Paper

Optical Polarization Technique for Observing Filler Packing-structure in the Ceramic-powder Filled Resin Polymer Composite Systems

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This paper offered a polarization in transparent optical microscopy as one of observing technique for internal-structure (powder packing-structure) of the resin polymer composite system filled with ceramic filler-powder. The technique visualized the heterogeneous powder packing as brightened features at crossed-polarization, and contributed to clarify the influence of particle size distribution and coupling treatment of filler-powder on internal microstructure of the polymer composite. The brightened features were appeared to have an optical anisotropic property; i.e., repeated changes between bright/dark under rotating specimens at every 45° increments, optical addition/subtraction retardations and positive optical character of elongation under observation using tint plate. The optical anisotropy was often observed in the filler systems with narrow size distribution and the systems under coupling treatment. The amounts of optical anisotropy showed the good agreement with rheological properties in systems, although they could not be understood only from filler primary properties; such as median diameters, particle size distributions and specific surface areas.

Keywords : Filler-powder packing-structure, Optical polarization, Rheological property, Particle size distribution, Coupling treatment

1. Introduction

An attempt was done to provide the detection of filler packing-structure and its link to viscosity for the resin polymer composite system filled with ceramic powder. The polymer composite is one of the important material systems for several recent electrical devices; i.e., a powder-polymer conductive paint [1]; an insulating spacer for gas-insulated switchgear (GIS) [2]; an electrorheological fluid [3]; a chemical mechanical planarization (CMP) buffing compound [4]; a semiconductor packaging material for high-fidelity VLSI electric devices, such as chip size package to under-fill the flip chip solder joints [5, 6]. The packaging material encapsulates IC and other components (likely as resister and capacitor) to disconnect electrically and reinforce mechanically, chemically. The material was initially made from ceramics in the 1960's, and eventually from resin polymer composite filled with silica (SiO_2) filler-powder. It necessitates a high thermal conductivity, a low thermal expansion and a good moldability. Higher the packing content of filler, higher the thermal conductivity (lower the thermal expansion); but then, the moldability degrades. There is a yearning demand, especially, how to mold the material into the new integrated devices such as the System in Package [7-9].

Detection of filler packing-structure is one of promising approach to improve the packaging properties. Presently, there were some empirical studies of relationship between moldability and filler primary properties, i.e., particle size distribution, particulate surface chemical/ structural contributions [10-14]. Or, theoretical prediction of filler packing-structure was presented based on a shear-thinning to effective volume fraction of silica flocculation and/or a flow-induced change in inter-particle interaction [15-19]. Linkage study is necessary for connecting the former empirical works and the later theoretical ones, but it was difficult to observe directly the filler packing-structure; because of the lack of appropriate observation method for this kind of material system having the liquid typed-matrix of resin polymer.

Figure 1 showed our optical microscopic technique to detect filler packing-structure [20]. The method visualized the influence of particulate surface structural contributions, and enabled the rheological property control [21, 22]. This paper concerned with the particle size distribution and coupling surface treatment as one of more practical conditions for filler-powder.

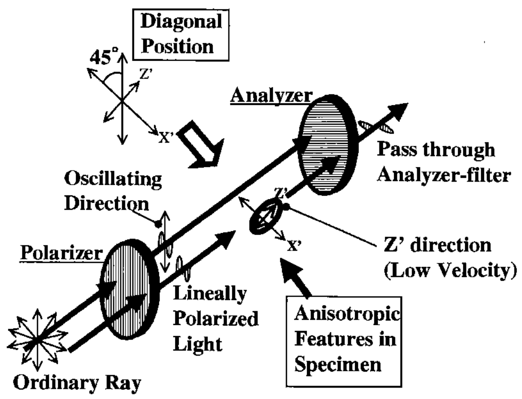

Fig.1 Optical polarized detection of filler packing-structure in resin polymer composite system filled with ceramic particles

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2. Experimental

2.1 Sample preparation Typical commercialized amorphous spherical SiO_2 particles prepared by flame pyrolysis, 6.6, 17 and 27 μm in median diameters were used as a filler particle (**Fig.2**). Coupling treatment of filler is used for several reasons such as lowering viscosity, reinforcing interaction of filler-powder and resin solvent. This paper examined the later matter with using 17 μm filler-powder, epoxy-system silane coupling agent and epoxy resin solvent. Surface treatment was carried out similarly in previous report [23]. Organosilane, 3-glycidoxy- propyltrimethoxysilane, KBM-403 Shinetsu Kagaku Kogyo Co. Ltd., was used for coupling agent. The amounts of coupling agent added to filler-powder were set to the ratio of SiO_2 total specific surface area, powder weight and coupling agent coating capability. Bisphenol-A type epoxy resin, RE-310S Nihon Kayaku Co. Ltd., was utilized as solvent for the composite system. The particles were filled into the epoxy resin with a solid loading of 70 mass%, which typified the constituent of recent electrical devices. The composite system was mixed with AR-360M, Shinky Co. Ltd. for 5 minutes at 1800 and 600 rpm for revolution and rotation speeds, respectively.

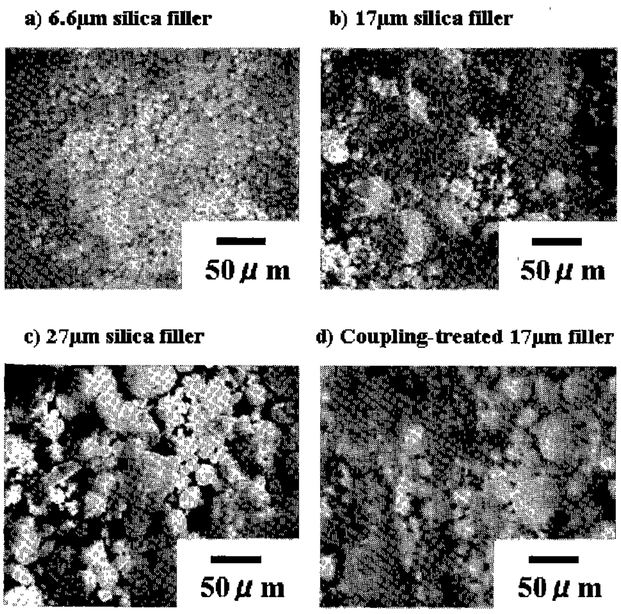
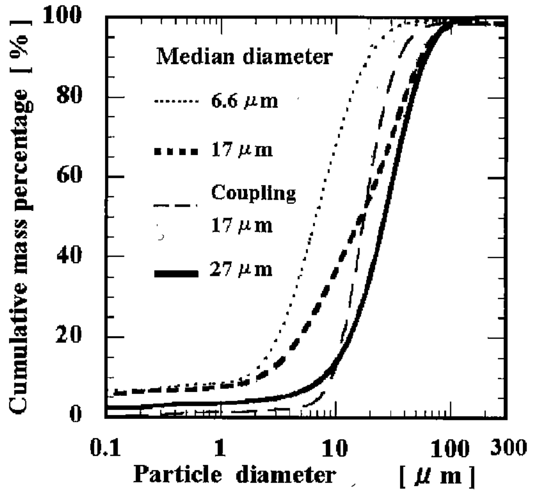


Fig.2 Scanning electron micrographs of SiO_2 filler-powder

2.2 Evaluation The SiO_2 particle size distribution in the composite system was measured with an X-ray particle size analyzer (SediGraph 5100, Micrometrics Co.) for the slurry of SiO_2 solid content of 7 mass% in water. Specific surface area of the SiO_2 filler particle was measured by the Brunauer, Emmett, and Teller (BET) method. The internal structure of the epoxy composite system filled with SiO_2 was examined with an optical microscope (Model E600, POL-TP21, Nikon Co. Ltd.) in transmission mode by using both normal and cross-polarized lights. The specimens were thinned to the thickness about 50 μm. Features having specific orientation in the composite system, such as alignment of elongated particle during its molding, could polarize the incident linearly-polarized light, and pass the ray though the analyzer-filter of microscope. Optical anisotropy of the features was confirmed under a crossed polarized light transmission microscope with/without tint plate; i.e., a repeated change between bright and dark with the specimen rotation of every 45° increments, an addition/ subtraction retardation and a positive/negative optical characters of elongation. The apparent viscosity of the composite systems was measured with a viscometer (VT550, HAAKE Co. Ltd.) at 80 C° with the shear rate varied from 0 to 500 s^{-1}.

3. Results

Figure 3 shows the particle size distributions and specific surface areas of SiO_2 filler-powder of 6.6, 17, 27 μm in median diameters, and those of coupling-treated 17 μm SiO_2. The coupling-treated filler appeared a narrow size distribution, which should be due to the change of particle aggregation. However, the median diameters and specific surface areas are almost the same both for non-treated filler and for treated-filler.



Median diameter [μm]	6.6	17	(Coupling) 17	27
Specific surface area [m^2/g]	4.7	3.8	3.9	1.7

Fig.3 Particle size distributions, median diameters and specific surface areas of filler-powder

Figure 4 shows the apparent viscosity of the resin polymer composite system filled with ceramic filler-powder of various sizes and coupling treatment. The viscosity values of monolithic epoxy resin (without SiO_2 filler-powder) are also illustrated in this figure for comparison. The viscosity values of 6.6 and 27 μm-SiO_2 particle systems were larger than those of 17 μm particle system. Their hysteresis value is the largest among the all filler systems. The hysteresis values of 17 μm particle system are not absent, but smaller than those of the 6.6 and 27 μm particle systems. Clearly, these polymer composite systems have the minimum value in viscosity as against the median diameter of filler particles, and the 6.6 and 27 μm systems had comparatively larger thixotropic rheological characteristics. The minimum viscosity system, the 17 μm particle system, showed enlargement at reinforcing interaction with epoxy solvent. The figure was illustrated with magnifying the ordinate to emphasize the differences of data, although the viscosity had a limited

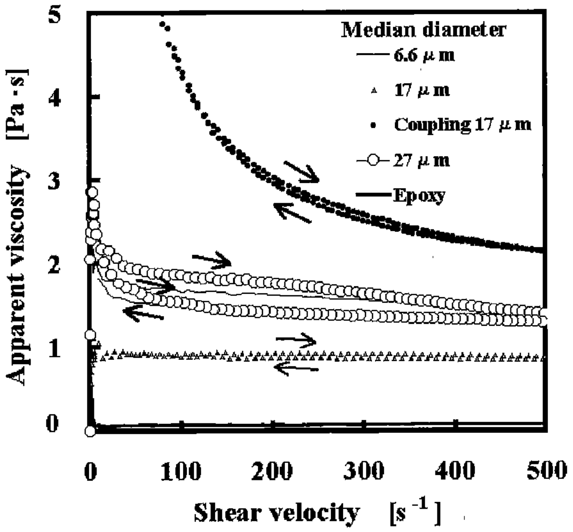
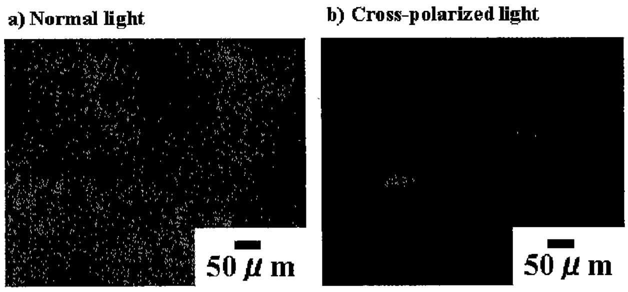


Fig.4 Apparent viscosity of resin polymer composite systems filled with ceramic filler-powder

value at 0 s^{-1} in shear velocity. The hysteresis values were not changed so much both for non-treated filler and for treated-filler.

Figure 5 shows the optical photomicrographs of 27 μm sized SiO_2 filled epoxy resin composite system taken in transmission mode by using both normal and cross-polarized lights with a tint phase plate; a) normal light, b) cross-polarized light and c) cross-polarized light with a tint plate. There are many gray elliptical bodies and their surrounding darker spaces in **Fig. 5-a)**. Each elliptical object was previously identified to each SiO_2 particle or their aggregate, and the darkened spaces were their boundaries [20-22]. The features of elliptical shapes are observed as brightened features at transmission crossed polarization in **Figs. 5-b) and c)**. The features of elliptical shape changed their brightness repeatedly with rotating specimen of every 45° increments. Note that the bodies located close together, simultaneously, changed their brightness with rotating specimen. These bodies had optically the same diagonal and extinction positions, i.e., anisotropic properties. In **Fig. 5-c)**, there are two optically anisotropic features under diagonal positions, which major axes crossed at right angles. One is blue and another is



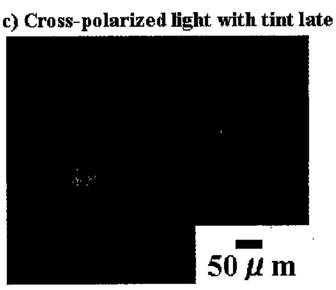


Fig. 5 Optical photomicrographs of 27 μm sized SiO_2 filled epoxy resin composite system taken in transmission mode by using both normal and cross polarized lights with a tint phase plate.

yellow; clearly, the former is optically in addition retardation and the later is in subtraction retardation. The ellipse major axis of the feature in addition retardation (blue) is parallel with the oscillating direction of low velocity ray of tint plate, which ray is made by birefringence. The features of elliptical shape had the positive optical character of elongation.

Figure 6 shows the optical photographs of 6.6, 17 and 27 μm-SiO_2 particle-filled epoxy resin composite systems taken in transmission mode by using normal light. As shown in **Fig. 6-a)**, the elliptical bodies are closely packed in the 6.6 μm particle system compared with the 17 μm system, although its density is not so large compared with the 27 μm-SiO_2. The photograph of the 17 μm particle system (**Fig. 6-b)**) appears that elliptical bodies are poor mostly, and the darkened spaces are rich, definitely as compared to others. Elliptical bodies in the 27 μm system, which sizes are larger than 6.6 and 17 μm systems, appear to be densely filled up (**Fig. 6-c)**). In **Fig. 6-d)**, the coupling-treated 17 μm filler also shows that elliptical bodies appear to be densely filled up. Dark shadowed bodies of 50-100 μm in diameters shown in **Figs. 6-c), d)** were also assigned in the previous work to the larger SiO_2 particle or its rigidly joined aggregate, which was so huge for the focus in the transmission [20-22].

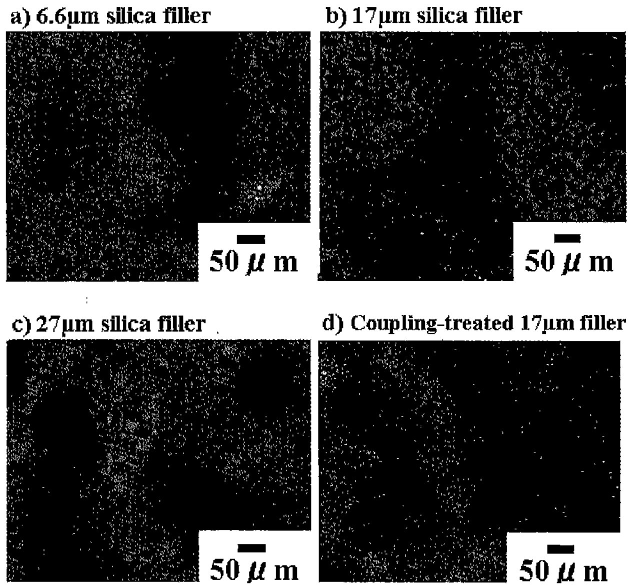


Fig. 6 Optical photographs of the filled epoxy resin composite systems taken in transmission mode by using normal light; a) 6.6 μm diameter filler system, b) 17 μm, c) 27 μm and d) coupling-treated 17 μm system.

Figure 7 shows the optical photographs of 6.6, 17 and 27 μm-SiO_2 particle-filled epoxy resin composite systems taken in transmission mode by using cross-polarized light. Brightened objects, which changed the brightness repeatedly with a rotation of every 45°as already shown in **Fig. 5**, are especially observed in the 6.6, 27 μm particle systems and the coupling-treated 17 μm (**Figs. 7-a), c) and d)**). The 6.6 μm SiO_2 system contains many smaller-brightened objects in the background, although the larger and brighter objects are less comparatively. In **Fig. 7-b)**, the non coupling-treated 17 μm particle system shows that the brightened objects are remarkably minor.

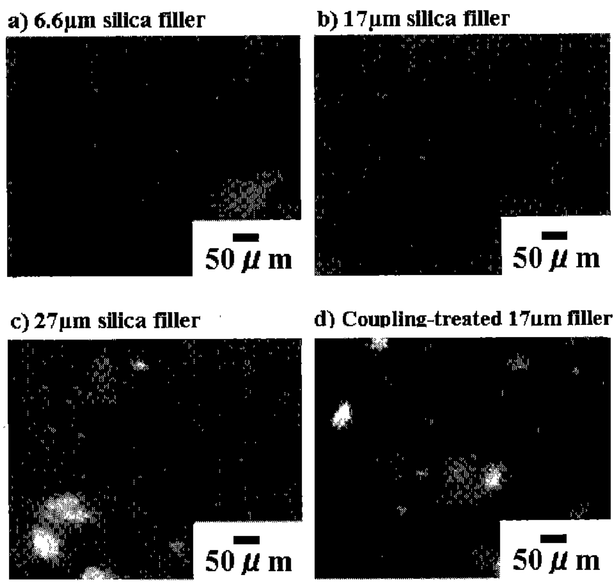


Fig. 7 Optical photographs of SiO_2 filled epoxy resin composite systems taken in transmission mode by using cross-polarized light; a) 6.6 μm median diameter filler system, b) 17 μm, c) 27 μm and d) coupling-treated 17 μm system.

4. Discussion

Epoxy resin composite system filled with silica particle characteristically contained the elliptical shaped objects observed as brightened domains at crossed polarization. The elliptical body exhibited optical anisotropic properties, and was often observed in silica particle systems with narrow size distributions and/or coupling surface treatments. Closely packed particle-aggregated structures also appeared in the silica systems. Origin of the elliptical brightness was induced from the particle-aggregated structures, which was presumably based on their mismatching of refractive index, etc. The optical anisotropy explained deconstruction/reconstruction process of particle aggregation and apparent viscosity as a rule of thumb.

Present experiments showed that the viscosity values of 6.6 and 27 μm-SiO_2 particle systems were larger than those of 17 μm particle system. This is out of order to well-known recognition of previous work about SiO_2 primary properties; median diameters, size distributions and specific surface areas. Further, the coupling-treated filler appeared a narrow size distribution, which should be due to the change of particle aggregation. However, the median diameters and specific surface areas were almost the same. Narrower size distribution is reported to increase the viscosity values, because much rather resin solvent is necessary for packing the silica filler with same weight as compared with broader particle system [24, 25]. Whereas, the coupling agent reinforces the interaction with particle surface and solvent (epoxy resin) in the same time. This is also expected to increase the viscosity, and it is not unclear which effect is crucial. These indecisive conclusions should be assigned to complicated primary properties of practically used raw powder provided in the present experiments; i.e., many variations in size distribution and specific surface area, insufficient spherical morphology, etc. It is conceivable conclusion that particular mechanism of viscosity is still unknown solely from SiO_2 primary properties.

Present "visual" work, transparent optical microscopy, revealed directly the particle-flocculated group in resin. The epoxy resin composite is predicted to have a primary or a secondary flocculated group of each SiO_2 particles before shearing, and the groups break up as the shear velocity increases at a thixotropic softening and hardening range [15-19]. Elliptical features observed in epoxy system were assigned to the primary and/or secondary flocculated groups, which were predicted at previous theoretical studies.

Particle flocculated groups in the composite system is supposed to contain a solvent among them before applying shear force [15-19]. The shearing breaks the flocculation, disperses the solvent into the system, and lowers apparently the SiO_2 filler concentrations in the structure. This means the reduction of viscosity. The amount of the flocculation expects to increase with the enlargement of the extent of non-spherical particle shape, narrow size distribution and reinforced interaction of particle and solvent. The larger amount of flocculation means the higher apparent concentration of SiO_2 filler particle in the structure. These explanations agreed well with the tendency of apparent viscosity of present experiment. The packing feature and optical anisotropy in transmission microscopy showed that non coupling-treated 17 μm system had especially smaller particle-aggregated structure, and the flocculated group among the 6.6 and 27 μm systems were larger than the 17 μm system, likewise in the coupling-treated 17 μm system. Consequently, the transmission optical microscopy offered one of linkage role for correlating with experimental results of viscosity and theoretical model of flocculation.

Origin of the optical anisotropy is urgent for understanding the rheological properties. Recent study in ceramics showed that the boundary of the granules of corundum particle of elongated shape made by powder compaction process could be a polarization point, which enabled to pass the incident linearly-polarized light though the analyzer-filter of microscope [26-31]. This study reported that non-uniformity in the structure, likely as the elongated particles aligned to one direction, had a relatively larger potential for the polarization. Polarized microscopy can detect the mismatching of refractive index at grain boundary and stuff like that. The SiO_2 particle used in this experiment were not completely spherical, more likely elliptical (**Fig. 2**). Similarly as previous study, the elongated shape of particles (their aggregates) and the mismatching of refractive index at the boundaries of SiO_2/resin are allocated to their optical anisotropic properties in the SiO_2 filled epoxy resin composite system.

As another possibility of the origin of this material system' optical anisotropy, it should be pointed out photo-elasticity phenomena. The photo-elasticity of resin polymer is fundamental characteristics used in liquid crystal displays (LCD). Polymer molecules impressed voltage or pressure are rearranged to specific direction, and they can be detected by polarized microscopy [32]. The SiO_2 filled epoxy resin composite system is also a polymer material system, and the photo-elasticity should be considered as a candidate. The optical anisotropic features in this experiment had the positive optical character of elongation, as shown in **Fig. 5**. This result strongly implied the characteristic orientation of polymer molecules. As one of assumable explanation, a heterogeneously packed SiO_2 aggregates locally provided a stress toward the resin polymers distributed in their surrounding areas. Previously, the role of interrelationship between particle and resin was noted as dominant for the filler-powder filled epoxy resin composite system [33]. Such kind of "locally induced photo-elasticity" might structure the rearrangement of polymer molecules, and make the polarization.

Sure, the further analysis is important to clarify the precise mechanisms of the relationship between viscosity and particle flocculation, and what's more, the origin of optical anisotropy. Anyway, the information of this work could be one of useful practical data for the SiO_2 filled epoxy resin composite system and contribute to understand the particle-resin (ceramics-polymer) interrelationship, which should be the decisive condition of the composite material. As one of application of the characterizing method, a feasible potential was suggested providing the product inspection tools for LSI package encapsulant or underfill material.

5. Conclusions

(1) The structural features in SiO_2 filled epoxy resin composite system were revealed directly through the characterization with transparent optical microscopy with normal/cross-polarized lights. The composite system essentially possessed the elliptical shaped features observed as brightened domains at transparent crossed polarization. The elliptical feature exhibited the repeated changes between bright and dark under rotating specimens at every 45° increments, and the positive optical character of elongation with microscopy using tint plate. The elliptical brightness and optical addition/subtraction retardations, i.e., optical anisotropic properties, were often observed in silica particle systems with narrow size distributions and/or coupling surface treatments. Closely packed particle-aggregated structures also appeared in their silica systems, more frequently. The interrelationship between elliptical brightness and particle aggregates was strongly suggested. An origin of the optical anisotropy was expected as inducing from the optical phenomena at the boundary between particle and resin.

(2) Optical anisotropy at transparent crossed polarization was supposedly affected by deconstruction and reconstruction process of primary/secondary-flocculated groups of each primary SiO_2 particle, which process was recognized as one of explanation of peculiar rheological characteristics of SiO_2 filled epoxy resin composite system. Packed particle-aggregated structure at transmission microscopy and elliptical brightness extent/size at crossed polarization were increased in silica particle systems with narrow size distributions and/or coupling surface treatments. Values of apparent viscosity observed in the silica systems were directly proportional to the closely packing and optical anisotropy extents. The optical anisotropy in transparent microscopy could be one of reliable guide for identifying particle-aggregated structure, and understand the rheological properties in SiO_2 particle filled epoxy resin composite system; nevertheless, the rheological property remained unclear solely from SiO_2 primary properties such as median diameters, size distributions and specific surface areas.

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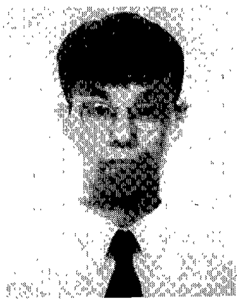

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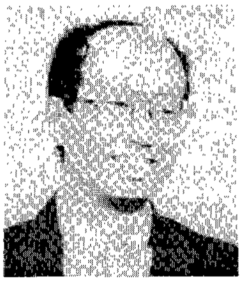

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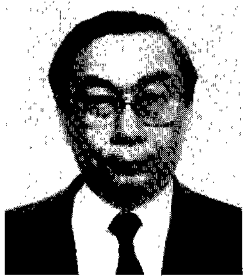

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