# Abstract

Eutectic Sn-Cu lead free solder was synthesized by pulse electrodeposition from tri-ammonium citrate bath. Current density was varied from 0.1 to 0.5 $A/cm^2$ to evaluate its effect on the characteristics of the deposit. Optimization for the eutectic bath was carried out with different concentration of $CuCl_2.2H_2O$ keeping $SnCl_2.2H_2O$ concentration and other parameters (current density, $t_{on}$ and $t_{off}$ time) the same. Characterization techniques like SEM, XRD, DSC, and AAS were used to confirm whether the deposit was indeed eutectic. The coating deposited at 0.2 $A/cm^2$ from bath having higher amount of $CuCl_2.2H_2O$ is found to be lamellar eutectic consisting of Sn and $Cu_6Sn_5$. The EDS results show the traces of $Cu_6Sn_5$ in the electrodeposits. This microstructure is more or less same as that of conventional cast structure of eutectic Sn-Cu solder. However, in pulse electrodeposition the morphology resembles a pseudo lamellar structure. This is a result of an illusion giving the appearance of a lamellar structure which in reality is the top view of the under potential deposits of $Cu_6Sn_5$ on the steps of Sn. The roughness studies show that with increasing current density the roughness of the surface increases. The induced roughness is a result of the shape and size of the crystals that form. Growth behavior of the deposits was studied through 3D crystal shapes. The shapes of the crystals were reconstructed using SHAPE V7.3 software. It is found that with increasing current density the growth direction shifted from <001> to <110>.

$Y_2O_3$ was synthesized using sol-gel assisted combustion synthesis route. The size lies in the range of ~30 nm. It is a new method of sol-gel combustion process where there is no need of primary heating to 500 °C. Sn-Cu-$Y_2O_3$ nanocomposite lead free solder was deposited form the optimized eutectic bath with additional constituents as CTAB and $Y_2O_3$. Different concentrations of CTAB (0.02, 1, 4, 6 l, and 8 g/l) and $Y_2O_3$ (1, 5, and 10 g/l) were used for electrodeposition of Sn-Cu-$Y_2O_3$ nanocomposite solder. The best deposit having homogeneous or uniform dispersion of $Y_2O_3$ in Sn-Cu matrix is obtained from the bath containing 1g/l CTAB and 1g/l $Y_2O_3$.

Creep behavior of the deposited film (deposited from the bath with higher concentration of $CuCl_2.2H_2O$) was evaluated by nanoindentation technique. Tests were carried out at 500 µN load for a dwell time of 900 sec. Modified Garofalo Equation of creep was used to describe the depth versus time curve. Creep resistance of thin film deposited at lower (0.2 $A/cm^2$) current density is found to be less. Incorporation of $Y_2O_3$ in the deposit helps in decreasing the creep rate from 3.64E-04 to 7.03E-05 $sec^{-1}$. $Y_2O_3$ particles act as obstacles in the path of dislocation motion and inhibit grain boundary sliding.

Eutectic Sn-0.7 wt% Cu was also synthesized by conventional melting and casting route. Different concentrations of $Y_2O_3$ were used for the synthesis of nanocomposite solder. Casting followed by repeated forging was used for the synthesis of nanocomposite solder. This helps the solid state mixing of $Y_2O_3$ in Sn-Cu eutectic. The optimum number of forging steps was determined. Impression creep tests were carried out at different combinations of loads (28, 55, and 83 MPa) and temperatures (298, 343, and 373 K). Nanocomposite solder shows higher creep resistance as compared to the monolithic solder. Creep rate shows a decreasing trend with the addition of higher amounts of $Y_2O_3$ in the solder. Nanoindentation creep test also reveals the same trend.

Nanoindentation creep tests at room temperature suggest that pulse electrodeposited solder has higher creep resistance in comparison to that made by conventional melting and casting route. This may be due to any one or a combination of the following three reasons: (i) the difference in their microstructures, (ii) the difference in the texture or the orientation of Sn, and (iii) the difference in the ways $Cu_6Sn_5$ forms and get distributed in the matrix during processing or synthesis.

***Keywords:*** Lead free solder; nanocomposite; nanoindentation creep; accumulative forging; impression creep.