JOM, Vol. 75, No. 4, 2023
https://doi.org/10.1007/s11837-022-05537-0

Effect of Temperature on the Solubility of Metallic Magnesium in Molten $MgCl_2$-KCl-NaCl

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Solubility experiments for magnesium metal in molten $MgCl_2$-KCl-NaCl were run at 500°C, 600°C, and 650°C. This study is relevant for understanding how to control corrosivity of the salt when used for high-temperature heat transfer and storage in systems such as concentrating solar power plants and advanced nuclear reactors. Metal dissolution is a viable option for reducing the redox potential of the salt, which should reduce the rate of corrosion of contacting structural metal alloys. Concentration of metallic magnesium in the salt mixture was determined via measurement of the volume of H_2 generated during dissolution of salt samples in water. Open circuit potential between a W working electrode and Ag|AgCl reference electrode was used to determine when equilibrium had been achieved. The dissolved Mg metal concentration was found to vary between 0.32 wt.% and 0.58 wt.% by hydrogen collection, which was confirmed by measurement of Mg rod mass loss.

INTRODUCTION

Currently, there are numerous proposed applications for molten chloride salt mixtures in the scope of energy capture, storage, and conversion to electricity. This includes concentrating solar power (CSP) plants and molten salt-fueled nuclear reactors (MSRs).[1–3] In addition, TerraPower has recently announced that their Natrium demonstration sodium-cooled reactor will feature energy storage with molten salt.[4] With their low melting point, good thermal stability, and high heat capacity, molten salts have become a promising option for this low-cost heat transfer material.[5,6]

However, metal corrosion is often cited as a problem when using molten chloride salts.[7,8] To reduce the corrosivity of the salt, the redox potential must be lowered. The redox potential can be quantified as the equilibrium potential (E_{eq}) relative to a reference electrode, and calculated using the Nernst equation given in Eq. 1.[9] In this equation, $E^{o}_{M^{n+}/M}$ is the standard reduction potential for reduction of M^{n+} to M versus a select reference potential. R is the ideal gas constant, T is the absolute temperature, n is the number of electrons involved with reduction/oxidation per atom of M, F is Faraday's constant, $a_{M^{n+}}$ is activity of M^{+} ions in the salt, and a_M is activity of M metal.

$$E_{eq} = E^{o}_{M^{n+}/M} + \frac{RT}{nF}\ln\left(\frac{a_{M^{n+}}}{a_M}\right) \tag{1}$$

One proven approach to lowering the redox potential is to dissolve a soluble metal into the salt.[9] Metal solubility has been found to occur in many different molten chloride salt mixtures that contain the chloride version of the metal being dissolved.[10–12]

This study was motivated by the need to better understand how to control the redox potential of the salt containing $MgCl_2$ by dissolving metallic Mg. Evidence has been shown that dissolved Mg will react with OH^- in $MgCl_2$-KCl-NaCl to form hydrogen and magnesium oxide in the following reaction:[13]

(Received May 10, 2022; accepted September 19, 2022;

$$Mg + Mg(OH)_2 = 2MgO + H_2$$

A key question is what concentration of Mg in the zero-oxidation state will stay in solution with this salt mixture at equilibrium. This will allow us to understand the capacity of Mg to buffer the redox potential of select salt mixtures. It is expected that Mg solubility varies based on salt mixture composition, so it was deemed important to select a salt mixture of current interest for the above-mentioned applications. The specific salt mixture chosen was $MgCl_2$-KCl-NaCl, which has a low melting temperature and was used in the study reported by Choi et al.[13]

In a study by Zhang et al. on molten salts in the reduction of TiO_2 with Mg, values currently found in literature for the solubility of Mg in $MgCl_2$ are summarized.[14] When performed with a Mg-Cu alloy at 750–900°C, solubility ranged from 0.016 wt.% to 0.065 wt.%.[15] When performed with a Mg-Al alloy at 800–1000°C, solubility ranged from 0.04 wt.% to 0.18 wt.%.[16] When carried out with a Mg-Pb alloy at 720–900°C, solubility ranged from 0.108 wt.% to 0.327 wt.%.[17] Finally, when carried out with Mg at 800–1000°C, solubility ranged from 0.11 wt.% to 0.332 wt.%.[17]

Our group previously published a preliminary analysis of Mg solubility in molten $MgCl_2$-KCl-NaCl, in which it was reported to be in the range of 0.03–0.039 wt.% from 500°C to 650°C.[18] However, the salt in this preliminary study was not carefully purified, and there is uncertainty as to whether a long enough time was given to allow the Mg metal and molten salt to equilibrate.

In our continued investigation of this topic, a ternary salt mixture of $MgCl_2$-KCl-NaCl was carefully purified and characterized for OH^- content prior to performing long-duration Mg powder or rod dissolution experiments. Purification was accomplished via sparging anhydrous HCl into the salt. Electrochemical potential measurements were made relative to a reference electrode to determine the equilibrium time needed for each experiment. The concentration of dissolved Mg was determined via measurement of evolved H_2 after dissolving salt samples in water, and confirmed with the mass loss of a Mg metal rod. It is, thus, believed that the results obtained in this stage of the investigation are more accurate than the preliminary published results.

METHODS

Materials

Mg powder (99%) was obtained from Sigma Aldrich, and dehydrated carnallite was obtained from Israel Chemical for consistency of results with a consortium of research teams under a U.S. Department of Energy-funded research program.[19] The supplier-reported composition of carnallite is 44–50 wt.% $MgCl_2$, 38–43 wt.% KCl, 2.5–10 wt.% NaCl, 1.5–2.5 wt.% MgO, and 3.5–4.5 wt.% H_2O. The MgKNaCl salt and Mg powder were stored in an inert argon atmosphere glovebox (PureLab HE; Inert Technologies) to prevent unwanted reactions with O_2 and H_2O. The moisture content and oxygen level in the glovebox were usually kept below 15 ppm.

HCl Purification Process

Before testing, the MgKNaCl salt underwent a chlorination purification process to lower the hydroxide concentration and minimize moisture. An amount of 200 g of MgKNaCl salt was placed in an alumina crucible that was then set on a quartz stand inside a quartz reactor. The top of the quartz reactor was sealed with quartz wool and a clamped-on Teflon™ lid. A Grainier type K Inconel 600 thermocouple was inserted through pre-cut holes in the lid to monitor the temperature and a lance allowed gas flow through the salt. Flow was controlled with a set of factory-calibrated mass flow controllers (GM50A; MKS). The lance was raised out of the salt to start the process. The quartz reactor was placed in a modified Thermolyne 1300 box furnace in a fume hood with more quartz wool wrapped around the top to prevent the Teflon™ lid from melting. The furnace was modified with a 3.5-cm-diameter hole in the top to allow the quartz reactor to be inserted from the top.

The lance was then attached to tubing that pulled a vacuum in the quartz reactor while the furnace was heated to 200°C at a rate of 5°C/min and held at 200°C for 2 h to remove as much water as possible. Next, the vacuum was released in the furnace, and 100 scc of ultra-high purity Ar (AirGas 99.999%) flowed through the salt as the furnace was heated to 500°C. Once the MgKNaCl salt reached 500°C, 92 sccm of Ar and 8 sccm of anhydrous HCl gas (AirGas 99.5%) was flowed directly to a pH 10 solution in which an auto-titrator (Titroline 7000; SI Analytics) recorded the volume of the 0.5 M NaOH titrant needed to keep the pH of the solution at 10. Once this value was constant, the volumetric flow of the titrant was recorded as a baseline slope measurement.

The lance in the quartz reactor was then lowered into the salt, and the 92 sccm of Ar and 8 sccm of HCl were switched to flow through the lance into the salt. The effluent from the quartz reactor was directed into the titrator cell held at pH 10. The Ar and HCl flowed through the MgKNaCl salt until the titrator recorded the same volumetric flowrate of the titrant as measured during the reactor bypass. This indicated that no further reaction of HCl with the salt was occurring. The MgKNaCl salt was then cooled and stored in a sealed container in a glovebox.

Samples of purified MgKNaCl were analyzed via acid titration using a Titroline 7000 auto-titrator. Solution pH was controlled to a value of 4.0. After dissolution of a salt sample with mass ranging from 0.7 g to 1.0 g, 0.1 M HCl was dosed to bring the pH back to 4.0.

Open Circuit Potential

To determine the redox potential of the MgKNaCl salt-Mg mixture, 25 g of the chlorinated salt mixture and between 0.2 g and 0.6 g of Mg powder were placed in the alumina crucible in the glovebox and transferred to a furnace in the glovebox. The furnace was set to heat to 500°C, 600°C, or 650°C at a rate of 2°C/min. An Ag|AgCl reference electrode, created by placing a 1.0-mm-diameter Ag wire (Alfa Aesar 99.99%) and 0.5 g AgCl (Alfa Aesar 99.997%) in a ceramic mullite tube with a silicone plug at the top, was placed in the crucible. Along with it was placed a 1.5-mm-diameter W rod working electrode encased in an alumina ceramic mullet tube with openings at both ends. The electrodes were aligned so that they were inserted through the pre-cut holes in a metal lid on the crucible to prevent evaporation of the mixture during heating. A schematic of the cell can be seen in Fig. 1.

Once the salts reached their respective temperatures, the electrodes were connected to a Gamry Interface 1000 potentiostat or Metrohm Autolab PGSTAT302N potentiostat to measure the open circuit potential OCP) for 16–19 h until the mixture stabilized and reached a constant potential.

Solubility of Mg

After allowing the OCP to stabilize, the salt was quickly taken from the furnace using needle-nose pliers and quenched by pouring the contents of the alumina crucible onto a room-temperature stainless steel pan in the glovebox. The salt then solidified.

The frozen salt was ground into a powder using an IKA A11 Basic Analytical Mill and stored in a sealed container in the glovebox. A 3-g sample of the powder was then obtained and transferred to a fume hood. When not in use, the powder was kept in the sealed container in the glovebox.

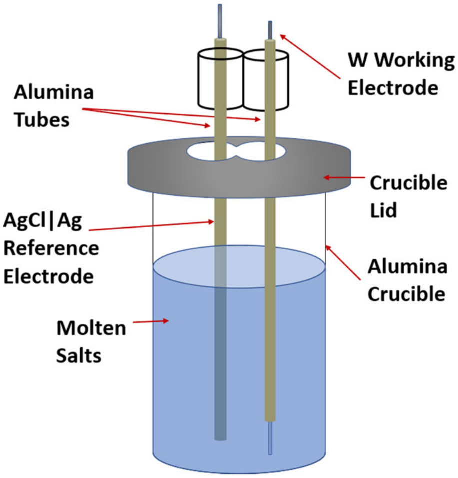

Fig. 1. A schematic of the cell used for electrochemistry with the MgKNaCl salt and Mg. This cell is heated in a furnace inside of an Ar-atmosphere glove box.

In the fume hood, a 1000-mL beaker was filled with 700 mL of deionized water, and a 100-mL beaker was submerged inside the larger beaker to seal it from the air. A magnetic stirrer bead was placed inside of the 100-mL beaker, and the setup was placed on a magnetic stirrer. A burette with an attached glass funnel, supported by a ring stand, was placed over the 100-mL beaker with its glass funnel covering the mouth of the beaker. A photograph of the beaker–burette setup is shown in Fig. 2.

The burette was carefully slid off the lip of the 100-mL beaker without breaking its seal in the water, and the 3-g sample of the MgKNaCl-Mg salt mixture was poured into it. The burette was quickly moved back over the mouth of the beaker to capture the gas released as the salt dissolved, and the magnetic stirrer was turned on to aid in the full dissolution of the salt mixture. The gas collected in the burette was assumed to be H_2 from either or both of the following reactions in which 1 mol of H_2 forms for every 1 mol of dissolved Mg^0:

$$Mg^0 + H_2O = MgO + H_2$$

$$Mg^0 + 2H_2O = Mg(OH)_2 + H_2$$

Using the water volume and the solubility of H_2 (7×10^{-6} M), the amount of H_2 that was absorbed in the water was estimated and added to the measured amount of H_2. The pressure in the burette was corrected by subtracting the saturated water vapor pressure.[11] The operating temperature for the experiment was room temperature.

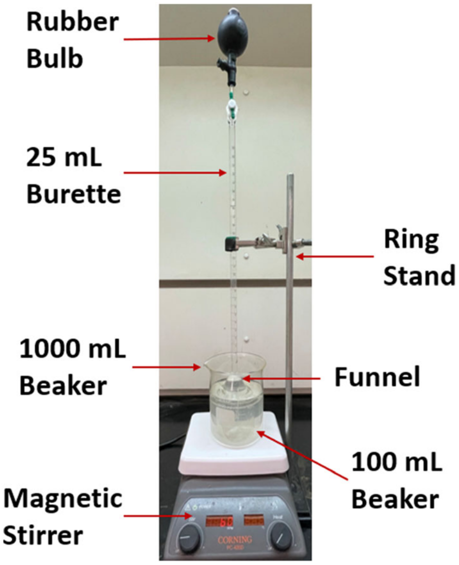

Fig. 2. The H_2 solubility setup for measuring the volume of H_2 produced by the reaction of the MgKNaCl and Mg mixture and water. This measurement was carried out at room temperature.

Dissolution of Mg Rod in MgKNaCl Salt

To verify the results from the H_2 solubility test, a second experiment using a 4.8-mm-diameter Mg rod (Sigma-Aldrich, 99.9% purity) instead of Mg powder was performed to test the solubility of Mg in the MgKNaCl salt. The initial weight of the Mg rod was measured and recorded. In the glovebox, 25 g of MgKNaCl salt were added to an alumina crucible and placed in a furnace. A metal lid with pre-cut holes was placed on the crucible to prevent the evaporation of the MgKNaCl salt. The same Ag | AgCl reference electrode and W working electrode that were used in section "Open Circuit Potential" were placed in the crucible along with the 4.8-mm-diameter Mg rod. The furnace was heated to 500°C at a rate of 2°C/min. Once the salts reached 500°C, the reference electrode and the working electrode were connected to the Metrohm Autolab PGSTAT302N potentiostat, and an OCP test was conducted. Just as with the OCP carried out with the Mg powder, the test was run until the redox potential stabilized (135 h), signaling the full dissolution of the Mg in the MgKNaCl salt. The Mg rod was then removed from the MgKNaCl salt to cool, and then it was lightly sanded to remove any MgKNaCl salt that had solidified on it. The Mg rod was then weighed and its final mass recorded.

RESULTS AND DISCUSSION

HCl Purification Process

As shown in Table I, the chlorination process resulted in a decrease in the concentration of OH^- ions in the salt of 58.8–72.0%, as measured via acid titration of salt samples taken before and after completion of each chlorination run. The final concentration of OH^- in the salt was determined to range from 1.39×10^{-4} mol/g to 4.12×10^{-4} mol/g.

These results confirm that the amount of OH^- in the salt decreased due to HCl sparging. This differs from the work of Choi et al., as they did not include purification of the salt in their experiment.[13] This could result in different solubility measurement results. It is believed that the highest quality data are obtained by achieving the highest possible salt purity. There is concern about the reaction of Mg metal with OH^- to form hydroxides or hydroxychlorides with unknown equilibrium phase behavior. Such compounds may also produce H_2 when dissolved in water, which would introduce error into the Mg metal solubility measurements. Or, if enough of the Mg reacted with the OH^- ions, the Mg would no longer exist in excess in the reaction, resulting in the MgKNaCl salt not being fully saturated with Mg.

Redox Potential Measurement During Mg Dissolution Tests

The Mg and MgKNaCl salt mixtures were allowed to equilibrate for up to 19 h while OCP was continuously measured. The measured potentials are plotted in Fig. 3. The redox potential stabilized at different values of − 1.6 V, − 0.12 V, and − 0.37 V at 500°C, 600°C, and 650°C, respectively. The potentials were measured relative to a Ag/AgCl reference electrode. The Nernst equation (Eq. 1) can be written specifically to using a Mg rod as the working electrode to relate the OCP (E) to the standard reduction potential and the ratio of activities of $MgCl_2$ and Mg. This is shown in Eq. 2:

$$E = E^{o}_{\mathrm{MgCl_2/Mg}} + \frac{RT}{2F}\ln\left(\frac{a_{\mathrm{MgCl_2}}}{a_{\mathrm{Mg}}}\right) \tag{2}$$

Activity of $MgCl_2$ should be invariant in these experiments, and activity of Mg should be equal to unity when the salt is saturated with Mg. Thus, stabilization of the OCP is a key requirement to indicate that Mg saturation of the salt has occurred. Figure 3 shows that the experiments were run sufficiently long in order to achieve this equilibrium state.

Mg Solubility and Temperature

Three 3-g samples of salt from each of the three Mg dissolution experiments were weighed in the glovebox and placed in an air-tight vial to transfer to the fume hood. The samples were then dissolved in water to measure metallic Mg concentration indirectly via generation of H_2. It was assumed that only dissolved Mg reacted with water to form H_2, and that undissolved Mg powder did not react with water to form H_2. A control test where Mg powder without MgKNaCl salt was dissolved in water was run to verify this. The control showed that no H_2 was formed from the Mg powder dissolved in water, and confirmed that the Mg powder that was not dissolved in the salt did not produce H_2 when dissolved in water. Additionally, a second control

Table I. Results of the OH^- concentrations of the MgKNaCl salt after HCl purification process

Chlorination run	Average initial OH^- concentration (Mol OH^-/g MgKNaCl)	Average final OH^- concentration (Mol OH^-/g MgKNaCl)	Average percent reduction (%)
1	10.0×10^{-4}	4.12×10^{-4}	58.8
2	0 8.77×10^{-4}	2.46×10^{-4}	72.0
3	0 4.94×10^{-4}	1.39×10^{-4}	71.9

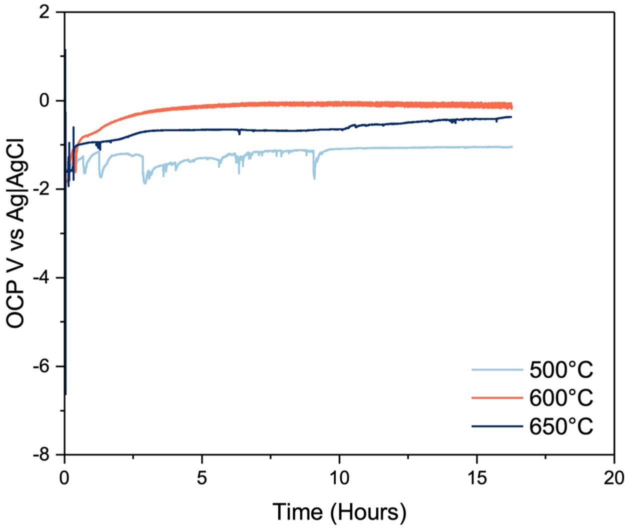

Fig. 3. OCP measurements of MgKNaCl salt and Mg at 500°C, 600°C, and 650°C, showing the complete dissolution of Mg in the MgKNaCl salt. Potential is given relative to a AgCl/Ag reference electrode.

test was run where MgKNaCl salt was mixed with excess Mg powder and heated at 500°C until molten. The product was added to water, and the excess Mg particles that did not dissolve in the MgKNaCl salt congregated to the bottom. From this, it was verified that the excess Mg particles did not dissolve in water.

The results of the Mg metal solubility measurements are shown in Table II. The solubility of the Mg was calculated with the ideal gas equation using the volume of H_2 produced from the reaction of the MgKNaCl-Mg sample in water. The temperature of the water was 298 K and the atmospheric pressure in the laboratory (Salt Lake City, UT, USA) was 85.7 kPa. The vapor pressure of the water at 298 K was 3.17 kPa, and this value was subtracted from the atmospheric pressure to result in a partial pressure in the burette of 82.53 kPa. By using the solubility of water at 298 K (1.41×10^{-5} g/L), the amount of moles of H_2 dissolved in the water was calculated. The total amount of moles of H_2 dissolved in water and from the burette were treated as equal to the total amount of moles of Mg. The moles of Mg that were dissolved in the MgKNaCl salt sample were calculated with Eq. 1.[11]

$$n_{\mathrm{Mg}} = n_{\mathrm{H_2}} = n_{\mathrm{H_2 dissolved}} + \frac{P_{\mathrm{H_2}} V_{\mathrm{H_2}}}{RT} \quad (3)$$

The standard deviation for the solubility of Mg at 650°C is relatively high when compared to that at 500°C and 600°C. This could be due to an outlier in the testing, or there could be immiscible liquid Mg present that causes the salt phase to be heterogenous. The melting temperature of Mg is 650°C.

The results from this paper for the solubility of Mg in the MgKNaCl salt are significantly higher than those found in Choi et al.'s work, by a factor of over ten. One explanation could be that the salt in this experiment was pre-treated with the chlorination process to remove excess OH^-. OH^- ions in the Choi et al. experiment could have reacted with Mg to form $Mg(OH)_2$ that may not dissolve in the salt, thus resulting in the Mg not being present in excess to fully saturate the MgKNaCl salt.

Another viable explanation for the difference in the solubilities could be that this experiment held the MgKNaCl salt at temperature for 16–18 h whereas the MgKNaCl salt was held for 5 h in the Choi et al. experiment.[13] This extra time could have allowed for the Mg to fully dissolve in the MgKNaCl salt, resulting in a higher final weight percent Mg.

Furthermore, the wt.% of $MgCl_2$ in the MgKNaCl salt was different between the salt used in the Choi et al. experiment and the salt used in this one, as shown in Table III. The difference between the average wt.% of $MgCl_2$ in the two salts was statistically significant, with the mean of the $MgCl_2$ in Choi et al.'s work being 75.0 wt.% and the mean for this experiment being 59.4 wt.% ($p = 0.007$). These numbers were based solely on the analysis of Na, K, and Mg, excluding water, MgO, and any other impurities that may be present. When calculating the wt.% of each chloride salt, it was assumed that each was present as only NaCl, KCl, and $MgCl_2$ respectively. Krumpelt et al. reports that, when Mg metal is added to molten $MgCl_2$, the activity

Table II. Results of the solubility of Mg powder in the MgKNaCl salt (Mg-Cl_2-KCl-NaCl)

Temperature (°C)	Mass of MgKNaCl salt (g)	Mass of Mg powder (g)	Mass of sample dissolved (g)	Volume of H_2 produced (mL)	Mg solubility (wt.%)	Average Mg solubility (wt.%)	SD
500	25	0.20	3.06	12.3	0.33	0.32	0.11
			3.03	15.9	0.43		
			3.10	7.7	0.20		
600	25	0.20	3.29	13.8	0.34	0.32	0.02
			3.12	12.0	0.31		
			3.23	11.9	0.30		
650	25	0.56	3.11	22.4	0.59	0.58	0.22
			2.90	12.3	0.35		
			2.58	25.0	0.79		

Table III. Measured wt.% of MgKNaCl salt from Choi et al. versus this work

	MgKNaCl salt from Choi et al. (wt.%)[13]					MgKNaCl salt from this work (wt.%)				
	1	2	3	Average	SD	1	2	3	Average	SD
NaCl	1.2	1.5	4.2	2.3	1.7	8.9	2.1	1.3	4.1	4.2
$MgCl_2$	80.0	74.9	70.0	75.0	5.0	57.9	61.6	58.8	59.4	1.9
KCl	18.8	23.7	25.8	22.8	3.6	33.2	36.3	39.9	36.5	3.3

Table IV. Results of the solubility of Mg rod in the MgKNaCl salt (Mg-Cl_2-KCl-NaCl) at 500°C

Mass of MgKNaCl salt (g)	Initial mass of Mg rod (g)	Final mass of Mg rod (g)	Change in mass of Mg rod (g)	Calculated Mg solubility (wt.%)	Average (wt.%)	SD
25.44	11.23	11.26	0.17	0.66	0.46	0.20
24.40	11.06	11.00	0.06	0.26		

coefficient increases as the concentration of $MgCl_2$ decreases.[15] When there is a lot of $MgCl_2$ present, there is less opportunity for the new Mg that is being added to dissolve in the solution. This could also help to account for the disparity between the two sets of results.

Dissolution of Mg Rod in MgKNaCl Salt

In order to provide a secondary approach to measuring Mg solubility, experiments were run with a Mg rod immersed in MgKNaCl salt that could be weighed before and after the dissolution experiment. The objective was to directly measure the mass of Mg metal dissolved in the salt. This could not be done with the results of the tests reported above, since they involved mixing Mg powder with salt that could not be recovered and weighed after the test. The solubility of the Mg rod in the MgKNaCl salt was calculated by dividing the mass difference from the initial and final mass of the Mg rod with the mass of salt in the crucible. The results are shown in Table IV.

The wt.% Mg found from the Mg rod experiment has a very large variability, as can be seen in Table IV (see Table II, where the Mg solubility is reported as 0.32 wt.% at 500°C). The weight loss-based measurements reported in Table IV enclose this value, which is considered strong confirmation that the Mg solubility measurements reported in this paper are indeed accurate. For the high result given in Table IV, reaction with OH^- could have occurred and increased the weight loss of Mg beyond what can be explained by solubility. There are more data to support the assertion that the H_2 measurement method is relatively accurate and repeatable. It uses an excess amount of Mg metal, and the formation of other soluble Mg species does not affect the Mg^0 solubility measurement. Figure 4 shows the appearance of the Mg rod after each of the rod-based solubility tests in MgKNaCl salt at 500°C.

Fig. 4. (a) First Mg rod after OCP test at 500°C and (b) second Mg rod after OCP test at 500°C. The diameters of the Mg rods were 4.8 mm.

CONCLUSION

This study has yielded data on the solubility of metallic Mg in a $MgCl_2$-NaCl-KCl molten salt mixture over a range of 500–650°C. Tests were run up to 18.4 h until OCP measurements in the salt had stabilized to establish that equilibrium had been reached. Based on H_2 collection during salt sample dissolution, solubility values measured were 0.32 wt.%, 0.32 wt.%, and 0.58 wt.% Mg at 500°C, 600°C, and 650°C, respectively. Additional tests in which the mass loss of Mg from metal rods was measured at 500°C yielded solubility values of 0.26 wt.% and 0.66 wt.%, which brackets the H_2 volume-based measurement of Mg. With the need for effective and efficient renewable energy sources continuing to be a challenge, these results provide important data to develop methods to control salt redox potential and the corrosion of contacting metals in systems such as concentrating solar power plants that utilize molten salt as the heat transfer and storage fluid.

ACKNOWLEDGEMENTS

This work was supported by the National Science Foundation through the University of Utah ReUSE REU Program (NSF—1950589).

CONFLICT OF INTEREST

On behalf of all authors, the corresponding author states that there is no conflict of interest.

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