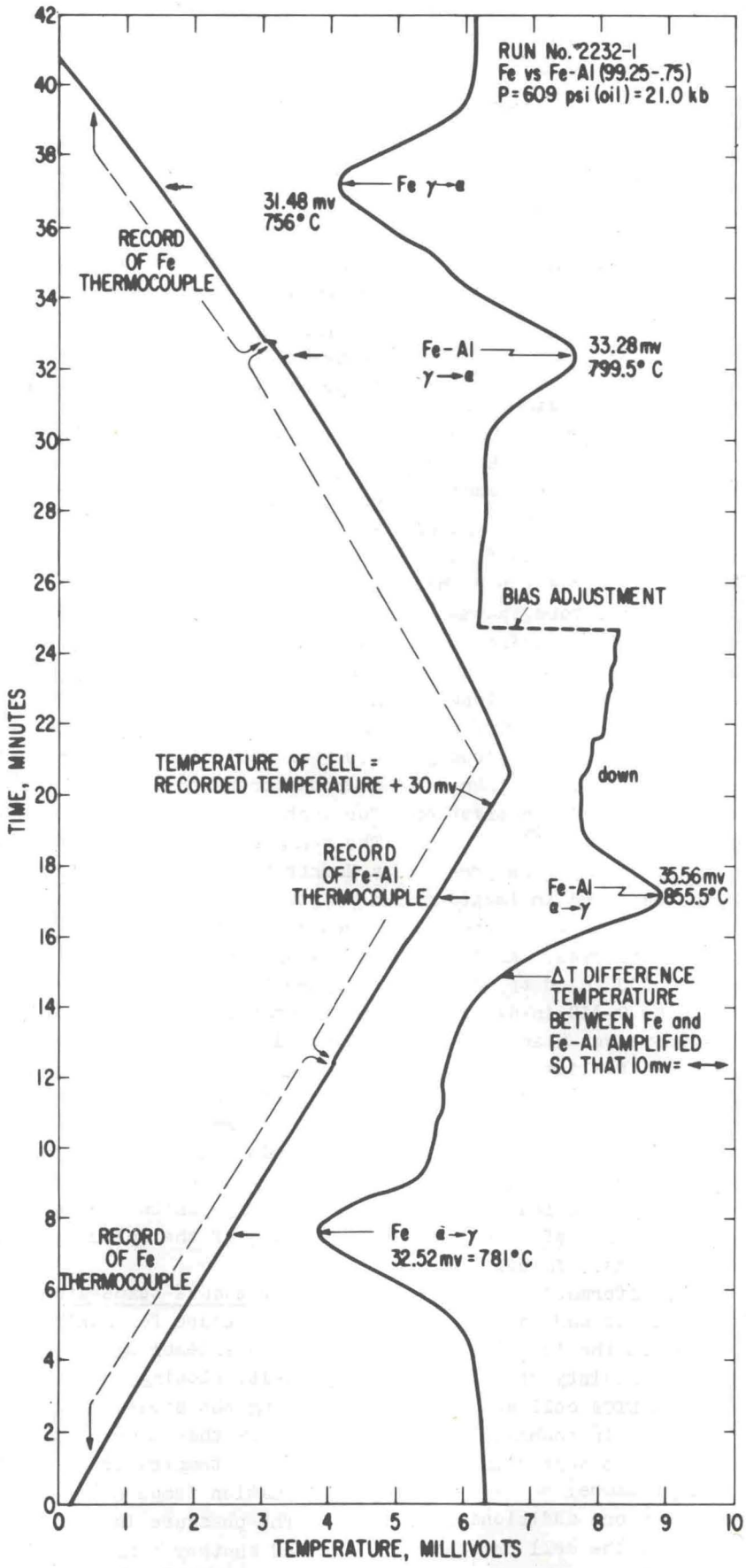

Fig. 3 Temperature versus ΔT trace from an actual duplex DTCA run

pills, the purpose of which is to provide good thermal conduction at the top and at the bottom between the metal being investigated and the reference pieces.

The electrical heating current is carried from the strips to current rings via the heater tabs or ears, shown in Fig.2. The current rings make direct contact with the punches of the punch-belt-punch superpressure apparatus, and the electrical leads are connected to these punches which are electrically insulated from one another with respect to the body of the press.

Pressure Calibration of Cell

The pressure within the DTCA cell was determined by observing the room temperature transformations of Bi wire (25.3 and 89 kb) and of Ba wire (59 kb). Two methods of calibration were used:

(a) The center 0.100-in-dia core of a typical DTCA sample holder, as shown in Fig.2, was filled with AgCl, into which was placed a hole for either a Bi or a Ba wire. With the heater tabs incomplete one could read the wire resistance from punch to punch, and hence could observe the ram loading required for these wire transformations. A second cell was then constructed for a DTCA run, loaded to a ram pressure close to that previously observed for the wire transformation, and then heated through the temperature for the observation of the iron transformation.

(b) An alternative, and probably more precise, method of calibration consisted in locating the Bi wire in the same DTCA cell in which the iron transformation was to be observed. An annular ring 0.100 in. wide and 0.030 in. deep was grooved into the outside of the 0.350-in-dia sample holder, and into this groove was placed 3/4 turn of 0.019-in. Bi wire and a filling of NaCl paste containing glyptal resin and acetone. The ends of the Bi wire were attached to platinum leads going through the gasket area in a manner similar to that used for the thermocouple leads. Values of the resistance across these leads were recorded, permitting the observation of both the 25.3 and the 89 kb transformations. Immediately after the room-temperature transformation was observed at 89 kb, the DTCA cell was warmed to observe the iron transformation in the identical cell, thus eliminating the uncertainty of cell-pressure variation between the DTCA cell and a second calibrating cell, as used in technique (1). The 25.3-kb points for iron were observed later in each run after lowering the pressure.

Both techniques suffer from one additional calibrating deficiency; namely, the cell pressure may change appreciably in warming up the DTCA cell to observe the iron transformation. For the present, this possible error is believed to be less than 1-2 kb, but it remains uncertain.

Reproducibility of DTCA Data

The observed transformation temperatures from successive temperature cycles at a fixed pressure generally varied less than 3 deg C. The first transformation of a run was generally low by a few more degrees and these data were not used. Successive runs on the same alloy usually reproduced to around 5 deg or less.

The reference curve for iron may possess an error of 5-10 deg, which corresponds to an error in pressure of about 2 kb. This estimate is based on the degree of agreement between the various sets of data, on the reproducibility of successive DTCA runs, and on the consideration of possible changes in pressure which might occur in the cell as it is warmed up.

Temperature Control

Temperature control depends upon the maintenance of constant voltage across the heater elements, and is accomplished by means of a Sorensen Model 2501, 115-volt, 2.5-kva voltage regulator, capable of holding the output voltage to within $\pm$ 0.01 percent. This constant voltage is fed to a stepdown transformer, with a reactor interposed for controlling the voltage to the transformer. The control winding of the reactor is energized by a Hamner Model H-108 power supply, which provides a constancy in voltage comparable with that of the Sorensen regulator; interposed between the Hamner supply and the reactor is a 25,000-turn Helipot, designed to give a variable and almost completely smooth change in voltage to the reactor. The Helipot is motor-driven and the speed can be varied stepwise in either direction 1000-fold by means of an Insco Model 00140 speed changer. This equipment permits the rate of temperature rise or fall within the superpressure cell to be controlled in the range from 500 to 0.5 deg C/min, with a fairly constant rate being observed at any one setting of the speed changer.

Making a Pressure-temperature Run

The procedure for making a superpressure run consists in assembling the cell and gasket parts in the belt, closing the punch-belt-punch assembly and raising the pressure to the desired value. A DTCA pass is then made by slowly raising and then lowering the temperature of the cell through the transformation temperature and difference temperature. The pressure is then changed to a different value and another temperature cycle is made. In this way, the entire P-T curve may be established for any one sample assembly.