

for designing the experiments in the current investigation where the extent of Bi-2223 conversion in various precursors are studied. In addition, the samples were subjected to slow cooling at different stages of the thermomechanical treatment to determine the influence of cooling rate on the properties of aerosol precursor PIT tapes.

## 2. Experiment

### 2.1. PIT tape fabrication

Bi-2223 precursor powders used in this study were produced by the aerosol pyrolysis technique. Details of this processing technique can be found elsewhere [3, 12]. Briefly, nitrate solutions were prepared from the oxides of the corresponding cations in the desired proportions. The individual solutions were atomized utilizing a series of ultrasonic transducers and fed into a vertical tube furnace. The temperature of the reaction chamber in the furnace was maintained in the range of 600–700 °C, and the residence time of the precursor in the reaction chamber was varied from 0.1 to 60 seconds. Since the size of the atomized nitrate droplets is sufficiently small, the droplets decomposed rapidly into metal oxides without lead loss. Precursor powders produced by this technique were determined to be less than 1  $\mu\text{m}$  in size, possessed a narrow particle size range, composed of many fine grains, and had excellent grain-to-grain chemical homogeneity. These powder characteristics combined to enable the oxide phase assemblage to react quickly to form the desired superconducting phase during subsequent thermomechanical treatment.

Following aerosol pyrolysis, silver-sheathed PIT tapes were fabricated using the standard PIT process. The aerosol precursor powders were packed into individual silver tubes of 6.25 mm outer diameter with wall thickness of 0.75 mm and opened at one end. Packing of the powders was performed in a glove box using a hand press, and the typical packing density was approximately 35% to 40% of theoretical. Openings of the silver tubes were then capped with silver plugs and swaged, and the composite tubes were drawn to roughly 1 mm diameter at 50 to 100  $\mu\text{m}$  reduction per pass. Following tube drawing, the monofilamentary wires were rolled at 5 to 10% reduction per pass to roughly 0.3 mm thick and 3 mm wide. The resulting PIT tapes were then cut into 2.5 cm long sections for thermomechanical processing.

### 2.2. Processing

**2.2.1. Variation in precursor composition.** It has previously been shown [14] that Bi–Sr–Ca–Cu–O, i.e., the  $\text{Bi}_2\text{O}_3$ –SrO–CaO–CuO system, is characterized by extended three-phase equilibria with flat four-phase equilibria existing between them. Consequently, small variations in composition and temperature can lead to significant changes in phase equilibria and phase distribution. Therefore, a large number of nominal precursor compositions have been utilized by various investigators [2–4, 15–17] each with optimized processing

parameters specifically designed for the composition under consideration. Even though the precursor composition may differ, it is generally agreed that, on heating, the precursor is constituted of varying amounts of  $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$  (Bi-2212) and/or Pb-doped Bi-2212 as the major phase, and  $(\text{Ca}, \text{Sr})_2\text{CuO}_3$  alkaline earth cuprate (2:1 AEC), CuO and  $\text{Ca}_2\text{PbO}_4$  as secondary phases. Under appropriate temperature–atmospheric conditions, these phases react, with the aid of a liquid phase, to form Bi-2223 [18]. Furthermore, it has been shown that during this reaction, CuO is consumed rapidly whereas the 2:1 AEC reacts at a slower rate [19]. A precursor rich in Cu, therefore, will result in more abundant secondary phases and may lead to enhanced reaction kinetics. Indeed, Cu-rich compositions in the Bi-2223 + 2:1 AEC + liquid and Bi-2223 + 2:1 AEC + CuO + liquid phase fields have been proposed to provide enhanced kinetics in the formation of single-phase Bi-2223 [20]. On the other hand, excessive amounts of Cu addition can result in an over-abundance of residual AEC and CuO, which will lower the superconducting volume, disrupt the local alignment of the Bi-2223 grains and lead to a deterioration of the superconducting characteristics. To investigate these effects as well as the influence of Cu content on slow cooling as described in the following section, three precursor compositions with varying Cu content were selected in this study.

Based on the original composition (powder 1) used in a previous optimization study [13], two other aerosol precursor compositions with differing amount of Cu additions were prepared. The nominal compositions of the three powders are as follows:

Powder 1 :  $\text{Bi}_{1.84}\text{Pb}_{0.34}\text{Sr}_{1.91}\text{Ca}_{2.03}\text{Cu}_{3.07}\text{O}_8$   
no addition,

Powder 2 :  $\text{Bi}_{1.84}\text{Pb}_{0.34}\text{Sr}_{1.91}\text{Ca}_{2.03}\text{Cu}_{3.15}\text{O}_8$   
low Cu addition,

Powder 3 :  $\text{Bi}_{1.84}\text{Pb}_{0.34}\text{Sr}_{1.91}\text{Ca}_{2.03}\text{Cu}_{3.21}\text{O}_8$   
high Cu addition.

The first set of experiments (set A), which included three experimental sequences, was conducted to determine whether the optimum thermomechanical schedule found for powder 1 [13] will also result in the best performances for PIT tapes fabricated using powders 2 and 3. The first sequence (sequence 1) involved two short initial sintering intervals followed by a long one with intermediate pressings. The second sequence (sequence 2) is constituted of a lengthy initial sintering interval and concluded with two short sintering steps. The third sequence followed the optimum schedule developed in the previous study. These thermomechanical treatment sequences are listed in table 1 where the heat treatment duration is given in hours, a notation which will be used in the description of treatment schedules throughout this paper. As seen in table 1, the total sintering times of the samples were maintained at 100 h. During each sintering interval, the samples were heated to 350 °C at 10 °C  $\text{min}^{-1}$  and held for 1 h. Then, the temperature was raised to 825 °C at a rate of 2 °C  $\text{min}^{-1}$ . After holding at 825 °C for the selected duration, the samples were cooled to room temperature at 5 °C  $\text{min}^{-1}$ .