Chapter 5

Conclusions

5.1 Powder Processing and Characterization

The PZT powder used as the ceramic filler material of the 0-3 piezoceramic-polymer composites was prepared by the solid sate reaction route [$Pb(Zr_{0.52}Ti_{0.48})O_3$] and sprayed dry commercial 40/30 powder [$Pb(Zr_{0.45}Ti_{0.45})(Mn_{0.03}Nd_{0.06})O_3$].

The X-ray diffractrograms of (100) and (001) peak indicate that transformation of the cubic to the tetragonal phase starts at 1125 °C which have the c/a value of 1.009. After annealing at various temperatures, it was found that the particle sizes of the powder were rapidly increased at annealing temperatures above 900 °C while the microstrain showed decreasing. Measurements were carried using the Fourier analysis and SEM. The particle sizes were about 0.02-0.10 μm when measured by Fourier method and 0.5-1.7 μm by SEM. However, the magnification of SEM shows that the particles of PZT powder form granule growth which consists of many small particles packing together.

The PZT powder both prepared by the mixed oxide method and commercial 40/30 PZT powder used as the dissolve phase in 0-3 composites were calcined at temperature of 1200 °C and sieved through 100 and 300 mesh. For own PZT powder, the particle sizes were measured to be 155 and 62 µm from X-ray sedigraph data and 150 and 55 µm from SEM micrographs, respectively. For commercial 40/30 PZT powder, the particle sizes were measured to be 159 and 55 µm from sedigraph data and 150 and 55 µm from SEM micrographs, respectively.

5.2 Composite Processing and Characterization

The 0-3 piezoceramic-polymer composites with 10, 20, 30, 40, 50 and 60 volumetric fraction of ceramic filler were prepared by the conventional method and 40, 50, 60 and 65 vol% ceramic filler were prepared by the centrifuge method. Polyethylene and commercial 40/30 PZT powder were used as matrix phase and dissolved phase in conventional composites, respectively. In the centrifuge method, the polyester resin and both own PZT and 40/30 PZT powder were used. Scanning electron micrographs show the distribution of the PZT powder in composites that the fabrication by the centrifuging method indicates a considerably better distribution of ceramic particles in the polymer phases. This result also leads to the increase in the properties of the composites. Furthermore, it can be seen that the composites fabricated by spinning in a centrifuge could load 65 vol% of PZT particle which could not be achieved in the conventional calendering method.

5.3 Composite Density

The density of the composites produced by both methods (calendering and centrifuge) were varied linearly as volumetric percentaged of PZT. The density increases with increasing of vol%. This correspond to equation (3.18)

$$\rho_{composites} = (vol\%_{ceramic}) \cdot \rho_{ceramic} + (vol\%_{polymer}) \cdot \rho_{polymer}$$

5.4 Dielectric Properties of Composites

The dielectric constant (ε_r) and loss angle (tanδ) of all composites were measured in the frequency range of 100-20,000 Hz. Both both ε_r and tanδ were found to decrease slowly with increasing of the frequency. At the constant frequency (at 1 kHz), the ε_r increased when the volumetric fraction of PZT ceramic filler increased. The tanδ was found to increase slowly with increasing of the volumetric fraction of PZT ceramic filler. Dielectric constant of 60 vol% composites prepared by the conventional method and the centrifuge method both using 40/30 and own PZT, were measured to be 62, 59 and 84, respectively, and with tanδ of 0.040, 0.014 and 0.014, respectively. The highest volumetric percentage of ceramic filler in composite in this work was 65 vol%, which was successfully carried out in the centrifuge method. The dielectric constants are 62 and 88 and tanδ are 0.043 and 0.035, when ceramics filler are own PZT and commercial 40/30 PZT powder, respectively. These are comparable to the values measured by Hanner et. al.[1] and Fries and Moulson[2].

5.5 Piezoelectric Properties of Composites

The piezoelectric charge coefficient and piezoelectric voltage coefficient of all samples were measured after poling in the same conditions. The values of d_{33} and g_{33} measured from composites prepared by centrifuge method were higher than those of the composites prepared by the conventional method. These may be due to the better distribution of ceramic phase. This can also lead to obtain higher dielectric constant in the composites prepared by the centrifuge method. The highest d_{33} value (29 pC/N) was found from 65 vol% polyester resin and mixed oxide PZT ceramic composite fabricated by the centrifuge method. These piezoelectric properties are comparable to the values measured by Hanner et. al.[1], Fries and Moulson[2] and Cai et. al.[3], who used different preparation methods. However, these are less than the composites poled by corona discharge technique[4].

5.6 Electromechanical Properties of Composites

In this work, the coupling factor and mechanical quality factor measured from the mixed oxide PZT powder and resin composite were very close to the 40/30 PZT powder and resin composite. The coupling factor found to be 0.33-0.45 while the mechanical quality factor found to be 4.4-9.9. These were comparable to those of others[4,5,6]. However, it cannot be achieved in composite prepared by the conventional method.

5.7 Acoustic Impedance of Composites

The acoustic impedance was measured from Equation (3.25),

$$Z = c_L\rho = (K\rho)^{0.5}$$

Where the longitudinal velocity (c_L) was measured by the echo-shift technique and the density (ρ) measured by the Archimedes method. The values of Z are in the range of 6.80-11.41 Mrayls for mixed oxide PZT composites and 7.43-

12.43 Mrayls for composites prepared by the centrifuge method. The results are comparable to those of others[5,6,7]. These data indicate that the composites may be appropriate to be employed as the transducer in water or human body which have low acoustic impedance about 1.5 Mrayls while that of pure PZT ceramic is about 30 Mrayls.

5.8 Suggestion for Future Work

In the present study, the dielectric and piezoelectric properties of 0-3 ceramic-polymer composites were mainly dependent on the poling process, percentage of the ceramic filler and the fabrication route of the composites. From this study, the following suggestions are recommended for the future work on the piezoelectric 0-3 ceramic-polymer composites.

1. The preparation of 0-3 composite by centrifuge method could lead to a high percentage of the filler which would result in high piezoelectric properties. Also smaller particles could improve the poling efficiency. Therefore, fine powder of PZT and fabrication of the

composites by the centrifuge method should be carried out in the attempt to obtain high piezoelectricity.

2. Low density, high permittivity and good adhesive polymer should be employed as the matrix phase since these properties could enhance the energy transfer and reduce the loss factor.

3. The corona discharge method should be employed in the poling process.

4. The experiment on the application of the (0-3) piezoceramic-polymer composites on the human bodies should be carried out. However, this requires high performance electronic equipment of high gain, low noise and so on.