

- The crystal growth (and/or dissolution) process inside individual slugs is discussed in Section 5.4, with control by both robust design and temperature zones.
- The pre-slug nucleation process is discussed in Section 5.5. Successful nucleation control can make the growth control much easier. As continuous nucleation methods are well reviewed in other chapters of the book, only recently developed methods are discussed, such as cooling micromixers and focused indirect ultrasonication.

## 5.2 Control Slug Stability

The slug-flow stability is analyzed for the enhanced control of the continuous crystallization process, with design and operational considerations, in Subsection 5.2.1. Detailed analysis for hydrodynamically stable flows is in Subsection 5.2.2. Transitions of different forms of slug flow are in Subsection 5.2.3.

### 5.2.1 Stable Slug Flow for Crystallization Purposes

*Slug stability* refers to the situation in which slugs do not combine or separate, nor switch to other flow patterns (*e.g.*, stratified flow, see Figure 5.4).<sup>2</sup> In this way, all slugs have the same residence time, without significant material transferred between slugs. Important factors that affect slug stability include: (1) tubing diameter, (2) velocity of air and liquid streams, and (3) tubing inner surface properties. The larger the tubing inner diameter, the more limited the operating conditions that generate and maintain stable slugs, that is, the smaller the operating regime of stable slug flow. For tubing with fixed diameter, the stable slug-flow regime is mainly affected by velocity, and the tubing inner surface properties are discussed in Subsection 5.3.2 for slug geometry. The tubing should be wrapped in parallel without kinks to reduce possible slug size change or slug combination.<sup>2</sup>

A common method for generating stable slugs is to search by trial-and-error for combinations of air and liquid flow rates. The slug stability (*e.g.*, whether any adjacent slugs combine, or slugs separate) can be easily checked (*e.g.*, visual observation) throughout the transparent crystallizer tubing.<sup>2</sup> If a “flow map” is available for systems of similar geometries, the air and liquid flow rates can be chosen within the stable operation regime. It is advisable to operate far away from regime boundaries to avoid unexpected flow transition (Subsection 5.2.3) or slug size change (Subsection 5.3.1). Note that liquid flow rate is also directly limited by the final production rate.

In practical continuous operation, slugs accelerated during the shutdown of experiments. The higher velocity likely comes from low resistance, with fewer liquid slugs in the system. If too fast, the slug flow rate can cause slug combination, or liquid left inside the tubing. To maintain nearly constant slug velocities throughout the entire experiment, slugs of water were used before and after the crystal-containing slugs so that the pressure drops from the inlets to the tube outlet are nearly constant.<sup>1</sup>