



Synthesis of polymer in electrolyte method by using the metal ION: Nickel (II) Chloride

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Abstract

A Shape Memory Alloy is an alloy undergoes deformation by applying heat. It changes its shape at higher temperature and return back to original geometry at lower temperature. There are of two symmetry transformation, Martensitic at lower temperature and Austenite at higher temperature. This extraordinary character is known as Pseudo elasticity. Pseudo elasticity is not only an exact elastic property as Polymer materials. It is a peculiar elastic property of some metal alloys and Polymer mixed metal alloys. It undergoes compression and elongation with ambient temperatures. This elastic property is otherwise known as Super- Elasticity. [Ni Ti] metal alloys have superior mechanical property than other Copper based shape memory alloys. This will be better for our research.

Keywords: titanium dioxide, cadmium chloride, tin chloride, mercuric chloride, sodium hydroxide

1. Introduction

The copper chloride compounds isolated in the present study, their analytical data, molar room conductance data, room temperature magnetic moment values, observed bands. In the electronic spectra and the characteristic infra reds absorption bands. The comparison of the red spectra of the copper chloride complexes with those of the corresponding uncoordinated ligand to spectrum facilitated the assignment of bands sulphate to group in the range 1300-1000 ml/l. Secondary lithium batteries are sophisticated energy storage devices that have received considerable attention over the last 30 years. As result of their superior energy and power density, and the absence of a "memory" effect, solid- state lithium iron batteries ^[1-3] have become the preferred power source for much portable application. The search for solid polymer electrolytes (SPEs) with high ionic conductivity and good mechanical, electro chemical and thermal stability continues to provide a challenge to many research group ^[4]. The development of a successful solid state lithium polymer battery is dependent upon the choice of a suitable host polymer matrix. Many of these materials suffer from the disadvantage that the ionic conductivity necessary for practical application sites it is not possible to use the conventional SPE notation that expresses the ratio of monomer units to lithium. To avoid this problem the salt composition has been given as wt% of the salt in the polymer electrolyte.

2. Experimental method

2.1 Ionic Conductivity

The total ionic conductivity of the p(EEO-AGE) Li X (wt%) electrolytes was determined by locating an electrolyte disk between two 10mm diameter ion-blocking gold electrolytes (good fellow, >99.95) to form a symmetry >99.95% to form a symmetrical cell. The electrode/ p(EEO) LiX (wt%) electrode assembly was secured in a suitable constant-volume support ^[12] and installed in a Buchi TO51 tube oven. A calibrated type K thermocouple was placed close to electrolyte disk to measure the sample temperature. Bulk

conductivities of electrolyte and ate samples were obtained during heating cycles using the complex plane impedance technique (Schlumberger Solartron 1250 frequency response analyzer and 1286 electrochemical interface) between 25 and 100°C and at approximately 7°C.

2.2 Thermal Analysis

Polymer electrolyte sections were removed from dry films and subjected to thermal analysis under a flowing argon atmosphere between 25 and 300°C and at a heating rate of 5°C min⁻¹ using a 10°C was used with all samples mettelle DSC 821 e. samples were transferred to 40 µL aluminum cans with perforated lids with in a dry argon can only be reached at higher- then ambient temperature, due to the degree of crystalline inherent in this host matrix at room temperature. An important consequence of using this terpolymer is that there are three different locations where interactions between the polymer and guest ion species may take place. Without prior knowledge of the distribution of guest salt between these field glove box. samples for thermo gravimetric studies were prepared in an identical manner, transferred to open platinum crucibles using a Rheumatic scientific TG1000 thermo balance operating under a flowing argon atmosphere.

2.3 Electrochemical Stability

Evaluation of the electrochemical stability window of electrolyte compositions was carried out under argon using a two- electrode cell configuration. A preparation of a 25µm diameter gold microelectrode surface, by the conventional polishing routine, was completed outside the dry box. the microelectrode was subsequently washed with THF, dried with a hot-air blower and transferred to the dry box. The cell assembly was initiated by locating a clean lithium disk counter electrode (Aldrich, 99.9%, 10mm didmeter, 1mm thick) on a stainless steel current collector. A thin-film sample electrode was placed over the counter electrode and the cell assembly was completed by locating by supporting the microelectrode in the center of the electrode disk. the

assembly was held together firmly with a clamp and electrical contacts were made to Auto lab PGSTAT-12 (Eco chemise) used to record volt ammo grams d at a scan rate of 100m Vs^{-1} . measurements were carried out at room temperature with in a Faraday cage located inside the measurement glove box.

2.4 Conductivity

The ionic conductivity in polymer electrolytes is assumed by occur by Lewis acid-base interaction between the cations and the polymer solvent. it also generally accepted that the ionic condition occurs mainly in the amorphous component of polymer electrolytes above their glass transition temperature, t_g , with the chain segment mobility playing a critical role in the conductivity mechanism. A strategy which has been developed by many authors for enhancing ion mobility, and consequently the conductivity, is based on the use of a completely amorphous polymer with low values of a class transition temperature. The terpolymer used in the present SPES combines and only amorphous properties of the epichlorohydrin but also solvating behavior of the ethylene oxide and allyl glycidyl segment. It has suggested that at high salt content formation of ion aggregates will contributes to a decrease in conductivity. In the p(EEO-AGE) Li TFSI (Wt%) electrolyte system very large amount of guest salt can be dissolved in the host polymer without phase separation, a situation that has already been reported for a previous salt/host polymer system.

2.5 Thermal Analysis

Figure 3 a) and b) represent the DSC curves for the p(EEO-AGE) LiClO_4 (Wt%) and p(EEO-AGE) LITFSI(Wt%) electrolyte systems, respectively, and confirm that the electrolytes characterized are completely amorphous. No fusion of regions of oxy-ethylene chain segments of un complexes polymer, or polymer-salt complexes, is apparent in any of the electrolyte studied and the case of the perchlorate-based electrolyte and 274°C in the p(EEO-AGE) Li THFS I(Wt%) electrolyte system. The absence of crystalline in samples of both networks was expected from conductivity measurements. It should be noted that this result is not in agreement with a previously report study. In this latter case the authors concluded that a polymer-salt complex was formed at high compositions of guest salt (low values of n), giving rise to a melting peak at close to 100°C . As may be confirmed by reference to figure 4, the p(EEO-AGE) LiTFSI (Wt%) electrolyte system shoes a higher thermal stability (293°C) than the perchlorate electrolyte system, which suffers a strongly exothermic process the intensity of which is dependent on the amount of salt present in the sample. A Shape Memory Alloy is an alloy undergoes deformation by applying heat. It changes its shape at higher temperature and return back to original geometry at lower temperature. There are of two symmetry transformation, Martensitic at lower temperature and Austenite at higher temperature. This extraordinary character is known as Pseudo elasticity. Pseudo elasticity is not only an exact elastic property as Polymer materials. It is a peculiar elastic property of some metal alloys and Polymer mixed metal alloys. It undergoes compression and elongation with ambient temperatures. This elastic property is otherwise known as Super- Elasticity [Ni Ti] metal alloys have superior mechanical property than other Copper based shape memory alloys. This will be better for our research.

2.6 Introduction to Shape Memory Alloy

Shape Memory Alloys regain its original crystal logic geometry and changes its original shape at different temperatures. This extra ordinary property's called as Pseudo-elasticity (or) super elasticity.

2.7 One Way Vs Two Way Shape Memory Alloys

There are two most common memory effects, i) one way and ii) two-way shape memory Alloys. In fig1 (a) one way irreversible deformation after higher temperature. In two-way –reversible deformation on cooling after heating, get back to its original position.

One way shape alloy: Two way shape alloy

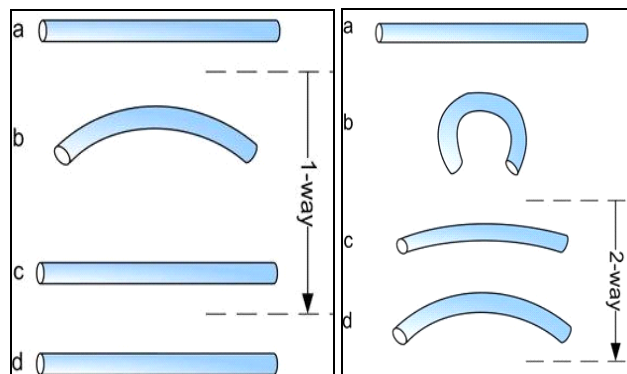


Fig 1

2.8 Preparation

The preparation of SMA is carried out in four stages. That is as follows:

Stage I: We have taken Titanium dioxide salt, Cadmium salt and Tin chloride, Mercuric chloride salt in the ratio of 15.0%, 14.0%, 14.5%, 4.5% respectively and we have mixed the same gently in the acidic media of sulphuric acid. The mixture is subjected to cation (Ultra sonic agitation) for two to three hours for Homogenous mixing. After thorough mixing, the solution is allowed in a hot air Oven at 60°C for four hours. Again sonicated the solution for two hours. Cooled the solution to 15°C and Tween agent is added 2 to 3cc.

Stage II: Kept the solution in the Ultrasonic Agitator. Sodium hydroxide pellets are slowly and gently added into the solution. The entire solution is kept at 15°C . Precipitates are formed from the solution. The solution is subjected to Centrifugation (10,000 rpm) for 15 minutes. The clear solutions decanted. The precipitate is washed with distilled water for 5 to 6 times. After washing the Precipitate is kept in Vacuum oven at 80°C for two hours.

Stage III: The Residue is kept at inert oven at 200°C for four to six hours in a Beaker. After drying, the residue is stirred well using glass rod and again kept in the inert condition at 300°C . After drying, the residue is stirred well using glass rod and again kept in the inert condition at 400°C .

Stage IV: Collected the dried power and kept in to the button die, (spanch) to make at him pellet. The power is spread uniformly in to the die and punch is kept on to the die carefully. The punch and the die are kept in an appropriate position of the pneumatic press of 1 to capacity. 150 Kg/cm^2 pressure applied for 5 minutes. After pressing at pellet of strong alloy we got.

3. Result and Discussion

3.1 Electronic Test Data

We make the two types of memory metal to the electronic

test. This also supports to the Two-way memory metal are better than the one way memory metal. This is due to its elastic property.

Table 1

	Comparator Output voltage (V)	Sensor volt(V)	Temperature (OC)	System
One Way memory metal	1.062	6.2	Room	Open
	4.0	5.0	40	Open
	4.2	3.0	50	Open
	4.8	2.0	60	Close
	5.0	1.0	70	Close
	5.0	0.0	80	Close
Two Way memory metal	1.062	6.2	Room	Open
	4.0	5.0	40	Open
	4.2	3.0	50	Close
	4.8	2.0	60	Close
	5.0	1.0	70	Open
	5.0	0.0	80	Open

3.2 Electronic Test Reading

The temperature dependence of the sensor response is

related to the strength of the polymer solvent interaction and the heat of vaporization of the solvent.

Comparative Analysis of One-way SMA and Two-way SMA

Table 2

S. No.	One-way SMA	Two-way SMA
1.	Composition of the Memory Metal are Ti, Cd.	Composition of the Memory Metal are Sn, Cd
2.	Preparation ratio is 15.0%, 14.0%, 14.5%, 4.5% of Titanium dioxide salt, Cadmium chloride salt and, mercuric chloride salt respectively.	Preparation procedures are same. Composition changes with respect to Al addition ratio.
3.	Property Study There is no change in the shape of the Memory metal after 60°C	Property Study Regain to its Original shape even at elevated temperature
4.	Electronic study After 70°C there is no change in the shape of the metal. It will not compress even after the higher temperature.	Electronic study There is a slight decrease in its shape at elevated temperature. So this made the memory metal to open the circuit. By the way the current cut OFF to the circuit.
5.	Piezo-electric property is very low 0.02 eV.	It is around 0.65 eV.
6.	Sensing property 1. Additional toggle switch is required for the operation at higher temperature. 2. Once expanded will not get back to its original position.	Sensing property 1. There is no need for any additional switches for switch OFF heater. 2. It regains its Original shape at elevated temperature as well as at room temperature.

Table 1: Temperature Vs Absorbance Characteristics.

S. No.	Absorbance in ppm	Temperature in 0°C	
		One Way SMA	Two Way SMA
1	0.1	32	34
2	0.2	39	35
3	0.3	46	36
4	0.4	53	37
5	0.5	60	38
6	0.6	67	39
7	0.7	74	40
8	0.8	81	41
9	0.9	88	42
10	1.0	95	43

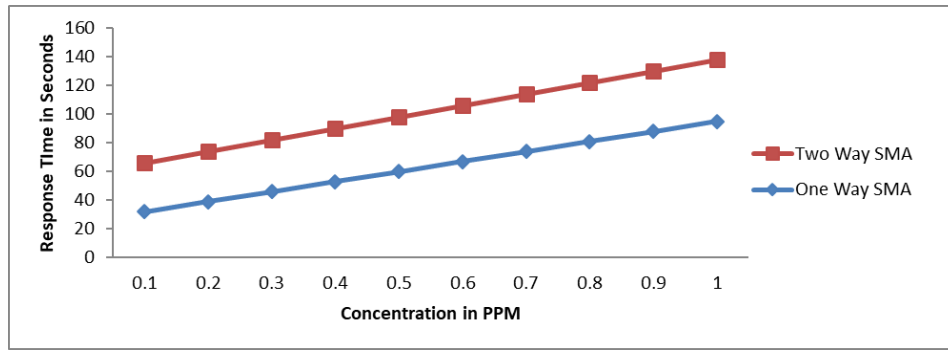


Fig 1: Figical representation of Temperature Vs Absorbance

- In the case of One-Way SMA all near line in fig observed because the temperature and the Absorbance are directly proportional.
- If the concentration increases then the temperature also increases.
- At higher concentration the One-Way SMA will not get back to its original shape. So the elasticity also decreases at higher concentration and temperature.
- But in the case of two-ways SMA, slight deviation noticed from the linear line at 1.2 ppm. This indicates the change in shape of that Memory Metal at higher temperature i.e. at 82 °C.

Table 2: Resistance Vs Temperature Characteristics.

S. No	Temperature	Resistance in Ω	
		One Way SMA	Two Way SMA
1	35	0.6	0.7
2	40	0.9	1.1
3	45	1.2	1.5
4	50	1.5	1.9
5	55	1.8	2.3
6	60	2.1	2.7
7	65	2.4	3.1
8	70	2.7	3.5
9	75	3.0	3.9
10	80	3.3	4.3

Resistance Vs Temperature Characteristics

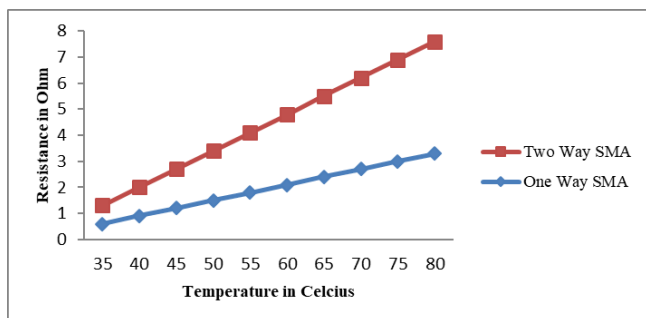


Fig 2: Physical representation of Resistance Vs Temperature.

- In the case of One-Way SMA shows a linear line observed in fig-2 since the temperature and the Resistance is directly proportional.
- If the Temperature increases then the Resistance also increases. From this we in ferried that the memory metal prepared by method-1 does not have the elastic and piezo- electric property. After higher temperature that will not get back to its original state.
- But in the case of Two-Way Memory Metal, up to a

particular temperature it is directly proportional after the higher temperature (say 65°C the resistance begun to decrease. From this we inferred that the Two-Way SMA has Elastic Character and shape Memory retrieval nature changes.

Table 3: Reponses Time Vs Concentration Characteristics.

S. No	Concentration in ppm	Response time in seconds	
		One Way SMA	Two Way SMA
1	40	40	50
2	45	50	70
3	50	60	90
4	55	70	110
5	60	80	130
6	65	90	150
7	70	100	170
8	75	110	190
9	80	120	210
10	85	130	230
11	90	140	270
12	95	150	290

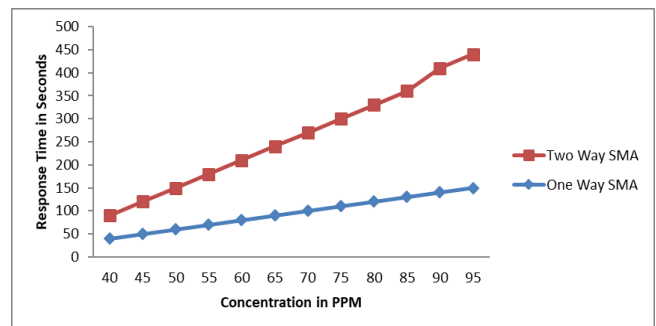


Fig 3: Physical representation of response time vs concentration

In the case of One-Way SMA and Two-Way SMA shows a linear line in the fig because Response Time and the Concentration are directly proportional. The Fig shows after a higher temperature also both may respond equally (i.e.) increase in concentration the time for the respond also same.

4. Conclusion

There are two most common memory effects, i) One way and ii) Two -way shape memory Alloys. In fig 1(a) one way irreversible deformation after higher temperature. In two way - reversible deformation on cooling after heating, get back to its original position. The Cooling from higher temperature does not cause a drastic shape change. In two - way shape memory effect, the metal alloy remembers two different geometries (i.e.) Marten site and Austenite. The

Material has trained to remember its higher temperature geometry at lower temperature geometry. it can be “trained” to “remember” to leave some reminders of the deformed low - temperature condition in the high-temperature phases.

- One-Way and Two-Way SMA is compared.
- Advantages are noticed in the Two-Way SMA.
- Even though there is a limitation Two-Way SMA. Preparation is very difficult time taken is very long.
- Continuous operation of Two-Way SMA yet to be studied for its repeatability performance and its life.

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