**Project Number: 234920**

**Project Acronym: NANOSOLD**

**Project Full Name: A Chemical Approach to Lead-free Nanosolders**

**Marie Curie Actions**

**Return phase Final Report**

**Period covered: from 01.11.2012 to 31.10.2013**

**Period number: 1**

**Start date of project: 01.11.2012**

**Project beneficiary: Dr. Ratikanta Mishra**

**Project beneficiary organization name: Bhabha Atomic Research Centre, Mumbay, India**

**Date of preparation: 15.032014**

**Date of submission (SESAM): 28.10.2011**

**Duration: 12 months**

**Version: 1**

**Nanosold – A Chemical Approach to Nano Solder**

In the project **“**Nanosold-A Chemical Approach to Nano Solder**”** it wasproposed to investigate the possibility of using Sn-Sb-M alloys (M = Ag, Cu, Ni) as high-temperature solders and to select compositions that would fulfill the necessary temperature requirements.

In the main phase we have carried out mainly phase diagram and thermodynamic investigations of Ni-Sn-Sb system. It was also proposed to form the basis for a CALPHAD type thermodynamic assessment of the systems.

In the return phase we have evaluated the experimental results and phase diagram calculations of Ni-Sn-Sb system carried during the main phase. We have also extended activities on the synthesis and characterization of Cu-Sn-Sb nano alloys. We present here evaluation results together with synthesis and characterization of Cu-Sn-Sb alloys.

**Work Program**

1. **Evaluation of experimental result:**

The experiments carried out at the host’s institute i.e Prof. Herbert Ipser’s lab at the Department of Inorganic and Materials Chemistry, University of Vienna, Austria have been evaluated and the experimental results have been published in international journals. The list of the publications from the above work is given below.

1. Phase Equilibria in the Ternary Ni-Sb-Sn System: Experiments and Calculations

Ales Kroupa, Ratikanta Mishra, Divakar Rajamohan, Hans Flandorfer, Andrew Watson, Herbert Ipser

CALPHAD: Computer Coupling of Phase Diagrams and Thermochemistry

45(2014) 151–166

2. Phase Equilibria in the Sn-rich corner of the Ni-Sb-Sn system

R. Mishra, A. Kroupa, A. Zemanova, and H. Ipser

J. Electronic Materials 42 (2013) 646-653

3. Thermochemistry of Liquid Ni-Sb-Sn Alloys

R. Mishra, A. Kroupa, P. Terzieff and H. Ipser

Thermochimica Acta 536 (2012), 68-73

4. Synthesis and characterization of Sn-rich Ni–Sb–Sn nanosolders

R. Mishra, A. Zemanova, A. Kroupa, H. Flandorfer, H. Ipser

J. Alloys and Comp. 513 (2012) 224– 229

1. **CALPHAD-type optimization of bulk Sn-Sb-M**

The optimization of phase diagrams of Ni-Sn-Sb system has been carried out with the help of Dr. Ales Kroupa of Czeck Republic. The results of the optimization work was recently published Ref. [1]. Further, we are in the process of implementing the optimization program in our institute. The results of the optimization of other ternary i.e. Cu-Sb-Sn and Ag-Sb-Sn systems will be published later.

1. **Final key experiments to improve CALPHAD modeling**

The available thermodynamic data on binary and ternary compounds Ag-Sn-Sb and Cu-Sn-Sb systems are being evaluated. The cases where the corresponding Gibbs energy data for the compounds of the above systems are not available are being measured employing solution calorimetric techniques. From the combined results of the standard enthalpy of formation and heat capacity of alloys, the values of Gibbs energy of formation will be derived. which will be used to further improve CALPHAD modeling.

1. **Synthesis of Sn-Sb-Cu Nano Alloys**

Nano particles of pure Sn, binary Sn-rich ternary Cu-Sb-Sn alloys (with compositions ranging from 80 to 97.5 at% Sn and a Ni to Sn molar ratio of 1:1) were synthesized by the reduction of stoichiometric metal chloride solutions with NaBH4 at 0°C in alkaline medium. Appropriate volumes of SnCl2 (0.08 M), SbCl3 (0.03 M), and NiCl2 (0.06 M) were mixed. To the resulting solutions required amounts of a 1 M solution of tri-sodium citrate was added as a chelating reagent. Another 0.5 M NaBH4 stock solution was prepared at a pH value of 14 from a 4.4 M NaBH4 stable solution. The two separate aqueous solutions were cooled to 0°C in ice water before reaction. 40 ml of metal chloride solution was added drop-wise to 40 ml of the NaBH4 solution under strong magnetic stirring within 10 min, and the mixed solutions were stirred for another 30 min. The resultant suspensions were separated by centrifuge and rinsed several times with distilled water and acetone and vacuum dried and characterized. Alloy powders with different particle sizes were prepared by annealing individual parts of a single alloy in evacuated quartz ampoules at 180°C for different lengths of time. The fine black alloy powders were characterized by X-ray diffraction (XRD), differential scanning calorimetry (DSC), and electron microscopic techniques.

Dr. Ratikant Mishra

(Project Beneficiary)

Through:

Dr. S. R. Bharadwaj,

Scientist –in-charge