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### Particle Chromatography

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## COMMUNICATION

### Particle Chromatography

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#### Abstract

Particle mixtures can be separated into bands by slowly increasing the freezing rate during directional solidification. Continuous size classification of spherical particles can similarly be obtained.

We (1, 2) and others (3-9) have shown that a foreign particle is generally rejected during solidification unless the freezing rate exceeds a critical value  $V_c$ . Above  $V_c$  the particle is trapped and incorporated in the solid. Values for  $V_c$  are sensitive to the particle-melt combination. Although one cannot yet predict  $V_c$  for a particular pair, it is expected that factors such as relative thermal conductivity, melt viscosity, and surface properties of particle and solid all influence  $V_c$ . The critical freezing rate of spherical particles has been found to vary roughly with the reciprocal of particle diameter. The critical rate for rough particles is higher than for spherical particles and is less size dependent.

These previous results suggest that one might use this particle pushing phenomenon to separate particle mixtures by size and type. The idea is to disperse the particles in a relatively pure liquid which does not act as a solvent for the particles, and then directional-freeze the liquid at a

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steadily increasing rate—programmed solidification. As  $V_c$  is exceeded for each type of particle, it will be trapped in a band. Thus the name “particle chromatography.” Because of the specificity of  $V_c$ , it might also be possible to identify particles by determining  $V_c$  in several liquids.

The purpose of the experiments reported here was to test this concept of particle chromatography.

## EXPERIMENTAL METHODS

Two experimental techniques were tried—the vertical Bridgman-Stockbarger technique and horizontal zone melting with rotation—as discussed in detail elsewhere (2). Carbon, copper, red ferric oxide, and silver particles were ultrasonically dispersed in molten naphthalene which was then poured into 10.5 mm i.d. Pyrex tubes. Particles were included only in material at the front end of the tube, in tubes intended for zone melting experiments, so that the initial zone contained all particles present. The remainder of the naphthalene contained no particles. All but the smallest particles of each type settled to rest on the interface during the Bridgman experiments. Particles were completely suspended in the rotating zone-melting experiments.

In a preliminary Bridgman experiment with ferric oxide and carbon particles, bubbles formed at the interface and caused irregular trapping of particles, preventing appreciable separation. Bubble formation was prevented in subsequent experiments by sealing the tube containing solidified naphthalene in a vacuum of 0.05 Torr prior to performing the experiments. Although bubbles were not a problem in horizontal zone melting, most of the gas was removed by a preliminary rapid zone pass of the naphthalene which initially contained no particles.

In order to avoid trapping at the wall, it was also found necessary to use a concave interface.

## RESULTS

Carbon and copper were quantitatively separated by horizontal zone melting with rotation at 44 rpm, as shown in Fig. 1. The zone travel rate was increased from 25 to 75 mm/hr over 4 hr. Carbon particles began to be trapped at 34 mm/hr. The carbon was all trapped in the center of the ingot over a length of 2 cm. Copper was trapped uniformly across the ingot from 54 to 75 mm/hr.

Carbon and copper were also separated by the vertical Bridgman method, although the trapping order was reversed. By programming the

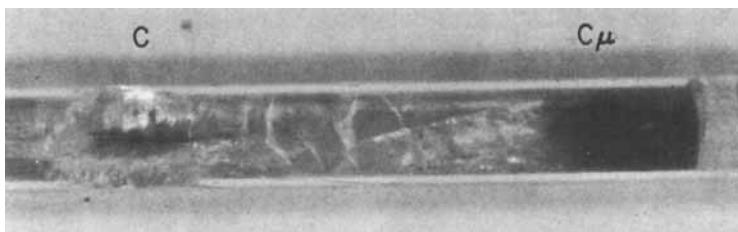


FIG. 1. Carbon and copper particles were separated during programmed solidification of naphthalene in horizontal zone-refining with rotation (HR-16 M).

tube lowering rate from 10 to 40 mm/hr, copper was trapped in the middle of the tube and carbon in the upper portion.

A limited amount of size classification was obtained for silver particles using the Bridgman-Stockbarger method by increasing the freezing rate from 6 to 26 mm/hr. The bottom contained predominantly large particles. The problems probably arose from the irregular shapes of the particles and the polycrystallinity of the solid naphthalene. Spherical particles with a single crystal solid should yield perfect continuous size classification even of submicron particles.

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