National Institute of Standards & Technology

Certificate

Standard Reference Material 740a

Zinc

Freezing Point Standard

419.527 °C

International Temperature Scale of 1990 (ITS-90)

The temperature given above is the value assigned to the freezing point of pure zinc as one of the defining fixed points of the International Temperature Scale of 1990 (ITS-90). The fixed point is realized as the plateau temperature (or liquidus point) of the freezing curve of slowly frozen high-purity zinc.

The zinc for this Standard Reference Material (SRM) is of high purity (99.9999 + %), with the total of all other elements that affect the freezing point of less than one part per million. Based on samples tested, the temperature range of melting of the bulk material is not expected to exceed 0.001 °C. Plateau temperatures for samples of this material are expected to differ by not more than 0.0003 °C from each other, by not more than 0.0002 °C from SRM 740 zinc, and by not more than 0.0005 °C from the assigned temperature.

The zinc metal for this Standard Reference Material was obtained from Johnson Matthey Co., Spokane, WA 99216. The metal is in the form of millimeter-size "shot" and is packaged in 200 gram units in mylar envelopes in an argon atmosphere. Temperature studies on freezing point cells prepared using metal from randomly-selected envelopes were performed by E. R. Pfeiffer and G. F. Strouse of the NIST Chemical Process Metrology Division.

The technical and support aspects involved in the certification and issuance of this Standard Reference Material were coordinated through the Standard Reference Materials Program by J. C. Colbert. The original coordination of certification efforts was performed by R.W. Seward.

November 13, 1990
Gaithersburg, MD 20899

William P. Reed, Acting Chief
Standard Reference Materials Program

(over)

SUPPLEMENTAL INFORMATION

TESTING: The thermal tests for the certification of this Standard Reference Material were performed on three freezing point cells prepared in a manner similar to that described in Reference 3, below. Each cell contained approximately 1000 grams of zinc obtained from randomly-selected envelopes of the lot.

The freezing points were determined using the recommended "induced inner freeze" method. With the metal completely melted, the furnace was set at about 0.75 °C below the freezing point temperature. After supercooling and recalescence had been observed with a standard platinum resistance thermometer (SPRT) in the cell, the thermometer was removed and several cool silica glass rods were successively inserted into the thermometer well to induce freezing of a mantle of metal around the well. The cooled thermometer was then reinserted in the cell and recording of readings was begun. After equilibrium was established, the freezing plateau was found to vary no more than 0.0001 °C during the first 50 percent of the approximately 15 hours duration of the freeze. A typical freezing curve obtained under such conditions is shown in Figure 1 (the region of supercooling and recalescence is not shown, as the curve begins after the reinsertion of the thermometer); some of the same freezing data are plotted at greater resolution in Figure 2.

After the metal was slowly and completely frozen in the above manner, the furnace was set at about 1.0 °C above the freezing temperature to slowly melt the metal over approximately 13 hours duration, and thermometer readings were continuously recorded until the melting was complete. A typical melting curve obtained under such conditions is shown in Figure 3; some of the same melting data are plotted at greater resolution in Figure 4.

During the freezing and melting curve measurements, an inert environment of helium gas at 1 standard atmosphere (101 325 Pa) pressure was maintained in the cells.

Following the freezing and melting curve measurements, the freezing plateau temperature of each test cell was directly compared with that of the Platinum Resistance Thermometer Calibration Laboratory standard freezing-point cell containing SRM 740 zinc.

OTHER: Any handling procedures on high-purity material are apt to introduce contamination. The "shot" form of this SRM minimizes the need for handling during freezing-point cell construction. Nevertheless, every possible effort should be made to maintain the purity of this SRM through the use of polyethlyene gloves during handling and a clean laboratory environment. In assigning a precise temperature value to realizations of the zinc freezing point for calibration purposes, corrections must be applied for the average depth of immersion, ℓ, of the thermometer sensing element below the surface of the metal ($dt/d\ell = 2.7 \times 10^{-3}$ °C/m) as well as for the ambient pressure over the cell ($dt/dp = 4.3 \times 10^{-8}$ °C/Pa) during the measurements, if not controlled at 1 standard atmosphere as discussed above.

REFERENCES: Further information on temperature scales and metal freezing points may be found in:

[1] H. Preston-Thomas, "The International Temperature Scale of 1990 (ITS-90)," Metrologia 27, 3-10 (1990).

[2] B.W. Mangum and G.T. Furukawa, "Guidelines for Realizing the International Temperature of 1990 (ITS-90)," Natl. Inst. Stand. Technol. Tech. Note 1265, 190 pages (1990).

[3] G.T. Furukawa, J.L. Riddle, W.R. Bigge, and E.R. Pfeiffer, "Standard Reference Materials: "Application of Some Metal SRMs as Thermometric Fixed Points," Natl. Bur. Stand. (U.S.), Spec. Publ. 260-77, 140 pages (1982).

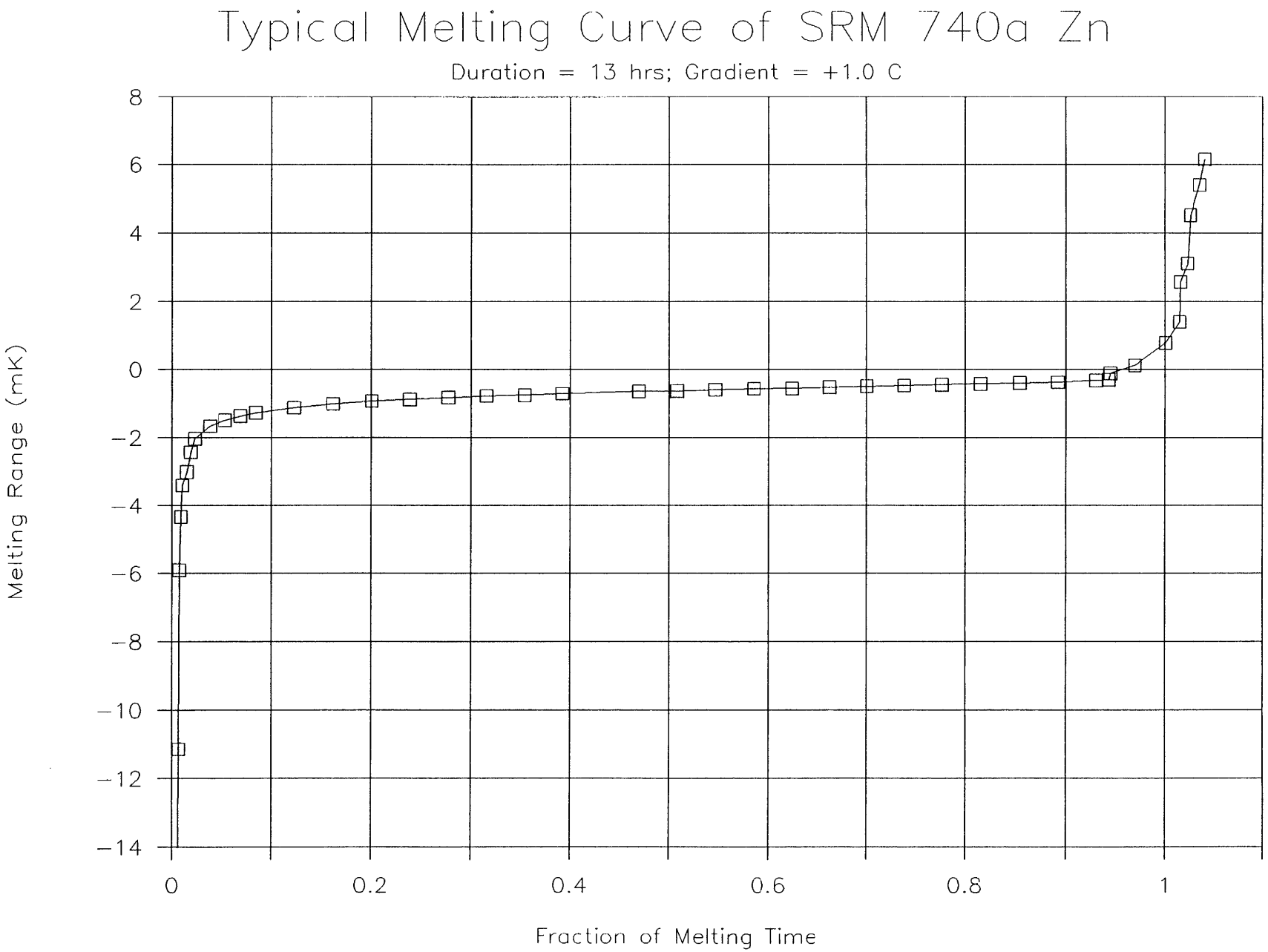

Figure 3. A typical melting curve of SRM 740a zinc following a slow freeze. This melt followed the slow freeze of Figure 1.

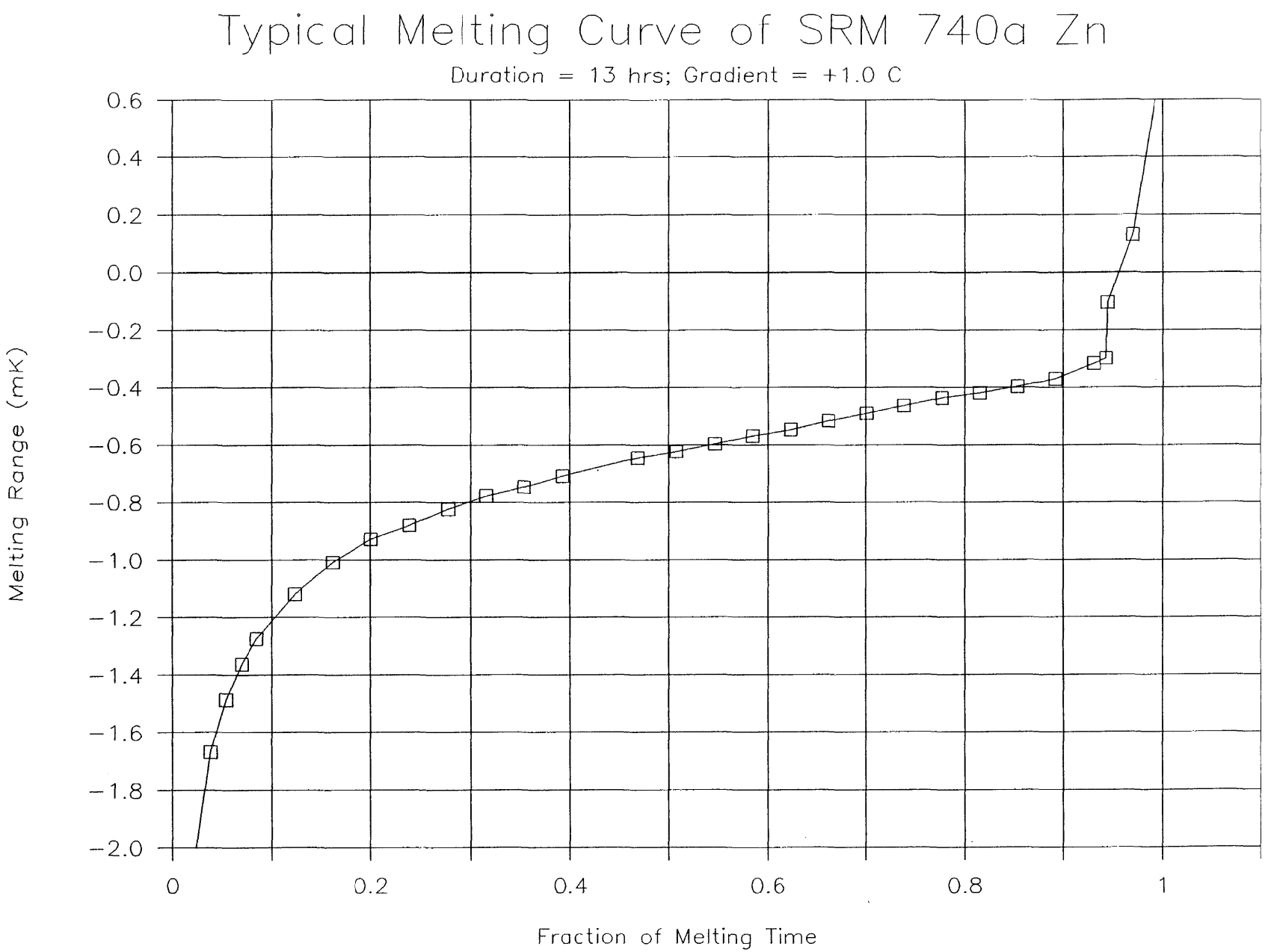

Figure 4. The melting plateau region of Figure 3 at greater resolution.

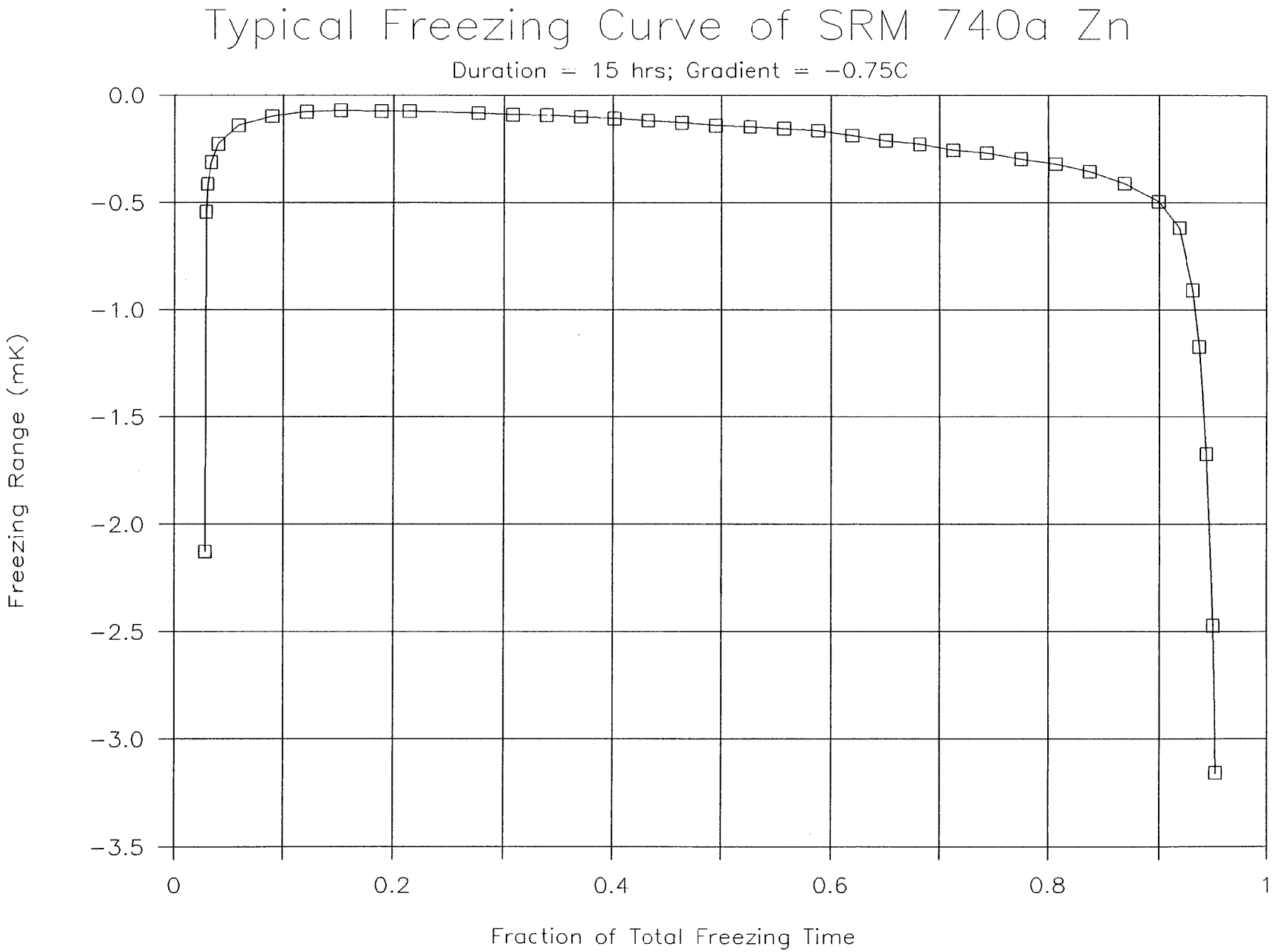

Figure 1. A typical freezing curve of SRM 740a zinc using the "induced inner freeze" preparation technique.

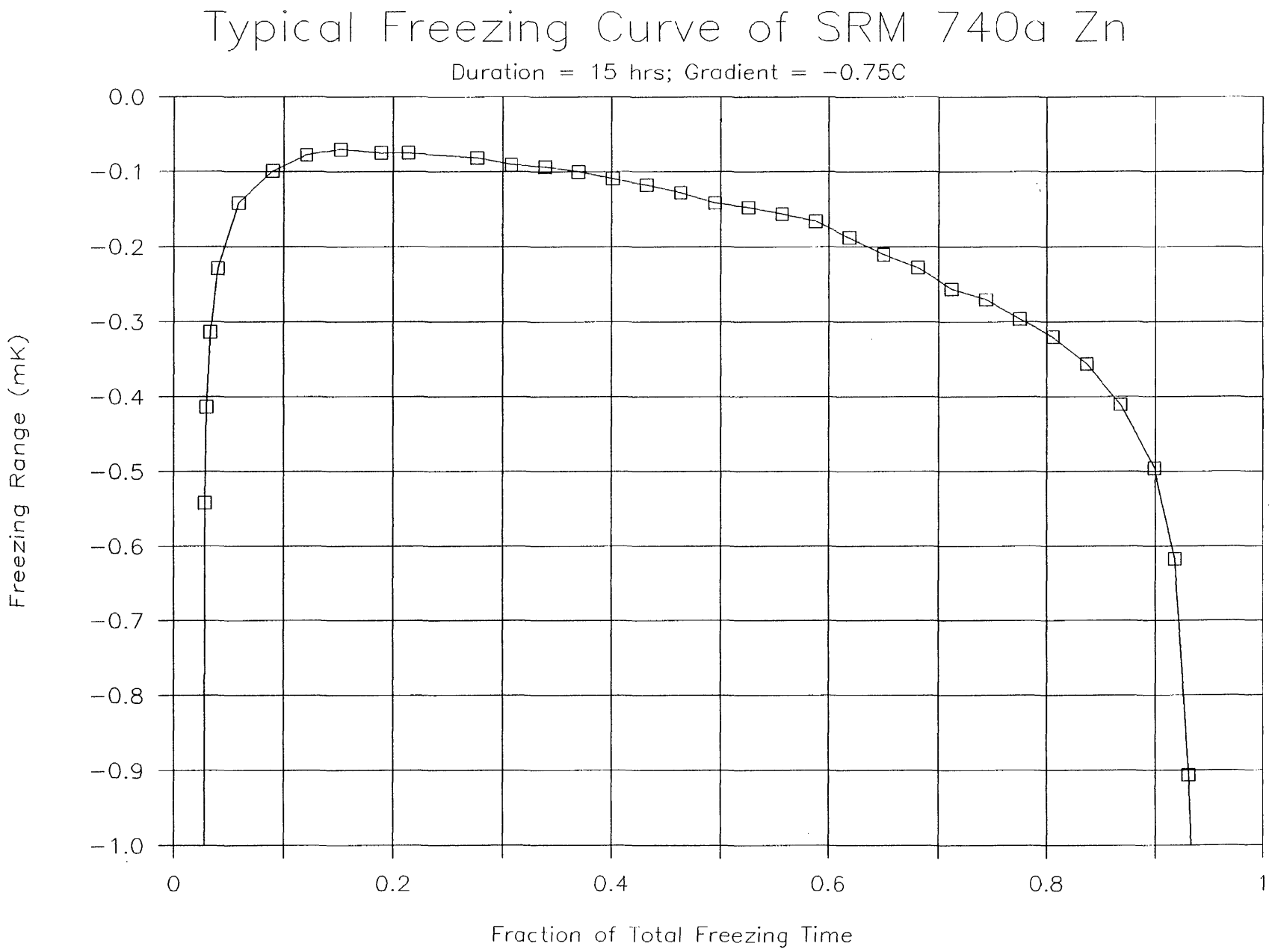

Figure 2. The freezing plateau region of Figure 1 at greater resolution.