Advanced Powder Technology 21 (2010) 652–657

Contents lists available at ScienceDirect

Advanced Powder Technology

journal homepage: www.elsevier.com/locate/apt

Original Research Paper

Agglomeration process of dry ice particles produced by expanding liquid carbon dioxide

Yi-Hung Liu, Hiroyuki Maruyama, Shuji Matsusaka *

Department of Chemical Engineering, Kyoto University, Kyoto 615-8510, Japan

ARTICLE INFO

Keywords:
Dry ice
Jet flow
Agglomeration
Particle deposition
Reentrainment

ABSTRACT

Formation of dry ice particles and their agglomeration process have been studied experimentally. The dry ice particles were produced by expanding liquid carbon dioxide at room temperature and pressure, and then introduced into an additional tube acting as an agglomeration chamber. In the experiments, the temperatures of the jet flow and the tube wall were measured by thermocouples, and dry ice particles in the jet flow were observed by a high speed camera with a zoom lens. It was found that two stages of temperature reduction occurred in the jet flow, corresponding to the agglomeration process. It was also found that the particle size of the agglomerates increased and the particle velocity decreased with increasing tube diameter. The agglomeration process of dry ice particles can be explained by the particle deposition and reentrainment, i.e. dry ice particles of several micrometers are deposited on the tube wall and form a deposition layer; then, agglomerates are reentrained from the layer into the jet flow.

1. Introduction

Dry ice jet is widely used as an industrial technique, which can not only be applied to surface cleaning for semiconductor devices, automotive molds, food processing equipments, etc. but also to food refrigeration and pharmaceutical granulation. This is because the system has the specific feature of low temperature gas–solids two phase flow containing sublimation particles.

The concept of dry ice jet applied to surface cleaning was first proposed in 1980s [1]. In this study, fine particles called dry ice snow was produced by expanding liquid carbon dioxide. Dry ice jet can be used for the removal of fine contaminants strongly adhering to the surfaces, due to the penetration of dry ice particles through the boundary layer. Compared with air jet where only drag force is applied for the removal of the contaminants, impact of dry ice particles substantially enhances the removal efficiency. The quantitative analysis of the cleaning effect of dry ice jet was carried out, including removal of organics as well as particulate contaminants [2–6]. The organics were found to be dissolved in liquid carbon dioxide due to the dry ice particles during impact. Other research investigated the particle removal mechanism based on a collision model [7,8]. The particle size of dry ice is thought to be an important factor which greatly affects the contaminant removal efficiency. Other than for cleaning applications, dry ice jet was also employed in the study of refrigeration [9,10] and pharmaceutical granulation [11].

To improve the production efficiency of the dry ice particles, a thermally insulated chamber is placed after the expansion nozzle. Small dry ice particles can agglomerate in the chamber [12]. However, the formation, size and state of the dry ice particles, and the agglomeration process have not been studied in detail.

In this study, we focus on the production of the dry ice particles by expanding liquid carbon dioxide, and elucidate the mechanism of the formation of agglomerated dry ice particles.

2. Experimental apparatus and procedures

Dry ice particles were produced from high purity liquid carbon dioxide. A flexible thermally insulated hose, 2 m long and 15 mm in inner diameter, was connected to a high pressure carbon dioxide cylinder and an expansion nozzle was installed at the end of the hose, as shown in Fig. 1. In order to change the conditions of the expanded flow, a glass tube was placed at the outlet of the expansion nozzle. The primary pressure of the carbon dioxide was measured at the entrance of the expansion nozzle.

Fig. 2 shows the details of the test section, which consists of the expansion nozzle and the glass tube. The expansion nozzle has an inner diameter of 0.2 mm and a length of 6 mm, while the glass tube was 2, 4, or 6 mm in inner diameter and 50 mm in length (L), and was installed onto the outlet of the nozzle. The temperatures of the dry ice jet ejecting from the expansion nozzle and of the glass tube wall were measured by a K-type thermocouple

---

* Corresponding author.
E-mail address: matsu@cheme.kyoto-u.ac.jp (S. Matsusaka).

0921-8831/$ - see front matter © 2010 The Society of Powder Technology Japan. Published by Elsevier B.V. and The Society of Powder Technology Japan. All rights reserved.
doi:10.1016/j.apt.2010.07.009

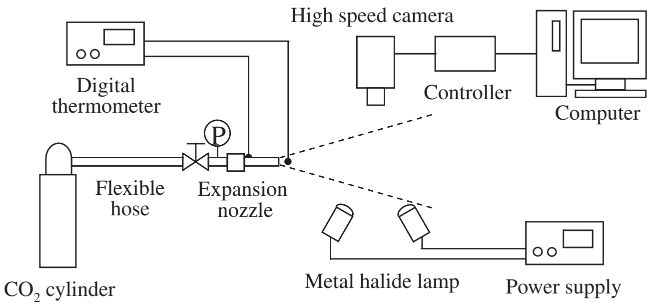

Fig. 1. Schematic diagram of experimental apparatus.

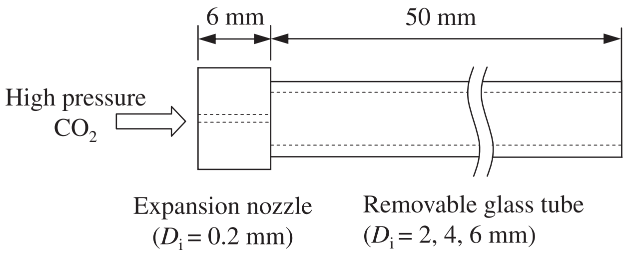

Fig. 2. Test section.

connected to a temperature recorder (NR-1000, KEYENCE Corp.). A high speed camera (Fastcam-Max, Photron Ltd.) with a zoom lens was used to observe the state of agglomerated dry ice particles in the jet flow.

To investigate the formation of a deposition layer of dry ice particles, a glass plate instead of the glass tube was placed at the outlet of the expansion nozzle. The structures of the dry ice particles deposited on the plate were observed by an optical microscope (DS-3040L, Olympus Corp.). All the experiments were conducted under room temperature and pressure (25 ± 2 °C; 1 atm).

3. Results and discussion

3.1. Effect of temperature of jet flow on producing dry ice particles

3.1.1. Experiment using expansion nozzle

Carbon dioxide was expanded from the nozzle at a primary pressure of 6.5 MPa to atmospheric pressure. The expanded gas flow was cooled by rapid expansion. Fig. 3 shows the temperature measured at the center of the expanded flow along the flow axis. The measurement position x is the distance from the nozzle outlet. The temperature measured at $x = 1$ mm was about -80 °C; however, it increased to -10 °C at $x = 50$ mm and closed to room temperature at $x > 100$ mm. The temperature of the flow increased sharply after ejecting from the expansion nozzle. According to a phase diagram of carbon dioxide, dry ice can be formed at -78.5 °C and 1 atm, which indicates that most of dry ice produced from the expansion nozzle sublimated to the atmosphere due to the temperature increase. Consequently, dry ice particles were not observed visually.

3.1.2. Experiment using expansion nozzle with glass tube

In order to avoid the direct contact of the expanded flow and the surrounding air, a glass tube was installed at the outlet of the nozzle, and an experiment was carried out under the same conditions as above. The dry ice particles were observed visually in the jet flow from the glass tube. Fig. 4 shows the temperature variation of the outer tube wall as a function of time elapsed. The room temperature was 25 °C. For $x = 5$ mm (Fig. 4a), the temperature decreased and approached about -30 °C after 300 s had elapsed. As for $x = 48$ mm (Fig. 4b), the decrease of the temperature was relatively slow, and the stable temperature after 300 s was slightly higher than that for $x = 5$ mm, due to the heat transfer between the jet flow and the surrounding air through the tube wall. It is worth noting that the temperature at $x = 48$ mm was still kept at sub-zero temperature, i.e. the thermal insulation of the tube wall was effective; thus the sublimation of the dry ice particles produced by expanding liquid carbon dioxide was reduced. As a result, the dry ice particles were observed visually in the jet flow from the glass tube. In this experiment, glass tubes with different diameters were also used. Although there were small differences in the results, the temperature variations were almost the same as each other.

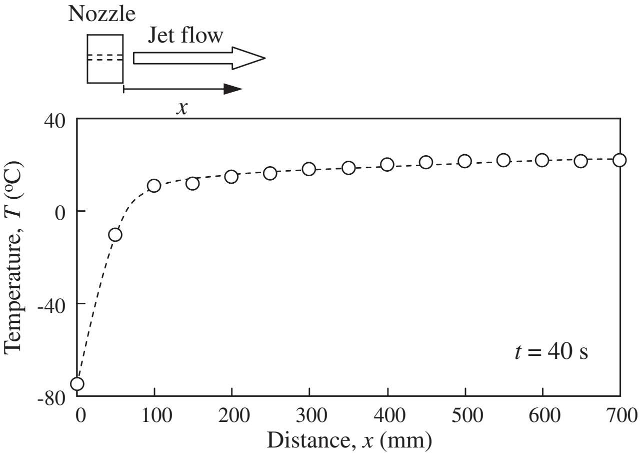

Fig. 3. Temperature variation with distance from expansion nozzle.

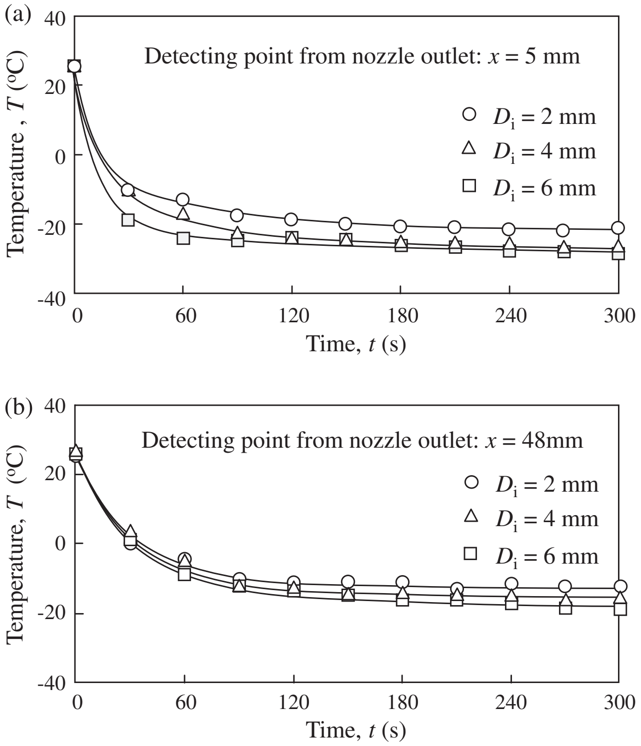

Fig. 4. Temperature variation of outer tube wall.

Next, the time course of the temperature in the jet flow was measured. As shown in Fig. 5, the temperature decreased from