lithium by evaporating excess lithium at temperatures up to 730° C within 1.5 hours at about 10^{-5} torr pressure and then determining the alkalinity of the residue, it is safe to assume that the residue is Li_3O_2 or that it corresponds to this composition and is not Li_2O. Previously, it was assumed that, on the evaporation of excess lithium under these mentioned conditions, Li_2O would form.

INTRODUCTION

Lithium is of interest as a heat-transfer fluid in nuclear systems. This is due to lithium's high heat-transfer coefficient, light weight, high heat capacity, wide liquidus range, and low vapor pressure (refs. 1 and 2). Lithium will remove oxygen from many refractory metals and alloys because dilithium oxide (Li_2O) has a more negative free energy of formation than most of the oxides of the refractory metals (ref. 3). As a result of this oxygen removal, it is of interest to be able to accurately determine the concentration of oxygen in lithium. One method used in determining the oxygen concentration in lithium consists of evaporating excess lithium in vacuum and then determining the alkalinity of the residue (ref. 4). In this method we must know the composition of the recovered lithium-oxygen residue. In the past it was assumed that this residue was

Li_2O. However, it is also possible that an oxide other than Li_2O could be formed on the evaporation of excess lithium in vacuum. If such an oxide is formed, then the value for the oxygen concentration in lithium will be different from the oxygen concentration value calculated on the basis of Li_2O. Indeed, Gahn (ref. 4) reported an X-ray diffraction pattern for lithium-oxygen residue with many lines not found in the pattern for pure Li_2O (refs. 5 to 8). Furthermore, Gahn's X-ray diffraction pattern also does not agree with the X-ray diffraction pattern reported for Li_2O_2 (refs. 9 to 11). This observation suggests that oxides other than Li_2O and Li_2O_2 may form during the evaporation of oxygen-containing lithium.

It is also of interest to know the composition of the recovered residue since this might be helpful in understanding how oxygen is removed by lithium from the refractory metals and alloys. For example, does lithium remove just oxygen, or is oxygen removed as a part of a refractory metal-oxygen-lithium complex? Since the residues from the refractory metal-oxygen-lithium systems are recovered in the same manner as the residues for oxygen determination in lithium, both residues could be identical, if lithium removes oxygen only and does not form a complex with the refractory metal and oxygen.

Because of these questions anhydrous lithium hydroxide (LiOH) was decomposed in various metallic containers at temperatures at which the excess lithium is normally evaporated, and these residues were characterized. The compositional and structural data on these residues, obtained by decomposition of LiOH, were then compared with data on residues obtained by evaporation of excess lithium in vacuum. Therefore, presented herein are the chemical analyses and X-ray diffraction data together with a discussion of the composition and crystal structure modification of the recovered residues from the decompositions of lithium hydroxide. A short discussion is also presented on the relation of these residues to the determination of oxygen concentration in lithium by the method described by Gahn (ref. 4).

MATERIALS, APPARATUS, AND PROCEDURE

The experimental method used in this study consisted of two steps. First, anhydrous lithium hydroxide was decomposed in nickel, niobium, molybdenum, tantalum, and the T-111 tantalum alloy, (Ta - 8 wt. % W - 2 wt. % Hf) containers between 360° and 730° C for up to 48 hours at pressures down to about 1×10^{-5} torr. Second, the residues were then characterized by wet quantitative chemical and X-ray diffraction analyses.

Materials

The anhydrous lithium hydroxide used in this study was at least 99.7 percent pure. It contained the following impurities: carbonate, chloride, sulfate, phosphate, calcium, iron, lead, magnesium, silica, and sodium. The nickel, niobium, molybdenum, and tantalum containers were of very high purity. The total impurities in any of these container metals were less than 0.05 weight percent. Total impurities in the T-111 containers were also less than 0.05 weight percent.

Apparatus and Procedure

The decomposition of lithium hydroxide was accomplished in the apparatus described by Gahn (ref. 4). Temperatures were measured with chromel-alumel thermocouples with an estimated accuracy of $\pm 15^{\circ}$ at 730° C.

About 0.2 gram of lithium hydroxide was added to each of the various containers and decomposed at desired temperatures. The temperature was increased slowly so that the pressure in the apparatus during the heat up did not exceed 10^{-3} torr. The sample was then held at the desired temperature for a specific time with the pressure at final temperature being between 1×10^{-5} and 2×10^{-5} torr. Each container was transferred under vacuum to a glove box. In the glove box, under an argon atmosphere, a portion of each residue was transferred to capillary tubes for X-ray diffraction analyses; the remaining residues were placed in glass vials to be analyzed for lithium and major metal container components. Lithium contents were determined by flame emission photometry with an estimated relative accuracy of 3 percent. Container impurities were determined by optical spectrography. X-ray diffraction patterns were obtained for these residues using a Debye-Scherrer camera with an effective diameter of 11.46 centimeters. Two X-ray radiation sources were used: (1) nickel-filtered copper radiation (Cu K α_1) of 1.54050×10^{-10} meter (1.54050 Å) wave-length, and (2) vanadium-filtered chromium radiation (Cr K α_1) of 2.28962×10^{-10} meter (2.28962 Å) wave-length. All residues were exposed to X-ray radiation between 12 and 16 hours.

RESULTS AND DISCUSSION

Characterization of Lithium-Oxygen Residues

Chemical analyses of the various residues obtained on the decomposition of lithium hydroxide showed that the composition of residues corresponds to the probable chemical formula Li_3O_2 (table I). This composition is also indicated by the weight loss of anhydrous lithium hydroxide on decomposition to lithium oxide (table I). All of these residues were also analyzed for the container metal impurities. Results from the optical spectrographic analyses showed that the residues obtained in nickel, niobium, molybdenum, and tantalum containers contained <0.03 weight percent nickel, <0.03 weight percent niobium, 0.0004 weight percent molybdenum, and <0.4 weight percent tantalum, respectively. Residues from the T-111 alloy container contained only traces of refractory metals.

Closer review of the data presented in table I indicates that decomposition of the lithium hydroxide sample at 360° C in 48 hours at about 5×10^{-5} torr resulted in the formation of a lithium-oxygen residue whose composition corresponded to Li_3O_2. In addition, all decompositions of anhydrous lithium hydroxide between 640° and 730° C in 0.5 to 1.3 hours at 1 to 2×10^{-5} torr resulted in the formation of lithium oxide residues whose compositions corresponded to Li_3O_2. However, it was also found in a separate experiment (results not included in table I) that, when lithium hydroxide is decomposed at 860° C in 1 hour at 2×10^{-5} torr, the lithium to oxygen ratio is different from that found in Li_3O_2. The percent of lithium found in the residue obtained at 860° C was about 44 weight percent. The weight percent of lithium in Li_2O_2, Li_3O_2, and Li_2O are 31.6, 39.4, and 46.5, respectively. These results suggest that the lithium to oxygen ratio might be changing with the temperature, time, and/or pressure.

The overall process for the decomposition of anhydrous lithium hydroxide to trilithium dioxide can be represented by the following equation:

$$6LiOH \rightarrow 2Li_3O_2 + 2H_2O + H_2$$

However, neither the chemical analyses nor the weight-loss measurements established that the recovered oxides were single compounds. It is possible that two or more stoichiometric lithium oxides could be present in such quantities that the results from the chemical analyses and weight-loss measurements would tend to confirm the presence of only one compound, namely, Li_3O_2. To establish that probably one compound was present, X-ray diffraction analyses were performed on these residues. If the X-ray diffraction patterns obtained for various residues can be indexed on the basis of one crystallographic modification, this would be an additional indication that the residues were composed of a single compound.

X-ray diffraction patterns obtained from the various residues were computer indexed by the Hesse-Lipson method (ref. 12) and also Ito's (ref. 12) method. These patterns were indexed on the basis of a simple orthorhombic system. A representative pattern for these residues is presented in table II.

The lattice parameters were calculated for all the d-spacing values reported in table II, and there was a very high degree of agreement among all the lattice parameter values. The final lattice parameter values were determined by plotting a, b, and c lattice parameters against $\cos^2\theta$ and extrapolating to 90°. These values are a = 108.4±0.4 nanometers (10.84±0.04 Å), b = 128.4±0.5 nanometers (12.84±0.05 Å), and c = 103.6±0.4 nanometers (10.36±0.04 Å).

It should be noted that the pattern reported in table II is not necessarily complete and that the intensities do not necessarily represent the true intensities of the different d-spacings. This could be due to the fact that (1) the residue samples were not exposed to X-ray radiation for a sufficient time to pick up the very weak lines and (2) the residues were not pulverized to particles small enough (less than 44 μm in diameter) to obtain random distribution of particles. Since the residues are hygroscopic, the pulverization was done inside the argon filled glove box to obtain suitable powder material only. However, it is believed that the particles were in 100 micrometer diameter range.

Additional evidence that the recovered residues probably were single compounds comes from the comparison of X-ray diffraction patterns obtained in this study with the X-ray diffraction patterns reported for Li_2O and Li_2O_2 (refs. 5, 9, and 11). This comparison is shown in table II. From these data it can be concluded that the five patterns reported in this table are different. The lines in the X-ray diffraction pattern obtained in this study cannot be accounted for by any combination of X-ray lines from the remaining four patterns.

True, all the diffraction lines found in Li_2O are also found in the pattern obtained in this study. However, many other strong lines cannot be accounted by the lines found in Li_2O_2 (refs. 9 and 11). Thus, three possible explanations can be offered. One, the patterns reported for Li_2O_2 are incomplete and incorrect. Two, the residues obtained in this study contain a mixture of Li_2O, Li_2O_2, and some other lithium oxide or oxides. Three, the residues obtained in this study are single compounds, and the X-ray diffraction pattern reported in table II is representative of Li_3O_2.

The first explanation could possibly be true. However, it requires the assumption that the X-ray diffraction patterns reported in references 9 and 11 are either incomplete or incorrect. Föppl (ref. 9) has presented an extensive study on Li_2O_2. From this study he has concluded that Li_2O_2 has a hexagonal crystalline modification and belongs to C_{3h} space group. His work is also supported by Rode and coworkers (ref. 11) who found that Li_2O_2 can exist in two crystalline modifications: (1) α-Li_2O_2, which has a hexagonal crystalline modification and is stable up to 225° C, and (2) β-Li_2O_2, which is stable up to 280° to 315° C depending on the rate of temperature increase. They reported an incomplete X-ray diffraction pattern for β-Li_2O_2 and did not index this pattern.

Indeed, there is no evidence presently available that would tend to contradict Föppl's careful work. Then it is assumed that Li_2O_2 at least up to 225° C has a hexagonal crystalline modification. In addition, the work presented by Rode and coworkers cannot be rejected since no contrary proof is now available. As the result of these two studies, it is assumed that the X-ray diffraction patterns reported for Li_2O_2 by Föppl (ref. 9) and Rode and coworkers (ref. 11) are correct.

The second explanation, namely, that three or more lithium oxides were formed, from the thermodynamic view point is very unlikely. It is highly improbable that three or more oxides, having different free energies of formation, could exist at the temperatures, times, and pressure at which lithium hydroxide was decomposed (table I). It is believed that under these various conditions (table I), changes in lithium to oxygen ratios would have been observed if the residues obtained in this study were made of two or more lithium oxides. However, no such changes were observed. Therefore, the second explanation is considered very unlikely.

In view of these discussions, it is concluded that the third explanation, namely, that Li_3O_2 was formed, is most reasonable. Thus, it is assumed that the residues obtained on the decomposition of anhydrous lithium hydroxide at temperatures up to 730° C consisted of a single compound, namely, Li_3O_2.

Determination of Oxygen in Lithium

In determining the oxygen concentration in lithium by evaporation of excess lithium and determination of alkalinity of residue (ref. 4), it is important to know the composition of the recovered lithium-oxygen residue since the oxygen concentration value depends on the composition of the recovered oxide. However, a literature review shows that many lithium oxides have been prepared: Li_2O (refs. 5 to 8), Li_2O_2 (refs. 9 to 11), and LiO_2 (ref. 13). Therefore, it must be known which of these oxides are formed or recovered in the determination of oxygen concentration in lithium.

Normally, excess lithium is evaporated at temperatures up to 740° C in times up to 2 hours at about 2×10^{-5}-torr pressure. These are the same conditions under which anhydrous lithium hydroxide was decomposed in this study. Therefore, on the basis of the data presented in this study and in reference 4, it is concluded that the residue recovered in the determination of oxygen concentration in lithium has a composition corresponding to Li_3O_2 and not Li_2O as was assumed in reference 4. The data obtained in this study further suggest that, above 730° C and after long heating, the recovered residues probably approach Li_2O. Because of this uncertainty, an X-ray diffraction pattern should be obtained for the sample whose alkalinity is being determined. This will enable one to determine the composition of the recovered residue and obtain a more accurate value for the oxygen concentration in lithium.

CONCLUSIONS

A study has been conducted on the characterization of lithium-oxygen residues obtained on the decomposition of anhydrous lithium hydroxide in nickel, niobium, molybdenum, tantalum, and T-111 tantalum alloy (Ta - 8 wt.% W - 2 wt.% Hf) containers. Decompositions were accomplished at temperatures up to 730° C in times up to 48 hours and pressure of 1×10^{-5} to 2×10^{-5} torr. The major results of this study were as follows:

1. Lithium oxide formed on decomposition of lithium hydroxide according to chemical analyses and weight-loss measurements has a chemical formula Li_3O_2. This oxide probably belongs to the simple orthorhombic system with the following lattice parameter values: a = 108.4±0.4 nanometers (10.84±0.04 Å), b = 128.4±0.5 nanometers (12.84±0.05 Å), and c = 103.6±0.4 nanometers (10.36±0.04 Å).

2. In the determination of oxygen concentration in lithium by evaporating excess lithium at temperatures up to 730° C in times up to 1.5 hours and at pressure of 1×10^{-5} to 2×10^{-5} torr, it is safe to assume that the recovered lithium oxide residue has a composition corresponding to Li_3O_2 and not to Li_2O.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, June 20, 1973,
501-21.

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TABLE I. - ANALYSES OF LITHIUM-OXYGEN RESIDUES

Container metal	Decomposition temperature, °C	Decomposition time, hr	Weight loss on decomposition, %	Lithium content, wt. %	Oxygen content[a], wt. %
Ni	640	1.2	----	39.0	[a]61.0
Ni	710	.5	28.0	41.6	58.4
Mo	700	0.8	----	40.0	60.0
Mo	700	1.3	29.0	41.0	59.0
Mo	730	.7	28.3	40.0	60.0
Nb	725	1.0	27.1	40.7	59.3
Nb	730	.8	----	40.8	59.2
Ta	690	1.3	28.3	40.7	59.3
Ta	700	.7	----	40.8	59.2
T-111	360	48.0	26.6	40.5	59.5
T-111	730	1.0	27.2	40.3	59.7
Average			27.8	40.5	59.5
Theoretical for Li_3O_2			26.5	39.4	60.6

[a] Percent of oxygen was determined by difference (i.e., 100 minus percent of lithium determined by flame photometry).

TABLE II. - COMPARISON OF X-RAY DIFFRACTION PATTERNS FOR LITHIUM OXIDES OF DIFFERENT STOICHIOMETRIES

Li_3O_2		Li_2O	Li_2O_2	α-Li_2O_2	β-Li_2O_2	Li_2O_2		Li_2O	Li_2O_2	α-Li_2O_2	β-Li_2O_2
This study		ref. 5	ref. 9	ref. 11		This study		ref. 5	ref. 9	ref. 11	
d-spacing, m	Intensity	d-spacing, m				d-spacing, m	Intensity	d-spacing, m			
4.79×10^{-10}	1	----------	----------	----------	----------	1.488×10^{-10}	5	----------	----------	----------	1.49×10^{-10}
4.30	6	----------	----------	----------	4.35×10^{-10}	1.471	1	----------	----------	----------	----------
4.14	1	----------	----------	----------	----------	1.447	1	----------	1.4515×10^{-10}	1.45×10^{-10}	----------
3.850	<1	----------	3.814×10^{-10}	3.84×10^{-10}	----------	1.409	1	----------	----------	----------	----------
3.786	1	----------	----------	----------	----------	1.389	6	1.3906×10^{-10}	----------	----------	----------
3.710	2	----------	----------	----------	----------	1.374	1	----------	----------	----------	----------
3.610	5	----------	----------	----------	----------	1.351	<1	----------	1.3599	----------	----------
3.460	2	----------	----------	----------	----------	1.328	<1	----------	1.3346	1.34	----------
3.411	2	----------	----------	----------	----------	1.305	<1	----------	----------	----------	----------
3.355	2	----------	----------	----------	----------	1.295	3	----------	1.2829	1.29	----------
3.285	1	----------	----------	----------	----------	1.279	1	----------	1.2769	----------	----------
3.037	2	----------	----------	----------	3.08	1.253	<1	----------	----------	----------	----------
2.964	4	----------	----------	----------	----------	1.219	<1	----------	1.2161	1.22	----------
2.947	2	----------	----------	----------	----------	1.204	1	----------	1.2008	----------	----------
2.930	3	----------	----------	----------	----------	1.189	<1	----------	----------	----------	----------
2.914	2	----------	----------	----------	----------	1.182	1	----------	----------	----------	----------
2.819	2	----------	----------	----------	----------	1.167	1	----------	----------	----------	----------
2.761	1	----------	----------	----------	2.75	1.152	4	1.1531	----------	----------	----------
2.731	6	----------	2.7200	2.74	----------	1.140	2	----------	----------	----------	----------
2.709	1	----------	----------	----------	----------	1.122	1	----------	----------	----------	----------
2.665	1	2.664×10^{-10}	----------	----------	----------	1.105	2	----------	1.1088	----------	----------
2.652	10	----------	----------	----------	----------	1.096	1	----------	----------	----------	----------
2.567	3	----------	2.5615	2.59	----------	1.066	<1	----------	----------	----------	----------
2.500	6	----------	----------	----------	2.52	1.057	3	1.0580	----------	----------	----------
2.468	2	----------	----------	----------	----------	1.042	1	----------	----------	----------	----------
2.425	4	----------	----------	----------	----------	1.031	1	----------	1.0284	1.02	----------
2.362	1	----------	----------	----------	----------	----------	--	----------	1.0175	1.00	----------
2.338	1	----------	----------	----------	----------	.9965	1	----------	.9911	----------	----------
2.300	6	2.306	----------	----------	----------	.9815	<1	----------	----------	----------	----------
2.254	1	----------	----------	----------	----------	.9591	2	----------	.95389	----------	----------
2.300	1	----------	----------	----------	----------	.9417	4	.9413	----------	----------	----------
2.198	1	----------	2.2201	2.23	----------	.9170	1	----------	----------	----------	----------
2.175	2	----------	----------	----------	----------	.9014	<1	----------	.90628	----------	----------
2.156	<1	----------	----------	----------	----------	.8952	<1	----------	----------	----------	----------
2.115	6	----------	----------	----------	----------	.8877	4	.8875	----------	----------	----------
2.107	2	----------	----------	----------	----------	.8631	<1	----------	.85338	----------	----------
2.075	1	----------	----------	----------	----------	.8446	<1	----------	----------	----------	----------
2.046	1	----------	----------	----------	----------	.8429	1	----------	----------	----------	----------
1.991	1	----------	----------	----------	----------	.8258	1	----------	.81968	----------	----------
1.960	1	----------	----------	----------	----------	.8154	5	.8151	.81661	----------	----------
1.935	<1	----------	1.9163	1.90	1.94	.7972	3	----------	.81081	----------	----------
1.864	<1	----------	1.8574	1.87	1.86	.7938	2	----------	----------	----------	----------
1.844	2	----------	----------	----------	----------	.7797	5	.7794	.78547	----------	----------
1.827	1	----------	----------	----------	----------						
1.804	2	----------	----------	----------	----------						
1.769	4	----------	----------	----------	1.76						
1.744	1	----------	----------	----------	----------						
1.641	7	----------	----------	----------	1.66						
1.628	8	1.6307	----------	----------	----------						
1.589	1	----------	1.5715	1.58	----------						
1.551	4	----------	----------	----------	----------						
1.537	1	----------	----------	----------	----------						
1.513	1	----------	----------	----------	----------						

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