【別紙２】

論文名 : Study on fabrication of submicron particles, surface treatment of nano-sized inorganic particles, and new classification system for anisotropic conductive film（要約）

新潟大学大学院自然科学研究科（論文博士は氏名のみでも可）
氏名　高井健次

（以下要約を記入する）

Anisotropic conductive film (ACF) is used as the adhesive to the desired interconnection between the glass substrate of flat panel display (FPD) and the driver IC. The conductive particles in ACF are made up of micron-sized polymer spheres plated with a thin layer of nickel and gold followed by submicron-sized insulation polymer particles.

ACF is being compressed thermally, the conductive particles between the connecting bumps and pads are sandwiched and would change from being sphere to oval. At this stage, insulation particles of the conductive particles wear out and electrical connections are formed in the z-direction. The other conductive particles remain as usual with insulation particles and prevent electrical conduction in the x-and y-direction.

Usually micron-sized polymer particles are made by seed polymerization. Submicron-sized polymers are commonly synthesized by soap-free emulsion polymerization of acrylic monomers or styrene monomers. Ionic initiators like potassium persulfate are used for soap-free emulsion polymerization. Particles made by soap-free emulsion polymerization have hydrophilic surface because of initiator.

In addition, the development of adhesives now requires not only the development of new resins, but also the design of surface-modified inorganic particles. Surface-modified nano-sized silica is usually used to control the viscoelasticity of films. In general, dispersing silica nanoparticles uniformly into a polymer or an organic solvent is very difficult because of aggregation of these particles.

The micron-sized polymer particles and the submicron-sized polymer particles should be monodispersed. Classification technology is one of the most important technologies for ACF. Centrifugation, sedimentation and wet sieve process are used for the classification of polymer particles.

In the thesis, the purpose of solving the problem mentioned above, (1) the preparation of hydrophilic poly(dimethylsiloxane) (PDMS) micron-size particles, (2) the grafting PDMS onto silica nanoparticle surface, (3) the development of vertical type water sieve with electro-potential, and (4) development of a new method of zeta-potential measurement by use of the liquid sedimentation balance were investigated.

In Chapter 1, background of ACF film and the purpose of the study were summarized.

In Chapter 2, the preparation of monodisperse hydrophobic particles in the submicron size was investigated. Dimethyldimethoxysilane (DMDMS), methyltrimethoxysilane (MTMOS), and tetraalkoxysilanes, such as tetramethoxysilane (TMOS) and tetraethoxysilane (TEOS), were used as monomers for synthesizing the hydrophobicsilicone particles. By optimizing the alkoxysilane concentration, the blending ratio of monomers, the amount of catalyst and the reaction temperature, the preparation of small monodispersed silicone particles was successfully achieved.

In Chapter 3, for the purpose of the prevention of environmental pollution and the simplification of reaction process, a dry surface treatment system and carefully controlled synthesis of oligomeric siloxane (*oligo*-SiO) was investigated. The grafting of the *oligo*-SiOs onto the silica surface was successfully achieved. The *oligo*-SiOs made by both bifunctional silane and tetrafunctional silane contained many hydroxyl groups that led to the higher chemical grafting efficiency. By grafting of *oligo*-SiO containing moderate number of phenyl groups onto the surface, the silica was easily dispersed in mixed solvent of toluene/ethyl acetate. In addition, the *oligo*-SiO-grafted silica dispersed in a composite polyimide film had excellent adhesion ability to a silicon chip.

In Chapter 4, in order to classify fine particles in slurry flow, vertical type water sieve with electro-potential applied to flow is newly developed. The 50% cut size decreases with the increase of under-flow rate of coarse side. The maximum particle size in the over-flow side decreases as the

over-flow rate decreases. By use of the electro potential applied to the upper and lower plates, the 50% cut size decreases with the increase of applied potential. The minimum cut size of about 0.62μm was obtained from the experimental data. The accuracy of classification also increases with the increase of applied potential. The experimental data of cut size agreed with the calculation results based on gravity and electro migration effect in particle sedimentation velocity. The experimental data of cut size agreed with the calculation results based on gravity and electro migration effect in particle sedimentation velocity.

In Chapter 5, a new method of zeta-potential measurement by use of the liquid sedimentation balance method is developed. The test samples used were spherical acrylic and silica particles, and beads mill was used to enhance particle dispersion. The new method can be used to estimate the zeta potential of each particle size range. The absolute value of the negative zeta-potential of small particles is greater than that of large particles. The results of measuring zeta-potential by the new method agree with that of conventional measurement devices. The slope of the zeta-potential with respect to particle diameter decreases as the waiting period following the beads mill pre-treatment process increases.

In Chapter 6, a summary of the results of this study was described.