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学 位 論 文 内 容 の 要 旨

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学 位 論 文 題 名

Process development for synthesizing core-shell nanoparticles with liquid cores utilizing dusty plasma techniques

(液体コアを有するコア・シェルナノ粒子のダストプラズマを用いた合成プロセスの開発)

Core-shell nanoparticles synthesized by depositing solid shells around liquid cores are useful as a reusable latent heat storage material. The core-shell type phase changing materials with liquid cores can actively address the increasing demand for thermal energy and enhance the overall energy efficiency. However, it is challenging to deposit thin films around liquid cores with the available process technologies.

This research work demonstrates a process flow to deposit amorphous carbon films around liquid tin nanodroplets retained in a capacitively coupled plasma (CCP). High-pressure magnetron sputtering was used for synthesizing tin nanoparticles from a tin target placed at the top of a vacuum chamber. Tin nanoparticles were transported to CCP and trapped above an rf electrode at the bottom of the chamber. Tin nanoparticles were heated above the melting point by ion bombardment in CCP, then we deposited amorphous carbon films as the shells using plasma-enhanced chemical vapor deposition (PECVD). Core-shell nanoparticles thus synthesized were collected by applying a positive bias voltage to an electrode placed inside the rf electrode. It was observed that the electrode potential was higher than the plasma potential while supplying a bias potential of +70 V, which enabled the efficient transportation of nanoparticles. The nanoparticles were analyzed using electron microscopy (SEM, TEM), energy dispersive spectroscopy and electron diffraction. TEM images of core-shell nanoparticles revealed that the cores were perfectly spherical. The temperature of nanoparticles retained in CCP was theoretically estimated, and it was confirmed that the temperature was above the melting point of tin. Further analyzed the nanoparticles using in-situ heating in TEM and observed the phase change in tin core at the melting point. This result also suggests that the amorphous carbon films protected cores from the oxidation. In addition, the breakdown of shells was not observed at the melting of cores, indicating that the amorphous carbon films were robust against the volume expansion of the cores.

The first chapter gives the general introduction.

The second chapter describes the principles underlying the core-shell nanoparticles generation, the growth, and the retention. The discussion is started on fundamentals of plasma and its features, fol-

lowed by principles and comparison of CVD and PECVD. The physics of plasmas used in this work, i.e., CCP and DC magnetron sputtering are discussed. Further, the mechanism of the nanoparticle growth and the physics of their charging in plasmas are described. Particle retention is directly connected with the balance among forces acting on nanoparticles in the plasma, hence discussed about the relevant forces and compared their magnitudes.

The third chapter describes the experimental methods used in this work. The equipment used for synthesizing core-shell nanoparticles was composed of CCP and magnetron sputtering. The later part of this chapter discusses the techniques used to measure the electron temperature, the ion density, the floating potential, and the plasma potential, followed by nanoparticle analysis techniques and additional plasma diagnostic methods.

The fourth chapter describes the results in chronological way from the production to the collection, the surface morphologies to the atomic composition analysis, followed by the gas temperature measurement, and the analysis of the shell robustness against core melting. The particle growth and transportation to the collection electrode are observed from the scattered laser light images captured using an electron multiplying charge-coupled device camera. The atomic composition analysis was performed using TEM attached with an EDS tool. The melting observations was performed using TEM equipped with a substrate heating system and an electron diffraction system.

The fifth chapter discusses the theoretical estimation of the nanoparticle temperature, the production mechanisms of tin clusters observed in melting experiments, followed by discussion on the particle collection. The nanoparticle temperature was calculated using heat transfer theories from literatures while considering the experimental findings on the gas temperature, the electron temperature, the plasma density, and the plasma potential.

The sixth chapter gives major conclusions of this work followed by the outlook.