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A novel method of temperature control for a crystal growth puller

A facile method to control the contracting rate of the thermal expansible bars for pulling crystal is first suggested. The thermal expansible bars, set in a modified Dewar flask whose vacuum degree is controlled, are heated to designed temperature and then switch off the power to let it cool down at a desired rate, which depends largely on the changeable vacuum degree. This new approach is expected to completely eliminate the effects, which possibly reduces the smooth extent of thermal expansion, caused by the minor temperature fluctuations during crystal growth process and to realize the utmost smooth and slow pulling rate. It is expected to install this apparatus in optical floating zone furnace, instead of traditional motor, to grow peritectic crystal, such as crystal Bi-2223, since for the peritectic reaction, in principle, the extremely slow growing rate is considerably essential.

Keywords: floating zone, crystal growth, thermal expansion, peritectic reaction, Bi- 2223, Czochralski technique

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1. Introduction

For most superconductivity crystals, such as Bi-2223, are the outcomes of peritectic reaction. In order to obtain the pure phase crystal, peritectic reaction requires, in principle, absolutely slow growing rate. Nevertheless, for the common floating zone apparatus, the moving rate, obtained from a motor, of mirrors is intrinsically finite. Investigations have been showing the slow growing rate is necessary to grow this kind of crystal [1,2,3]. Although the optical floating zone furnace notes the pulling rate range is 0.05 - 27mm/h, our experiments indicate obviously when the pulling rate of mirror is less than 0.1 mm/h, the actual pulling rate is far from the designed one (see Fig.1). This does not mean the floating zone apparatus is not sufficiently precise, but the intrinsic limitation of machinery.

Y. Z. HE proposed a novel puller in 1998, which utilizes the displacement of thermal expansible bars, instead of the traditional motor [4], to obtain a smooth pulling rate. Subsequently an improvement was suggested a facile approach to settle the conflict between large displacement and limited furnace size [5]. Nevertheless, people argue that minor fluctuation of temperature will result in the fluctuation of pulling rate. Although it is believed that the colossal thermal inertia of materials will substantially counteract the influence caused by the departure of smoothness of heating rate, we still felt obliged to improve the puller further. In principle, a much smaller and smoother pulling rate, which will be used to grow peritectic crystal, such as Bi2223, can be obtained via the improved puller.

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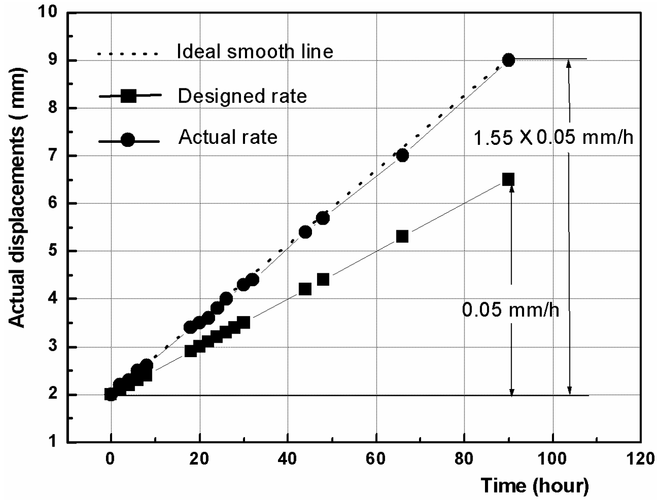

Fig. 1: Comparison of designed and the actual rate of moving mirror for optical floating zone furnace.

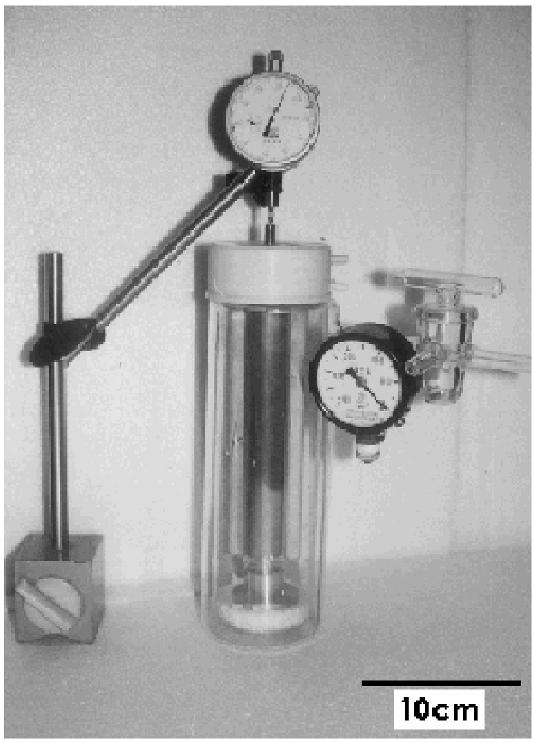

Fig. 2: Photograph of modified Dewar flask that is equipped with the vacuum-meter, micrometer and contains the thermal expansion system.

In this paper, a new method based on the contraction of metal during the cooling process is presented and the corresponding experiment is carried out. Changing the vacuum degree of Dewar flask means changing its thermal conductivity and the cooling rate of materials contained in it as well. As a result, the controllable contracting rate is obtained with great ease [6].

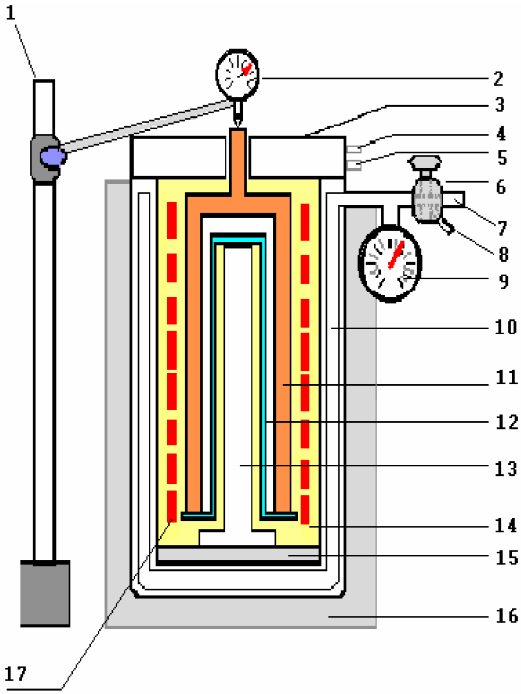

Fig. 3: Scheme for Fig. 2; Schematic representation:
(1) stand of micrometer,
(2) micrometer to show expansible and contracted displacement,
(3) cover of Dewar flask,
(4) tube used to pass electric wires,
(5) tube used to pass the thermocouples for measuring temperature,
(6) three-way glass stopcock,
(7) tube for vacuum- pumping,
(8) air vent valve,
(9) vacuum-meter,
(10) Dewar flask,
(11) Cu-Zn alloy tube 20cm in length,
(12) quartz tube 20cm in length,
(13) aluminium bar 21cm in length,
(14) & (15) heat preservation material,
(16) crust of heat preservation,
(17) heating elements.

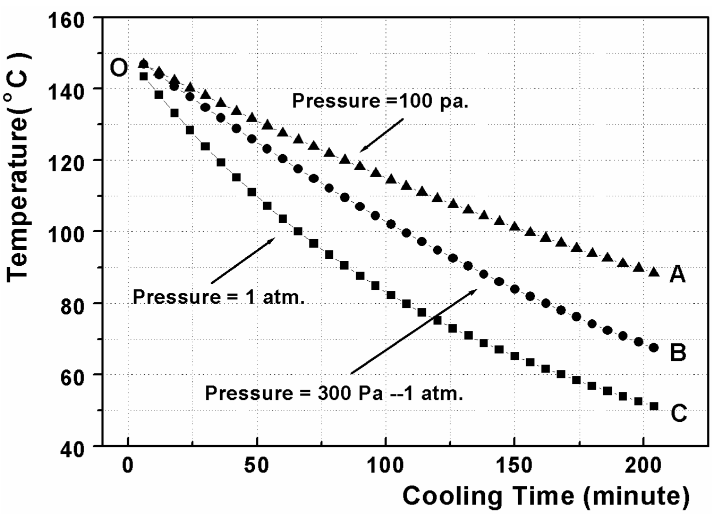

Fig. 4: Curve of temperature vs cooling time.

2. Experiment

Assemble the thermal expansion system and the modified Dewar. The photograph of Dewar and its schema are shown in Fig.2 and Fig.3, respectively. Heated thermal expansion system slowly in modified Dewar till temperature reached 150 °C and held the temperature for half an hour and then turned off the power. Adjusted the three-way glass stopcock to make the Dewar's vacuum degree equal to (a) zero, (b) 300 Pa and (c) change from 300 Pa to 1 atm at rough linear rate respectively. Wrote down the readings from the micrometer and vacuum-meter at the interval of 6 minutes. The experimental results were shown in Fig.4 and Fig.5.

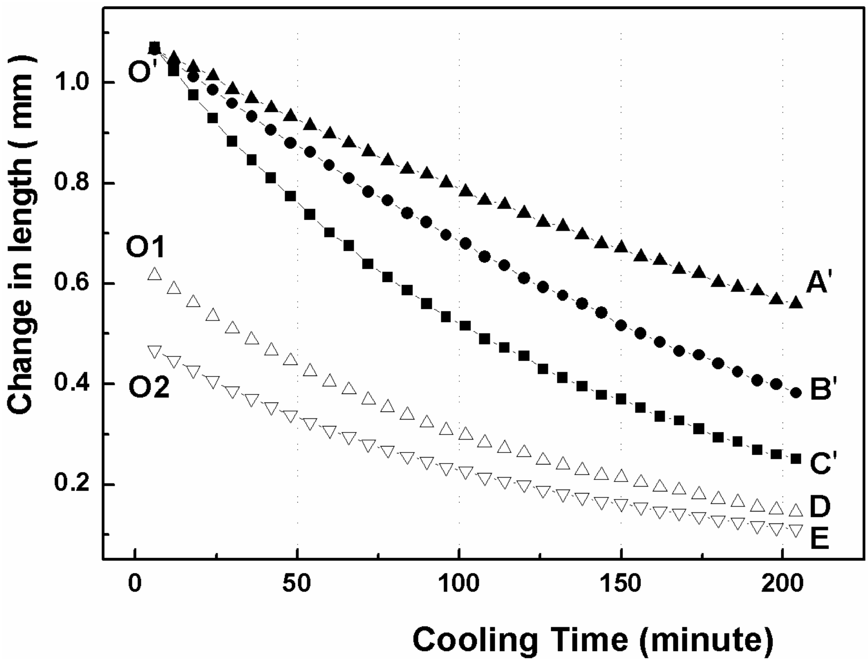

Fig. 5: Curve of contracted displacement vs cooling time; O'A', O'B', O'C' correspond to OA, OB,OC curve shown in FIG.4. O_1D,O_2E are the curves corresponding to the theoretical values of Al metal and Cu-Zn alloy of thermal expansion in 1 atm and their sum approximately equals that shown by O'C' since thermal expansion of quartz is much smaller than that of Al metal and Cu-Zn alloy**.**

3. Results

Every curve in Fig.4 there corresponds to one in Fig.5, that is, OA,OB,OC corresponds to OA', OB', OC', respectively. Usually the pulling rate of crystal growth is a constant and the nearly linear thermal expansion coefficient of metal above room temperature meets this requirement if heat conductivity can be controlled linearly. The lower vacuum degree, the larger of heat conductivity coefficient. The linear decrease of thermal expansion coefficient with temperature decrease compensates the change of heat conducting coefficient coincidentally. So it is easy to obtain various linearly contracting rate.

Despite of the existence of several factors that influence the thermal transfer efficiency, the cooling rate of thermal expansible bars is still controlled easily by Dewar with changeable vacuum degree. The new pulling approach (vacuum degree $\rightarrow$ heat conductivity coefficient $\rightarrow$ cooling rate $\rightarrow$ contracted rate) is expected to eliminate the influence caused by tiny temperature fluctuation and realize the utmost smooth and slow pulling rate.

4. Summary and outlook

The experimental results indicate obviously it is feasible to control the contracting rate (pulling rate) of metal bars by adopting Dewar with a changeable vacuum-degree. Although the coefficient of thermal expansion is not a constant, a changeable vacuum-degree will compensate this shortcoming to realize linear (or designed), slow and smooth pulling rate.

It is expected that this new puller provides an ideal puller to eliminate the growth striations introduced by the fluctuation of pulling rate during crystal growth, and is a powerful tool for studying the mechanism of crystal growth since crystal growth striations write down the history of crystallization objectively. On the other hand, the new method can also be possibly used to pull such a kind of crystal in which concentration of the solute distributes periodically and the period is much smaller than that of the crystals pulled by the traditional method. In a word, it is a worth to research further.

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