Characterization of Intermediate Phases Formed Between Solid Nickel and Liquid Zinc During Use as an Encapsulated Phase Change Material in Solar Thermal Energy Storage Systems

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Abstract Of the new material systems under investigation for use in higher temperature phase change material-based solar thermal energy storage (TES) systems, Zn encapsulated in Ni is proposed to increase storage capacity and efficiency by allowing solar TES systems to operate at higher temperatures. However, it is possible that the formation of intermediate phases could impede such storage. This study serves to characterize the intermediate phases formed between Zn and Ni at approximately 450 °C for various exposure times between 1 and 16 h. Out of four possible intermediate phases present in the Ni–Zn equilibrium phase diagram, only two were found to exist between Ni and Zn upon quenching under experimental conditions. The combined layer thicknesses for each specimen increased with increasing exposure time and ranged from 25 to 335 µm in the specimens exposed 1 and 16 h, respectively. Electron probe microanalysis showed that the compositions of each intermediate phase corresponded to the γ and δ phases of the Ni–Zn equilibrium phase diagram. Electron backscattered diffraction confirmed the presence of the γ and δ phases and showed that their structures corresponded to the NiZn₃ and Ni₃Zn₂₂ structures, respectively.

Keywords Ni–Zn intermediate phases · Phase identification and characterization · EBSD

Introduction

Solar energy, one of the most attractive renewable energy sources, is being considered as a base load power supply because of its seemingly limitless abundance. Similar to many renewable energy sources, solar energy is intermittent in nature and does not match the load patterns for energy consumption. Thus, more efficient and higher capacity storage of solar energy is an essential part of any strategies to make it competitive as a base load power supply, and the current effort is directed toward the development of methods and materials for its more efficient storage. Without such explicit storage, many solar energy plants, specifically concentrated solar power plants, cannot store or supply energy for more than seven and a half hours after energy collection has ended [1]. The use of low melting temperature salts as phase change materials (PCMs) to store thermal energy as sensible heat in current solar power plants is limiting the operating temperatures, thereby limiting the capacity and efficiency of the solar thermal energy storage (TES) systems.

In an effort to increase the storage duration, storage capacity, and overall efficiency of solar TES systems, materials that can store thermal energy using the latent heat of phase change and operate within a 400–500 °C temperature range are being considered. Materials with higher melting temperatures will enable solar TES systems to store energy at higher temperatures, which can then generate higher enthalpy steam for more efficient Rankine cycle performance [2]. Some metals are being considered as encapsulated PCMs (EPCMs) because of their

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comparable energy storage capability and higher storage temperatures with respect to salts. Not only are some metals more stable above their melting points, but they also do not suffer from dissociation of their constituents as some salts do at elevated temperatures or corrode their encapsulating materials. Lauf and Hamby [3] have shown results for the use of gallium as a PCM encapsulated in graphite canisters at elevated temperatures around 927 °C. They also reported results on LiF–20%CaF contained in Haynes 188 canisters at 767 °C. These systems, however, were intended for gas turbine systems operating on Brayton cycles, but show that the EPCMs' ability to operate at high temperatures exists. Recently, Zn with a melting temperature of 420 °C has been proposed as a phase change storage medium encapsulated in Ni. An analysis of the application of Ni as an encapsulation material for Zn as a phase change medium is discussed in more detail by Blaney et al. [4]. Their study established that the system could safely withstand internal stresses caused by volume and gas expansion at 450 °C and also operate at even higher temperatures. Preliminary results, however, obtained from drop calorimetry tests performed on lab-scale specimen by Zhao et al. [2] showed that the efficiency and amount of pure Zn available to store energy decreased with increasing numbers of melting/solidification cycles. It was found that interaction at the interface between molten Zn and the solid encapsulation material produced intermediate phases, which consumed the Zn. The formation of these intermediate phases was reported to have hindered the storage performance of the system and has led to an investigation characterizing the intermediate phases formed between Ni and Zn at or around 450 °C for various high temperature exposure times.

In the Ni–Zn binary system, there exist two parent phase regimes, Ni and Zn, as well as four intermediate phase homogeneity regions, β_1 , β , γ , and δ , which are seen in the Ni–Zn binary equilibrium phase diagram proposed by Nash and Pan [5] (Fig. 1). These intermediate phases are of importance to the Ni/Zn encapsulation/PCM material system, because the formation and growth of one or more of these phases is detrimental to the storage ability of the TES system. Prior knowledge of the structure and compounds associated with the Ni–Zn intermediate phases will aid in identifying those phases which formed between Ni and molten Zn upon solidification.

The Ni phase regime has a FCC crystal structure and has a high solubility for Zn [5]. Tamaru [6] confirmed this and determined that the magnetic transition in the Ni phase regime decreases as Zn content increases. The Zn phase regime, having a HCP crystal structure has been found to have negligible solubility for Ni [5]. The β_1 phase has a crystal structure corresponding to a CuAu-type [7, 8] structure with $P4/mmm$ symmetry [5]. Liang et al. [9]

proposed that the β_1 phase existed as an FCC superlattice structure. Heike et al. [7] determined a stoichiometric formula for the β_1 phase to be NiZn, which was also determined by Hansen and Anderko [10]. This phase has also been modeled as a structure with substitutional defects and vacancies on two sublattices [11, 12]. Unlike the β_1 , the β phase is a high temperature phase that cannot be retained upon quenching. Its crystal structure is that of the CsCl-type as reported by Heike et al. [13]. This phase has limited coverage in the literature on characterizing its crystal structure because of difficulties of retaining it at room temperature. However, Liang et al. [14] observed the transformation of the β phase to the face centered tetragonal AuCu-type, β_1 phase.

Not necessarily agreed upon among studies of the γ phase, the crystal structure associated with the γ phase is the Cu_5Zn_8 -type (γ brass) structure, which was determined by Ekman [15] who also determined the stoichiometric formula to be $\text{Ni}_5\text{Zn}_{21}$. Heike et al. [7] and Nover and Schubert [16] found that the γ phase had a structure that correlated with the formula, NiZn_3 , which is at the composition where congruent melting of this phase takes place. Johansson et al. [17] reported the stoichiometry for the γ phase as $\text{Ni}_4\text{Zn}_{22}$. Schramm [18] reported an extension of the γ phase, γ_1 , from 73.5 to 78 at.% Zn that had a distorted γ brass structure. Morton [19] then reported that there was very little difference in the diffraction patterns of the γ_1 phase and the γ phase, which led to the suggestion that the γ_1 phase and the γ phase be combined into one single γ phase region.

The δ phase crystal structure is that of a CoZn_{13} -type structure with $C2/m$ symmetry as found by Critchley and Denton [20]. Owing to its narrow homogeneity range, validated by Schramm [18], the δ phase is commonly considered a stoichiometric, intermetallic, line compound, NiZn_8 [7, 10, 11, 21]. However, Su et al. [12] considered the δ phase a stoichiometric intermetallic compound with a fixed composition of $\text{Ni}_7\text{Zn}_{15}$. Tamaru and Osawa [22] and Duchenko and Dybkov [23] also reported that the δ phase was a stoichiometric compound, but with the formula, $\text{Ni}_3\text{Zn}_{22}$.

The focus of this research is to characterize the intermediate phases formed between molten Zn and solid Ni during various durations of exposure and to determine the identity of each of the phases present. This research is motivated by the need to find storage alternatives for renewable energy sources as well as enhance existing energy sources to improve upon the cost and capacity of storage systems. Studies conducted by Lauf and Hamby [3] and Zhao et al. [2] have already shown that there is potential in using metal PCMs; however, it has also been reported that the efficiency and storage capabilities decrease because of unwanted metallic interaction between Ni and Zn during storage cycles. Describing the metallic interaction between Ni and Zn and characterizing its

Fig. 1 Ni–Zn binary equilibrium phase diagram [5]

Table 1 Composition of commercially pure Ni Alloy 200 (values are reported in wt.%)

Element	C	Cu	Fe	Mn	Ni	Si	S
Concentration	<0.020	<0.25	<0.40	<0.35	>99.0	<0.35	<0.010

Table 2 Composition of 99.997% pure Zn (values are reported in wt.%)

Element	Al	Cd	Cu	Fe	Pb	Sn	Zn	OT
Concentration	<0.002	<0.003	<0.002	<0.003	0.003	<0.001	99.997	<0.010

products to support the decrease in storage capability in the Zn–Ni EPCM system form the scope of this research.

Materials and Experimental Procedure

Specimen and Testing

The materials used in this investigation were Ni Alloy 200 (commercially pure Ni) and 99.997% pure Zn (UNS-Z-13001 Zn-2 special high grade Zn). The chemical compositions of each are given in Tables 1 and 2, respectively. The Ni was supplied as 12.7-mm (0.5 in.)-diameter rods, and the Zn was supplied as 36 kg (80 lbs.) ingots.

Five 41.2-mm-long (1.63 in.) rods were sectioned from the original Ni rods. A 4-mm (0.17 in.)-diameter hole was then drilled perpendicular to the long axis 11.1 mm (0.44 in.) from one end, through which a 316 stainless steel rod was placed during hot dipping in molten Zn. The purpose

of the rods was to span the inner diameter of the crucibles to suspend the Ni rods in the molten Zn. The surfaces of each rod were then ground in succession with 320, 400, and 600 grit SiC papers to make the surfaces uniform and remove imperfections. Small, cylindrical billets with dimensions of 24.6-mm (0.97 in.) diameter and 25.4-mm (1 in.) height were machined from the Zn ingot. The rods and billets were cleaned with a degreasing agent in an ultrasonic cleaner. They were then rinsed with water and ethanol. Custom crucibles were made from an alumina silicate ceramic (lava stone), and they were baked out at 250 °C to remove any moisture before the addition of the Ni–Zn specimen.

Single Zn billets were placed in each of the five crucibles, then heated to 450 °C, and held there for approximately 2 h using a box furnace. Argon gas was flowed into the furnace at a rate of approximately 250–300 mL/min to ensure minimal oxidation of the melt. Once the Zn was completely molten, one Ni rod was dipped into each crucible of molten Zn. Each Ni/Zn couple was held at 450 °C

Fig. 2 Light micrographs of (a) NZ1, (b) NZ2, (c) NZ4, (d) NZ8, and (e) NZ16 samples under crossed polarized light

for various amounts of time. The hold times were 1, 2, 4, 8, and 16 h. After the hold times were completed, the Ni rods were removed from the molten Zn and immediately quenched in water. Water quenching was performed to retain the microstructure and phases found at the elevated temperatures. The specimens were designated NZ1, NZ2, NZ4, NZ8, and NZ16 according to their exposure times.

Metallographic Preparation

Disk-shaped specimens were sectioned from each rod, and 2 mm × 2 mm × 7 mm specimen containing the intermediate phases were then sectioned from each disk. The specimens were mounted and metallographically prepared down to a 0.25-μm diamond finish using a completely waterless process. The grinding lubricant in place of water was a 60/40 mixture of ethyl alcohol to ethylene glycol, which had an adjusted pH of approximately 7–8. The pH was adjusted with the addition of dissolved sodium hydroxide in ethanol. Standard waterless lubricants and suspensions were used for polishing. This inhibited galvanic corrosion of the Zn and Zn-containing phases in contact with Ni. Rinsing and cleaning between each successive grinding and polishing step was also performed without the use of water.

Optical Analysis

Intermediate phases, which formed between the molten Zn and solid Ni, were examined using light microscopy. Polarized light was used to increase grain contrast and differentiate the intermediate phases present in each

specimen. Thickness measurements were made for each intermediate phase present as well as total layer thickness measurements for each specimen.

Electron Probe Microanalysis (EPMA)

An electron microprobe was used to determine the concentration of Ni and Zn within the intermediate phases that formed between the solid Ni Alloy 200 exposed to molten 99.997% pure Zn. The electron microprobe was operated with an accelerating voltage of 20 kV, a beam current of 30 nA, a 5-μm probe diameter, and at an analysis magnification of ×4000. Concentration profiles from the Ni, across the intermediate phases, to the Zn were measured for each of the five samples.

Electron Backscatter Diffraction (EBSD) Analysis

EBSD was performed on each of the five specimens. Five to ten patterns were acquired and indexed within each intermediate phase region of each of the five specimens to provide statistical confirmation that the indexed phases were correctly indexed. The patterns and indexes were recorded for each layer from each sample.

Results and Discussion

It was observed that only two out of four possible intermediate phases indicated in the equilibrium phase diagram in Fig. 1 were present in each specimen. This is in contrast

to the model of the Ni–Zn system under similar conditions proposed by Alzahrani [24], in which all four intermediate phases (α (Ni), β_1 , γ , and δ) were predicted to be present. Figure 2 shows representative light micrographs of each of the five specimens. These intermediate phase layers (IPL1, adjacent to Ni; IPL2, adjacent to Zn) were distinguishable because of different grain morphologies color, which was enhanced with polarized light. In order to identify the intermediate phase layers, EPMA and EBSD were used as characterization techniques.

Using EPMA, concentration profiles were made from regions of pure Ni, across both intermediate phase layers, to regions of pure Zn in NZ1, NZ2, NZ4, NZ8, and NZ16. Figure 3 shows the concentration profiles obtained from EPMA. The average composition ranges measured across IPL1 and IPL2 for each specimen were found to be approximately 78.5–85.4 and 87.6–90.1 wt.% Zn, respectively. The individual composition ranges for each layer and specimen are shown in Table 3 along with the layer thicknesses over which the compositions were measured. These composition ranges lie within the γ and δ phase homogeneity regions of the Ni–Zn equilibrium phase diagram shown in Fig. 1, which exist from 76.0 to 87.0 wt.% Zn and 89.5 to 91.0 wt.% Zn, respectively. It should be noted that the average composition range determined for IPL2 extends slightly beyond the Ni-rich side of the δ phase into the $\gamma + \delta$ phase regime.

Further confirmation of the identification of the intermediate phase layers was established with EBSD. Figure 4 shows representative EBSD patterns for pure Ni, the first and second intermediate phase layers, and pure Zn. Alongside each pattern is the same pattern with a simulated and indexed pattern overlay. The first intermediate phase layer, adjacent to the Ni, was indexed as NiZn_3 , which agrees with the compound formula and structure ascribed to the γ phase by Heike et al. [7], and Nover and Schubert [16]. The second intermediate phase layer, adjacent to the Zn, was indexed as $\text{Ni}_3\text{Zn}_{22}$, which agrees with the compound formula and structure ascribed to the δ phase by Tamaru and Osawa [22] and Duchenko and Dybkov [23]. These two compounds have crystal structures of the gamma brass and monoclinic CoZn_{13} -type structures, respectively. The gamma brass, orthorhombic structure as indexed by the EBSD software for NiZn_3 had the following associated lattice parameters and lattice vector angles: $a = 33.326 \text{ \AA}$, $b = 8.869 \text{ \AA}$, $c = 12.499 \text{ \AA}$, and $\alpha = \beta = \gamma = 90^\circ$. Likewise, the lattice parameters and lattice vector angles associated with the monoclinic $\text{Ni}_3\text{Zn}_{22}$ phase were the following: $a = 13.370 \text{ \AA}$, $b = 7.47 \text{ \AA}$, $c = 7.65 \text{ \AA}$, and $\alpha = \gamma = 90^\circ$ and $\beta = 113.3^\circ$. These values and structures were determined by Nover and Schubert [16] and Critchley and Denton [20] for the γ and δ phases, respectively, and were found to be appropriate fits for IPL1 and IPL2.

In all cases, the diffraction patterns obtained for IPL2 were not well defined and obscured because of what looked like multiple patterns existing simultaneously. This caused the patterns to occasionally be identified by the indexing software as $\text{Ni}_5\text{Zn}_{21}$, $\text{NiZn}_{7.33}$, and NiZn_3 . A plausible explanation for the indexing of multiple phases is that the composition range associated with the second intermediate phase layer extends into the $\gamma + \delta$ phase regime (IPL2: 87.6–90.1 wt.% Zn; $\gamma + \delta$: 87.0–89.5 wt.% Zn; δ : 89.5–91.0 wt.% Zn). This slight extension into the $\gamma + \delta$ phase regime may cause the presence of two crystal structures to simultaneously exist in IPL2, which would in turn distort the EBSD patterns and make discerning a unique, indexed pattern difficult. However, all of the possible crystal structures identified by the software ($\text{Ni}_5\text{Zn}_{21}$, $\text{NiZn}_{7.33}$, and NiZn_3) for IPL2 have all been assigned to the γ and δ phases in the literature.

The composition ranges of IPL1 and IPL2 (γ and δ) in each specimen exist over thicknesses, which are proportional to their exposure times at 450 °C. Figure 5 shows the relation between layer thickness and exposure time. As the exposure time increases, the layer thicknesses of IPL1 and IPL2, as well as the total combined layer thickness increase; however, the thicknesses of IPL1 and IPL2 consistently differed from each other. The thinnest total combined layer was of 25- μm thickness, with IPL1 and IPL2 having respective thicknesses of 11 and 14 μm found in NZ1. The thickest total combined layer, found in NZ16 was of 335- μm thickness, with IPL1 and IPL2 being of 134- and 201- μm thicknesses, respectively. Table 3 summarizes the thicknesses as well as composition ranges measured for IPL1 and IPL2 in each specimen.

The IPL2 layer was found to be consistently thicker than the IPL1 layer in each specimen. This is due to the different diffusivities in each phase as well as a difference in crystal structures of γ and δ , which have a gamma brass structure and monoclinic CoZn_{13} -type structure, respectively. According to Duchenko [25], the reaction diffusion constant for the γ and δ phases can be described by the Arrhenius equation:

$$k_1 = A \exp(-E/RT) \quad (1)$$

where k_1 is the reaction diffusion constant (m^2/s), A is the pre exponential (m^2/s), E is the activation energy (J), R is the ideal gas constant (J/mol K), and T is temperature (K). The values of A and E for γ and δ were determined by Duchenko to be $1.1 \times 10^{-9} \text{ m}^2/\text{s}$ and 55.4 kJ and $6.4 \times 10^{-6} \text{ m}^2/\text{s}$ and 103.3 kJ, respectively. Using the values found by Duchenko and the Arrhenius relation with $T = 723 \text{ K}$ (450 °C), values of k_1 (diffusivity) for each phase were found. The diffusivities for the γ and δ phases were 1.09×10^{-13} and $2.20 \times 10^{-13} \text{ m}^2/\text{s}$, respectively. The diffusivity in the δ phase is approximately two times faster than that in the γ phase. This could account for the formation of thicker

Fig. 3 Linear concentration profiles for NZ1, NZ2, NZ4, NZ8, and NZ16 samples determined by EPMA. The average composition ranges of the IPL1 and IPL2 layers are approximately 78.5–85.4 and 87.6–90.1 wt.% Zn, respectively

Table 3 Composition ranges and layer thicknesses for IPL1 and IPL2 determined for each specimen using EPMA

Specimen	NZ1	NZ2	NZ4	NZ8	NZ16
IPL1					
Composition range (wt.% Zn)	79.5–86.5	77.9–85.0	78.1–84.6	79.6–85.4	77.9–85.8
Thickness (μm)	11	27	44	85	134
IPL2					
Composition range (wt.% Zn)	87.8–90.5	87.0–90.2	86.8–90.0	88.9–90.4	88.5–89.5
Thickness (μm)	14	40	72	136	201

Fig. 4 EBSD patterns for Ni, IPL1, IPL2, and Zn with corresponding simulated pattern overlays alongside on the right of each pattern

layers of δ phase, because atoms can move quickly through the volume of the IPL2 layer and to the interface to promote growth of the IPL2 layer. Another possible reason for the thicker layers of IPL2 is that these layers are adjacent to molten Zn. The Zn atoms in the molten state are highly mobile and will be able to diffuse into the IPL2 layer, in effect directly supplying Zn atoms to form $\text{Ni}_3\text{Zn}_{22}$. In comparison, to form the IPL1 layer (γ phase) the Zn atoms have a longer path to travel to form NiZn_3 , because they must first diffuse through the IPL2 layer.

A typical power law expression in the form of

$$x = kt^n \quad (2)$$

where x is the thickness, k is the growth rate constant, t is time, and n is the growth exponent set equal to 0.5 (indicating

Fig. 5 Experimentally measured thicknesses and thickness trend lines with respect to exposure time for IPL1, IPL2, and the combined (total) layers

diffusion controlled layer growth), was found to not fit the experimental data well. A growth exponent equal to 0.5 is typical for solid/solid metallic interaction and has been reported to fit well to layer formation in binary systems by others [23, 26–28]. Duchenko and Dybkov [23], who have performed much research on the Ni–Zn system, reported that a parabolic relationship ($n = 0.5$) between the layer thickness and isothermal hold times shows strong correlation with experimental data for the Ni–Zn system at elevated temperatures below the melting temperature of Zn (420 °C). In the current experiment, a parabolic correlation could not be used, because of kinetics associated with solid/liquid interactions. The presence of a liquid component at an elevated temperature causes diffusion to occur at rates, which are higher than rates associated with solid/solid diffusion, as well as introduces exacerbated reaction–diffusion kinetics because of dissolution of the solid component into the liquid [29].

Even though a simple parabolic power law curve did not fit the experimental data, the data did show typical trends for layer formation. The data presented in Fig. 5 still show common trends for experimentally measured layer thicknesses in relation to exposure time. As the exposure time increased, the thickness of the layers increased, but at a decreasing rate. The decreasing growth rate may be associated with increased layer thickness. As the layer thickness increases, the diffusion path for Ni and Zn atoms to the layer interfaces also increases, in effect decreasing the contribution to growth from atomic diffusion. Ni and Zn atoms cannot be supplied to the interfaces so quickly, and further growth will be limited by diffusion.

In each case, only two intermediate phases were observed, and their presence was confirmed with EPMA and EBSD techniques. The formation of fewer phases than predicted by equilibrium phase diagrams has been reported for many other binary and multicomponent metallic systems such as

the Fe–Al system [30], the Cu–Zn system [31], and the Fe–Zn system [32]. For the Ni–Zn system, in particular, it has been proposed that formation of only two phases occurs because of chemical reactions at the interface between Ni and Zn and at the interfaces between intermediate phases. Dybkov [29] suggests that $\text{Ni}_3\text{Zn}_{22}$ (δ phase) immediately forms, and after its thickness becomes significant to the point where appreciable diffusion of Zn through the layer occurs, NiZn_3 (γ phase) begins to form. Once the two phases are present, they grow simultaneously according to chemical reactions expressed by Dybkov [29], but at unequal rates because of the diffusivity through each phase and the relative mobility of atoms from the parent materials (Ni and Zn).

Conclusions

In this study, solid Ni rods were dipped into molten Zn at 450 °C for exposure times of 1, 2, 4, 8, and 16 h. The subsequent intermediate phases, which formed between the Ni and Zn, were analyzed, and it was found that in each specimen only two intermediate phase layers formed under the experimental conditions. The average concentration ranges over the two respective thicknesses were 78.5–85.4 and 87.6–90.1 wt.% Zn. These concentration ranges correspond to the γ and δ phase homogeneity regions of the Ni–Zn equilibrium phase diagram, respectively. Further confirmation of the presence of the γ and δ phases was provided by EBSD, which resulted in the identification of NiZn_3 and $\text{Ni}_3\text{Zn}_{22}$. These two compounds have been ascribed to the formation of the γ and δ phases, respectively, by other authors. Results also showed that as the exposure time increased, the total layer thickness increased at a decreasing rate, which indicated that the layer growth rate decreased because of longer diffusion paths due to increasing thicknesses.

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