

# THE EFFECT OF KCl PLATING BATH TEMPERATURE ON THE REFLECTANCE OF Cu/Ag

*By* M. TOIFUR



# Proceedings

## <sup>6</sup> The 2007 Conference on Solid State Ionics

*in conjunction with*

## Scientific Gathering on the International Joint Research Program



August 1-3, 2007

NATIONAL RESEARCH COUNCIL

PUSPIPTEK Area, Serpong, TANGERANG 15314 INDONESIA

<sup>2</sup>  
Organized by:

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Agency of Assessment and Application of Technology (BPPT)

In collaboration with:

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## FOREWORD

Solid state ionics are attracting particularly from industry and academia. These materials play an important key in many technologies, including energy conversion, microelectronic, sensor, and health diagnostic. Solid state ionics materials which deal with ionically conducting materials cover a wide spectrum, ranging from inorganic crystalline and polycrystalline solids, ceramics, glasses, polymers, composites and also nano-scale materials. A large number of scientists all over the world are engaged in research activities in solid state ionic materials and devices. The Asian Society of Solid State Ionics and the International Society of Solid State Ionics have played a key role in organizing conferences on solid state ionics. However, Indonesia having a large number of scientists working in this field has yet to convene in a formal forum to discuss the solid state ionics current status. Therefore, it is important to organize an event that can accommodate and meet the need of formal meeting where an intensive communication between scientists could be arranged. The theme of this conference, *Advanced Research for Better Life*, describes the expected results coming from this event.

Useful result in the future could be achieved through an intensive research and development activity. The 2007 Conference on Solid State Ionics (2007-CSSI) is expected to become a media for related parties coming from research and development institutions, universities and industries in order to take the advantages from opportunity offered by solid state ionics field of research through intensive collaboration.

The Organizing Committee has accepted 11 papers from Invited Speakers, and 38 papers from local and international authors. They were divided into 6 groups as follow:

Solid State ionics	18 papers
Synthesis	5 papers
Sensor and Instrumentation	3 papers
Energy	9 papers
Health	1 papers
Others	2 papers

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This conference is expected to become the initiation for another conference in the field of solid state ionics and its related aspect in the future.

Serpong, August 1, 2007

Editor

# PROCEEDINGS

6

**The 2007 Conference on Solid State Ionics***in conjunction with***Scientific Gathering on the International Joint Research Program****August 1-3, 2007****NATIONAL RESEARCH COUNCIL****PUSPIPTEK Area, Serpong 15314, TANGERANG, INDONESIA****Advanced Research for Better Life**

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SALINAN :

PENYELENGGARAAN *THE 2007 CONFERENCE ON SOLID STATE IONICS (2007-CSSI) IN CONJUNCTION WITH SCIENTIFIC GATHERING ON THE INTERNATIONAL JOINT RESEARCH PROGRAM* DAN PEMBENTUKAN PANITIA

KEPALA BADAN TENAGA NUKLIR NASIONAL,

- Menimbang : a. bahwa dalam rangka menjalin komunikasi ilmiah yang efektif dalam berbagai aspek mengenai perkembangan terakhir bidang *solid state ionics* dan aplikasinya dalam bidang energi, lingkungan dan kesehatan, sehingga mendorong timbulnya kemitraan yang kondusif antar lembaga litbang dan perguruan tinggi baik di dalam maupun luar negeri, maka dipandang perlu untuk menyelenggarakan *The 2007 Conference on Solid State Ionics (2007-CSSI) in Conjunction with Scientific Gathering on The International Joint Research Program*;
- b. bahwa untuk ketertiban dan kelancaran penyelenggaraan *The 2007 Conference on Solid State Ionics (2007-CSSI)* sebagaimana dimaksud pada huruf a, dipandang perlu membentuk Panitia;
- Mengingat : 1. Keputusan Presiden Nomor 103 Tahun 2001 tentang Kedudukan, Tugas, Fungsi, Kewenangan, Susunan Organisasi, dan Tata Kerja Lembaga Pemerintah Non Departemen sebagaimana telah beberapa kali diubah terakhir dengan Peraturan Presiden Nomor 64 Tahun 2005;
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3. Peraturan Kepala BATAN Nomor 392/KA/XI/2005 tentang Organisasi dan Tata Kerja BATAN;

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- Menetapkan : KEPUTUSAN KEPALA BADAN TENAGA NUKLIR NASIONAL TENTANG PENYELENGGARAAN *THE 2007 CONFERENCE ON SOLID STATE IONICS (2007-CSSI) IN CONJUNCTION WITH SCIENTIFIC GATHERING ON THE INTERNATIONAL JOINT RESEARCH PROGRAM* DAN PEMBENTUKAN PANITIA.
- PERTAMA : Menyelenggarakan *The 2007 Conference on Solid State Ionics (2007-CSSI) in Conjunction with Scientific Gathering on The International Joint Research Program* yang selanjutnya dalam Keputusan ini disebut *The 2007-CSSI* pada tanggal 1 – 3 Agustus bertempat di Kawasan Puspiptek Serpong, Tangerang.



BADAN TENAGA NUKLIR NASIONAL

- 2 -

- KEDUA : Membentuk Panitia *The 2007-CSSI* yang selanjutnya dalam Keputusan ini disebut Panitia dengan susunan seperti tersebut dalam Lampiran Keputusan ini.
- KETIGA : Segala biaya untuk penyelenggaraan *The 2007-CSSI* dibebankan pada Anggaran Riset Unggulan Terpadu Internasional (RUTI) IV Tahun 2007.
- KEEMPAT : Panitia wajib memberikan laporan mengenai penyelenggaraan *The 2007-CSSI* termasuk pertanggungjawaban keuangan kepada Kepala BATAN selambat-lambatnya 1 (satu) bulan setelah penyelenggaraan *The 2007-CSSI* berakhir.
- KELIMA : Keputusan ini mulai berlaku pada tanggal ditetapkan.

Ditetapkan di Jakarta  
pada tanggal 2 April 2007

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## OPENING REMARKS CHAIRMAN OF CSSI-2007

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*His Excellency, State Minister of Research and Technology of the Republic of Indonesia,  
Dr. Kusmayanto Kadiman*

*The Honorable Chairman of National Nuclear Energy Agency, Dr. Hudi Hastowo*

*Distinguished Guests*

*Ladies and Gentlemen,*

7

Welcome to the The 2007 Conference on Solid State Ionics *in conjunction with* Scientific Gathering on the International Joint Research Program

It is our great honor today to gather here at this precious occasion with all of you to share knowledges and experiences among us.

Unlike the other meetings, this conference was initiated by the idea of scientific gathering among the members of the International Joint Research Program (known as RUTI) launched by the State Ministry of Research and Technology, which was conducted since 2005 up to now.

However, we realize the importance of sharing the knowledge to other people and to disseminate the results to scientific public. From this idea, all collaborators met an agreement to conduct workshop and conference, instead of an exclusive meeting, to bring the event to be more useful and fruitful.

The whole program was then divided into 3 (three) main events:

The Workshop and Official Meeting, were conducted yesterday.

The Conference & Scientific Meeting is being held today.

And the technical meeting or RUTI Gathering will be performed tomorrow.

The purpose of the workshop was to introduce the 'Solid State Ionics Field' that covers several topics on 'Development of Lithium-ion battery, Supercapacitor, Polymer Electrolyte Membrane Fuel Cell, and Bio sensor. The lectures were given by worldwide recognized lecturers, from McMaster University, Canada, Ibaraki University, Japan, Nottingham University, Malaysia, BATAN and BPPT, Indonesia. The workshop includes the demonstration and experiment of the devices, which coordinated by the team from BATAN, BPPT, LIPI and Nottingham University, Malaysia. It was attended by more than 50 participants, from either local or international institutions.

The official meeting was intended for bringing up the international collaboration to be more officially implemented. From the discussion, we came into the agreement to sign the MoU between the Technology Center of Nuclear Industry Materials and other collaborating parties as you will see soon.

The conference today consists of a Keynote lecturer, that will be delivered by the State Minister of Research and Technology, followed by 7 (seven) plenary lecturers which are delivered by our distinguished guests from Japan, Australia, Canada, Singapore and Malaysia. There is also a poster presentation from about 43 contributed papers, that covers several topics including the solid state ionics, energy, health, synthesis and others. The authors are coming from local and international institutions and show

the variety in the solid state ionics field. We expect that by this meeting, we can share our knowledge and put the result together as part of the contribution to the national and international research activities. We hope that this very first conference for solid state ionics in Indonesia becomes a sign for the existence of the Indonesian Society for Solid State Ionics activity. We expect in the near future, it will be recognized by both regional and international society of solid state ionics.

Finally, I will express my sincere appreciation to the State Minister of Research and Technology who will deliver the keynote speech; to the Chairman of BATAN, who will open the conference; to the Head of Center for Technology of Nuclear Industry Materials who provide all facilities; to the guest speakers who will present the plenary lectures; and to all the audiences who have given the time to attend the meeting.

At last but not least, my sincere gratitude to all of my RUTI team and the organizing Committee, for their incredible work, and never ending supports, their dedications, until the '2007 Conference of the Solid State Ionics in conjunction of Scientific Gathering of the International Joint Research Program' can be realized.

We hope you have a wonderful time, enjoyable and fruit full meeting.

Serpong, August 1, 2007

Dr. rer. nat Evvy Kartini  
Chairman of the 2007-CSSI

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## THE EFFECT OF KCl PLATING BATH TEMPERATURE ON THE REFLECTANCE OF Cu/Ag

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### ABSTRACT

#### THE EFFECT OF KCl PLATING BATH TEMPERATURE ON THE REFLECTANCE OF Cu/Ag.

Deposition of Ag on the Cu substrate has been done at the various plating bath temperature to obtain the optimum temperature of plating bath that can produce the highest reflectance coefficient. KCl which is used as plating bath was set at various temperatures from 60 up to 90 °C. Electrolysis process was supplied with 5 volt DC voltage during 7 minutes deposition. Many characterizations were done including the ordering of crystalline structure, sheet resistivity and reflectance, each of these was done by XRD, four point probe and UV-vis spectrometer from 390 up to 790Å wave length. The result shows that the optimum plating bath temperature for producing the highest quality Ag film was 80°C which has the highest ordering crystalline structure, sheet resistivity of 0.52 ohm/sq and reflectance coefficient of 98%.

**Key words** : Plating bath temperature, reflectance

### INTRODUCTION

Research concerning Ag (silver) thin film is most widely directed as reflective film or reflective semitransmissive film. These film are used as optical information recording media together with Au, Al, and its alloy [1].

The Ag film has several advantages including the high thermal conductivity (429 W/m-K). This high electrical conductivity compared with other materials, combined with the high reflectance, and high durability in the field containing information recording media add to the advantage of using the material. By these excellents, Ag is used as protective film from electromagnetic without demaging.

The Ag higher also has the ability to reflect completely the UV light for wavelength more than 400 Å, but not lower than it. Concerning to the limitation of ability of Ag to reflect the beam of light for wavelength less than it, Thomas and Wolve has revealed that by coating Ag film above Al, the reflectance will be high though for wavelength decrease down to 200 nm [1]. For increasing durability of Ag, sandwich method is proposed, in which Ag film is wedged by two NiCr<sub>x</sub> films.

Copper (Cu) is a suitable material for substrate to form Cu/Ag film. With Cu substrate,

durability of Ag film can be increased, which is needed especially for reflective recording film. Beside that, the 0.03-0.05 mm Ag above the Cu film can protect Cu film.

In the fabrication of Cu/Ag film with electroplating technique, the plating bath temperature influence the solubility of solvent, that is increasing solubility of plating bath. The more liquid the plating bath, the easier the migration of ions formed on cathode to anode. Due this the fact, the increasing plating bath temperature will accelerate process of formation of film in cathode. The rate of film formation influence the ordering of crystalline structure of film, which will affect the appearance of Ag film.

This paper, it is presented the results of the study about the effect or KCl plating bath temperature on the reflectance of Cu/Ag thin film. To complete the information, characterization of the sheet resistivity and the ordering of crystalline structure as a function of plating bath temperature, where each of them was done by FPP (four point probe) and XRD (X-ray diffractometer) equipments. This research is a progress to the Cu/Ag research done before, which is carried out by varying



the deposition time 7 minutes is the most optimum deposition times for resulting the highest reflectance, that is 94,20% [3].

## EXPERIMENTAL METHOD

### Material

The materials used in this research including: copper plate as substrate, silver plate as coating material with size of 3 cm x 1 cm x 0,04 cm and Kalium Clorida (KCl, potasium) as plating bath.

### Instrument

The instrument used in this research includes: ultrasonic cleaner 60 Hz for washing the surface sample, electroplating machine with regulators for electrode distance, electrode voltage, and solution temperature controller. For characterization of the sample, the following were used: XRD spectrometer to observe microstructural of Cu/Ag film, four point probes to obtain sheet resistivity of sample, UV vis spectrometer with wave length set from 390 to 780 nm.

### Procedure of Research

#### Preparation of Cu Substrate

Process for preparing Cu substrate [24] lude: Cutting the copper (Cu) plate with size of 2.5 cm x 1 cm x 0.002 cm. Then refining the surface of Cu with abrasive paper number 300, 900 and finished with brasso. The next step is washing the samples with detergent in the ultrasonic cleaner for 10 minutes, 40°C temperature, and with distilled water during 10 minutes, and finished with alcohol 95% for of 10 minutes.

#### Deposition Process

[11] Deposition process was done with following procedure:

- Filling the bath with plating bath of potasium with concentration of 10 gram/liter
- Putting the Cu plate on the cathode and Ag on the anode
- Connecting the two electrodes on the 5 volt DC power supply.

- Heating the plating bath until temperature of 60°C.
- Operating deposition process during 7 minutes, which is divided in to two stages: the first is the deposition in 3 minutes, and the second is deposition for 4 minutes
- Repeating of (e) for various temperatures of plating bath, that are 70, 80 and 90 °C.

## RESULTS AND DISCUSSION

In the part below, the results from several characterizations of Cu/Ag including XRD spectrum, sheet resistivity and reflectance of Cu/Ag are discussed.

### XRD Spectrum of Cu/Ag Film at the Various of KCl Plating Bath Temperature

Considering the 7 minutes optimum deposition time of Ag that produced the highest reflectance as mentioned in the research before, the temperature of KCl plating bath is varied from 60 to 90 °C. The XRD spectrum for these is displayed in Figure 1.

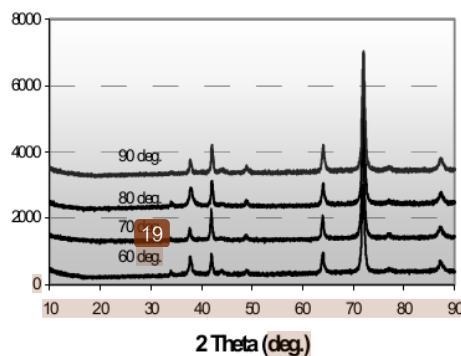
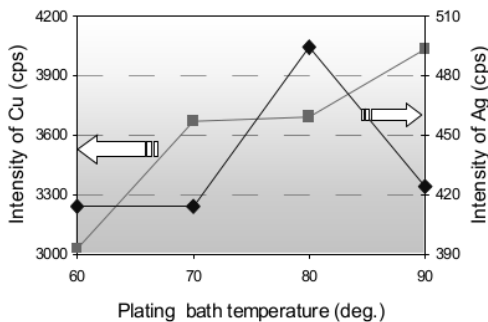


Figure 1. Spectrum of XRD for Cu/Ag film as function of plating bath temperature from 60 to 90 °C.

From the figure it appears that all of the samples show the diffraction peaks. This indicates that the microstructure of film is crystal. The positions of 2 theta angle for Ag are at approximately 34° and at about 72° for the dominant peaks of Cu. The change of plating bath temperature determine intensity of diffraction peaks, and quantitatively is loaded in Table 1. Figure 2 shows the graph of intensity

**Table 1.** The diffraction angles and intensity for Ag dan Cu for various plating bath temperature.

Plating bath temperature (°C)	Ag		Cu	
	angle	intensity	angle	intensity
60	33,94	414	71,94	3026
70	33,98	414	71,94	3672
80	34,00	494	72,04	3690
90	34,04	424	72,06	4034



**Fig. 21.** 2. Intensity of XRD pattern of Cu/Ag as function of plating bath temperature for Cu and Ag.

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as function of plating bath temperature for Cu/Ag and Cu samples.

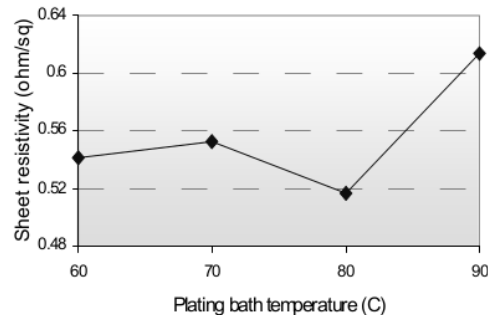
From the figure it is shown that at the KCl plating bath temperature of 80 °C, the Ag film has the highest intensity (494 cps) among the other samples. It means that at the temperature of 80 °C, the Ag film has the highest ordering crystalline structure. Mean while, the intensity of Cu film is 3690 cps, that is the second order after Cu film resulted in the KCl temperature of 90 °C (4034 cps). It is difficult to produce the highest crystalline structure for both Ag and Cu films simultaneously. The difference of lattice constant of the two elements is the reason of it difficulty. The fact shows that temperature of plating bath affect to the microstructure of surface of Cu substrate. This is the opposite with Khakpor and Hadi S<sup>3</sup>aloni statement, who expressed that the substrate temperatures on the microstructure of thin metallic films is well established[4].

The lowest intensity for Ag is 414 cps occurred in the 60 °C KCl temperatures. As

explained in the introduction, the purpose of heating the plating bath is to supply thermal energy on the KCl molecule or atoms, so that the molecular/atomic distance between them become larger. As a consequence, the concentration of plating bath is decreased under this condition, Ag<sup>+</sup> ions produced in the anode are easily mobilized toward the cathode to coat the Cu plate. If temperature of plating bath is so high, the movement of ion is uncontrollable and the deposition rate is unstable. If this occurs, the microstructure of the film is disordered. By the fact, the optimum of plating bath temperature to produce the highest order of crystalline structure is 80°C. The rising temperature up to 90 °C causes the decrease of the ordering of crystalline structure.

### Sheet Resistivity of Cu/Ag Film

In Figure 3, it is displayed graph of sheet of resistivity ( $R_s$ ) of Cu/Ag as a function of temperature of plating bath. It appears that  $R_s$  is varied with temperature of plating bath. The lowest  $R_s$  is 0.52 Ω/sq, that is according to the 80 °C plating bath temperature.



**Figure 3.** Sheet resistivity of Cu/Ag film as function of various plating bath temperature.

The low  $R_s$  indicates the flow of conduction electron from probe 1 to probe 4 is only meeting the little impedance. It is caused by the high ordering of crystalline structure of Ag. It is the opposite from Ag film produced at 90° plating bath temperature, that has the highest sheet resistivity i.e. 0.61 Ω/sq which is caused by the less ordering of crystalline structure, that the conduction electron find more impedance at the time they pass in the film.

### Reflectance of Cu/Ag Film for the Various Plating Bath Temperature

In the part below, the reflectance ( $R$ ) of Cu/Ag film characterized by UV-vis spectrometer on the wavelength from 390 to 780 nm (from UV to red) were displayed.

From the figure, it appears that Cu/Ag film produced in the plating bath temperature of 60 °C (sample 1) has the highest reflectance, that is about 98 %. It means that all of the beam of light which arrives at the surface is almost completely reflected at the range of wavelength from 490 to 790 nm (from yellow to red). While Cu/Ag film resulted at 90 °C temperature (sample 2) has the same reflectance relatively for all wavelengths that is 96 %. The lowest reflectance was found for Cu/Ag resulted at plating bath temperature of 70 °C; that is about 85 %. The sample 1 and 2, each of them have excellent reflectively, respectively, depending on the range of wavelength that is used.

*Figure 4.* Reflectance of Ag film as function of various plating bath temperature

### CONCLUSION

Temperature of plating bath influences the quality of Cu/Ag film including reflectance, sheet resistance and microstructure. The Cu/Ag film produced at temperature of KCl plating bath of 80°C has the highest reflectance (98%) in the wavelength ranged from 490 to 790 nm. The film displays good enough ordering of the crystalline structure, and low sheet resistance. The film produced at 80°C plating bath temperature shows the highest ordering crystalline structure and the lowest sheet resistivity.

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