A new method for determining the fluid-absent solidus temperature in piston-cylinder experiments

ANTONIO CASTRO,* MOHAMMED EL-BIAD, AND HASSAN EL-HMIDI

Department of Geology, University of Huelva, Campus de La Rábida, 21819 Huelva, Spain

ABSTRACT

We describe a new piston-cylinder method for determining the fluid-absent solidus curves of geological materials. Samples are heated incrementally at pressures from 3 to 20 kbar for periods less than one hour. The effects of melting on the measured oil pressure are small but detectable and yield an accurate solidus by interpretation of pressure-time data. The technique successfully reproduces the solidus curve for LiCl. Results for the Ollo de Sapo pelitic gneiss are superior to those obtained previously using a more conventional technique.

INTRODUCTION

The identification of magma sources in the Earth's mantle and crust is facilitated by the results of experimental petrology. The *P-T* positions of solidus curves for deep protoliths are critical to understanding the processes of magma generation. Solidus curves for potential crustal source materials, such as amphibolites and pelites, have been determined experimentally (e.g., Wyllie 1977; Singh and Johannes 1996; Petö 1976; Patiño Douce and Beard 1995, 1996; Patiño Douce and Harris 1998; Vielzeuf and Clemens 1992; Vielzeuf and Montel 1994). These results have shown that variations in the mineral assemblages, or in the compositions of the mineral solid solutions involved in the melting reactions (e.g., mica and plagioclase), can exert a strong influence on solidus temperatures. Consequently, to properly characterize magma production processes in a particular environment it is essential to determine the solidus curves for specific crustal protoliths. However, the work is arduous, requiring many time-consuming experiments in which the presence or absence of melt is determined by observation of scanning electron microscope (SEM) images of the run products. The technique described below can be used to determine the position of a solidus curve in *P-T* space by direct observation of the effects of melting on the oil pressure in piston-cylinder apparatus. These effects are very small but can be detected by examination of a pressure-time (*P-t*) curve produced by incremental heating.

RATIONALE

We have noticed in piston-cylinder experiments that 5–10 °C variations in temperature have a measurable effect on pressure. This is observed during the first minutes of an experiment when temperature is ramped up with a temperature controller. A corresponding ramp is obtained when measured

pressure is plotted against time. Consequently, compaction of the capsule container and/or the sample capsule can be detected by variations in the measured pressure during heating, which affect the slope of the pressure-time curve. Melting of the sample within the capsule will produce a compaction effect that may be detected if pressure is measured very accurately and data are taken continuously. Applying this method, we have conducted several melting experiments to identify the solidus temperature by changes in the pressure-time curve induced by heating of the sample in the piston cylinder.

EXPERIMENTAL PROCEDURES AND STARTING MATERIALS

Experiments were performed in end-loaded, solid-medium piston-cylinder apparatus with 12.7 mm (0.5 inch) diameter NaCl-graphite cell assemblies. Temperatures were measured and controlled with Pt_{100}-$Pt_{87}Rh_{13}$ thermocouples wired to Eurotherm 808 controllers. Oil pressures were measured with electronic DRUCK PTX 1400 pressure transmitters, connected to OMRON E5CK controllers. We used both natural rock samples and synthetic materials. About 20 mg of finely crushed powder (10–50 μm) were sealed within a gold capsule, which was subsequently embedded under pressure in an NaCl plug at room temperature. Because determination of the solidus temperature relies on detecting sample compaction, the technique only works for fluid-absent experiments. Water-bearing samples are not appropriate because water will fill the porous aggregate before melting occurs. The effects of volume reduction by melting-induced compaction of the sample can be magnified by using a porous aggregate that acts as a melt trap. We used quartz and diamond aggregates and obtained good results in both cases. For rock samples with excess quartz (e.g., pelites and gneisses), the introduction of a quartz trap had no influence on the melting temperature. For silica-poor compositions (e.g., amphibolites), the use of a diamond trap is more appropriate. Measured changes in pressure due to compaction of the

*E-mail: dorado@uhu.es

sample are normally very small (<1 bar oil pressure), and generally last only for a short time (<30 s). To detect any change due to melting within the capsule, we performed several experiments applying small increments of heat and observing the response of the system over short time intervals.

The assembly was heated by increasing the power manually a predetermined percentage of the total power every 30 s. We chose this time interval because it is sufficient to ensure that thermal equilibrium within the assembly was reached before the next energy input (Fig. 1a). In experiments using a diamond or quartz trap, compaction may be slower because melt must migrate through the porous aggregate. For those experiments heating increments were lengthened to 60 s. During the first 10 s after a heating increment, a ramp is produced for temperature (Fig. 1b) and a similar ramp for pressure (Fig. 1c). Temperature and pressure are then restabilized before the next power increase. If volume reduction occurs within a time interval it is indicated by a change in the slope or an inflection in the pressure ramps. The heating increment is induced by increasing the power manually from the temperature controller. With this procedure we ensure that the same energy increment is applied for each time interval (Fig. 1). The energy increment

must be as small as possible to determine accurately the temperature at which melting occurs. The energy increase must also be large enough to produce temperature increases that are greater than measurement error. We have estimated that an increment in the power supply of 0.4% is sufficient to produce a temperature increase of about 10–20 °C.

Measurement equipment and data recording

To detect the small changes in P and T produced in these experiments, it is necessary to use high-precision measurement equipment and to record pressures and temperatures continuously. The resulting data are used to construct P-t and T-t curves. In our experiments, temperature was controlled and monitored continuously using an Eurotherm 808 controller, with an internal ice-point compensator, connected to a computer for data acquisition and storage. The controller has a manual mode that we used to produce temperature increases within the desired time intervals. Oil pressure was measured at the gate of the piston ram by an electronic DRUCK PTX 1400 pressure transmitter, and monitored by an ONROM E5CK controller. The precision of this transmitter allows us to detect variations of 0.1 bar oil pressure (ca. 5 bar in the capsule). The ONROM
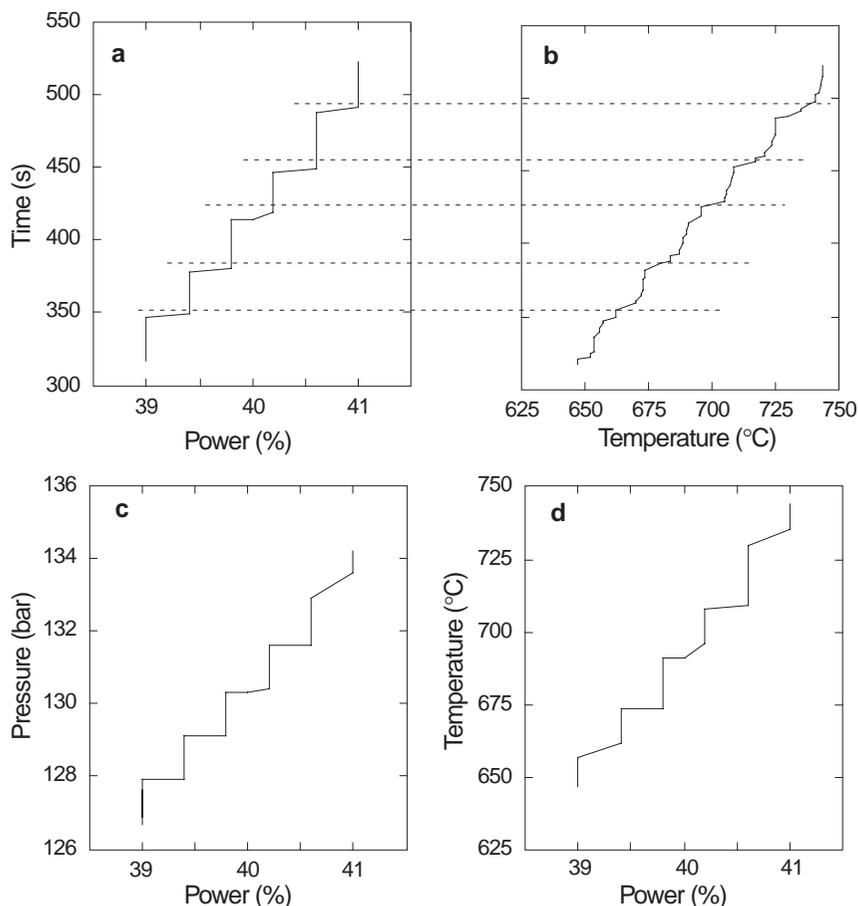
FIGURE 1. Response of the experimental assembly to power increments of 0.4% of the total power supplied to the experimental charge every 30 s.

controller was also connected to the computer, and measured pressures were recorded continuously and displayed in real time.

Materials used in the experiments

Solidus temperatures were determined at different pressures for pelitic gneiss, and for LiCl. A solidus curve for the gneiss was determined previously using the classical method; that is, detecting the presence or absence of melt in runs after reaction for several days. The mineralogical composition of this rock is approximately 42% Qtz, 20% Ms, 10% Bt, 18% Pl, and 8% Kfs. Details on its composition and melting relations are given in Castro et al. (1999). Its solidus curve is controlled by the breakdown of Ms in the presence of Qtz, Kfs, and Pl.

LiCl melts at temperatures lower than that of the NaCl pressure medium, and the *P*-T position of the solidus curve for LiCl is well known (Clark 1959; Bohlen 1984). Because LiCl is compacted at room temperature, a diamond trap (e.g., Hirose and Kushiro 1993) was used in these experiments.

RESULTS

Figure 2 shows the curves obtained from the melting experiments performed with the pelitic gneiss. Trends in the *P*-*t* curves exhibit distinct styles. A pressure decrease occurred at 6.5 kbar, 770 °C (Fig. 2a). Note that the *P*-*t* curve resumed its initial slope after several seconds, once the melt filled the pores. A second *P*-*t* result is a plateau during which pressure is nearly constant. This is the case for the gneiss solidii determined at 6.1 kbar, 752 °C, and 11 kbar, 800 °C (Figs. 2b and 2d). The plateau occurred over a time interval of about 150 s, the time needed to fill the pores of the melt-trap used in these two ex-
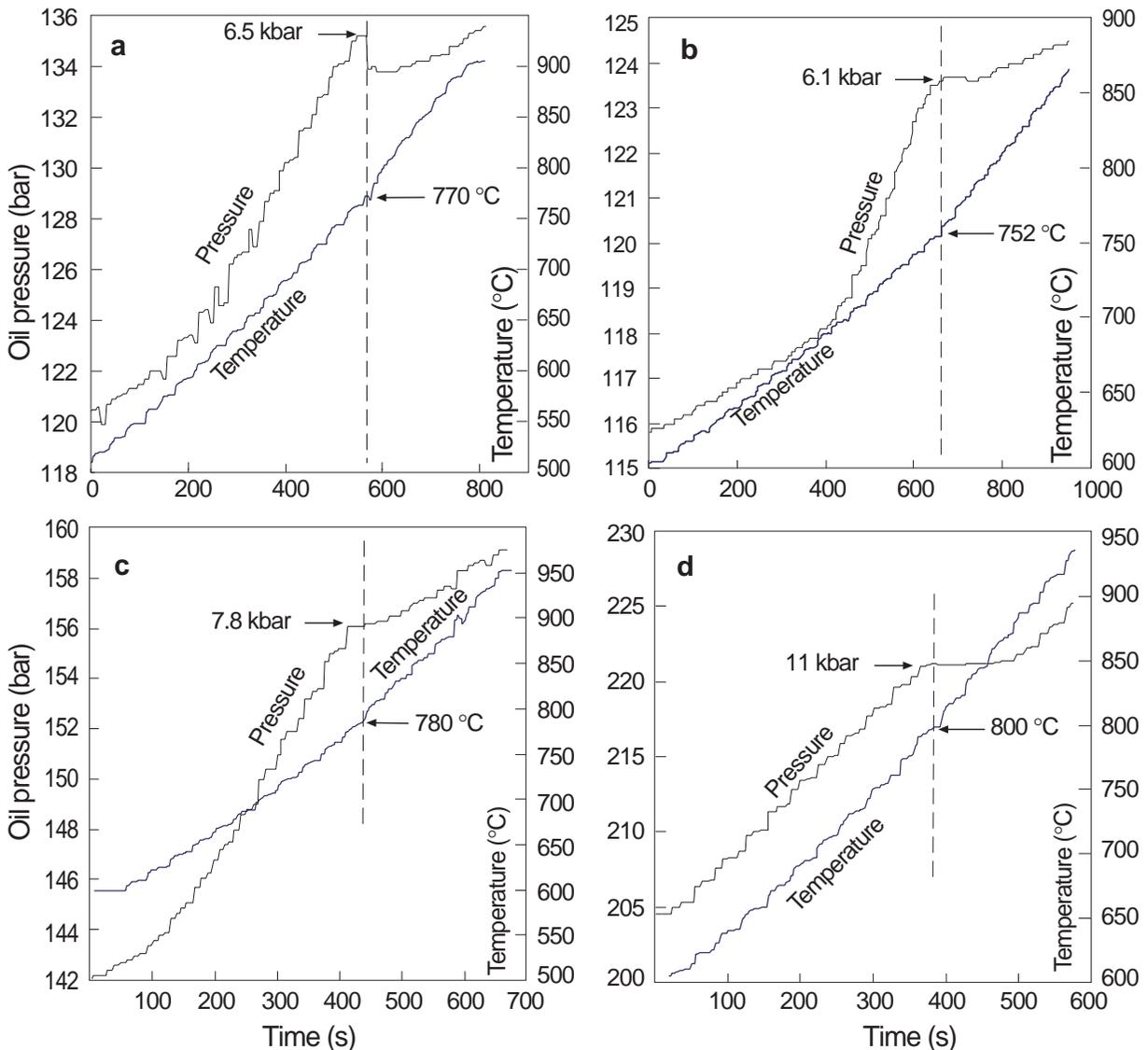
FIGURE 2. Pressure-time and temperature-time curves obtained for the Ms-Bt gneiss at four different experimental conditions. The melting point of the sample is indicated by changes in the pressure-time curves.

periments (diamonds in b and quartz grains in d). A third result is a change in the slope of the *P-t* curve. An example is the gneiss solidus determined at 7.8 kbar, 770 °C (Fig. 2c).

Similar results were obtained for the experiments with LiCl powder using a diamond trap (Fig. 3). The diamond trap filled slowly producing slight changes in the *P-t* curve. The effect indicated by the *P-t* curve in Figure 3d is a sigmoidal inflection in the slope at 9.6 kbar, 805 °C.

DISCUSSION

Interpretation of the pressure-time curves

The three experiments performed with the pelitic gneiss at *P* <10 kbar produced similar *P-t* curves. The rate of pressure increase is slower at the beginning of the experiment and faster near the melting point. After the pressure plateau (or pressure decrease), the rate decreases to a value close to the original one. These fluctuations are systematic, and reflect mechanical effects within the assembly during heating. For example, in the experiment performed at 6.1 kbar (Fig. 2b), the rate of pressure increase was ~5 × 10⁻³ bar/s during the first 500 s, and ~3 × 10⁻² bar/s from that point until the plateau was reached. After the

plateau, pressure increased at a rate close to that observed in the first 500 s. Similar results were obtained in the experiment performed at 6.5 kbar. The nearly constant slope of the temperature-time curves suggests that thermal equilibrium was reached within each power increment. However, this is not the case for pressure. The variations in the slopes of the *P-t* curves suggest that mechanical effects (dilatation and deformation) are not absorbed completely by the assembly within each time interval of power increment. Evidently, some of the effects propagate to the subsequent power increment, causing the slope of the *P-t* curve to increase. These accumulated effects are absorbed completely by the assembly during the plateau, and pressure increases resume at the initial rate. It is our view that these systematic slope changes are not artifacts of our experiments; rather, they are due to the mechanical responses of the assembly. Clearly, by making the time intervals longer, the mechanical effects could be dissipated completely by deformation of the assembly. However, results would not improve, because the main aim is to detect the temperature at which a plateau, or pressure decrease, is observed in the *P-t* curve. Consequently, the measured pressure during the plateau interval is the real pressure in the sample and, therefore, the pressure at which melting of the sample occurred.
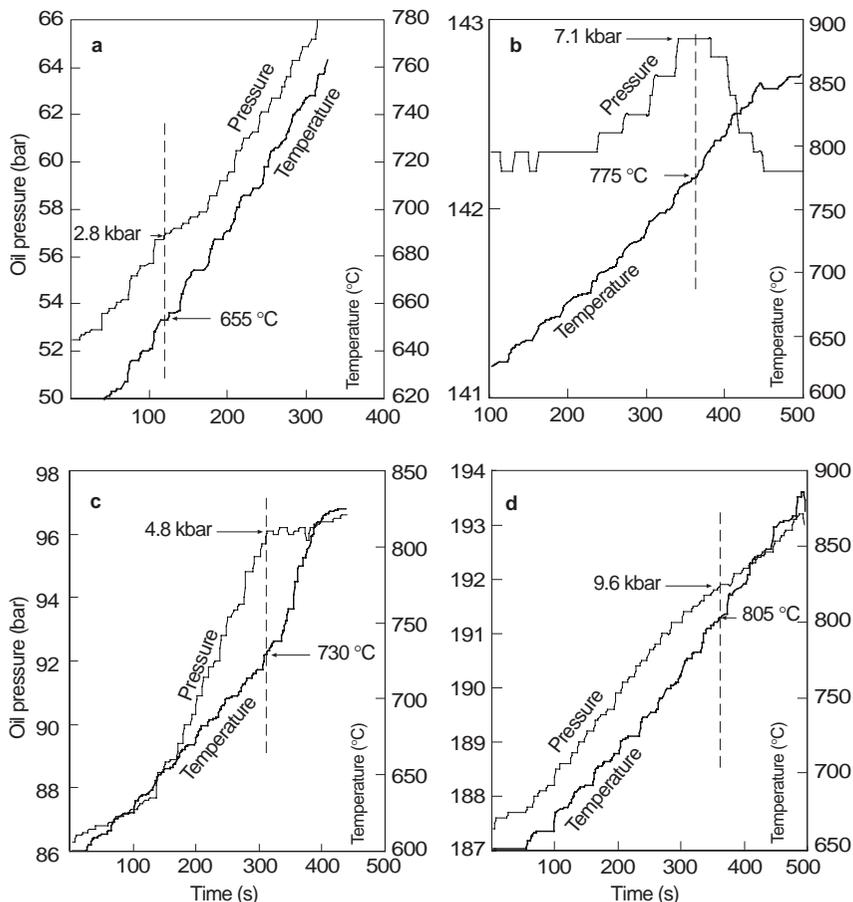
FIGURE 3. Pressure-time and temperature-time curves obtained for LiCl, using a diamond trap, at four different experimental conditions. The melting point of the sample is indicated by changes in the pressure-time curves.
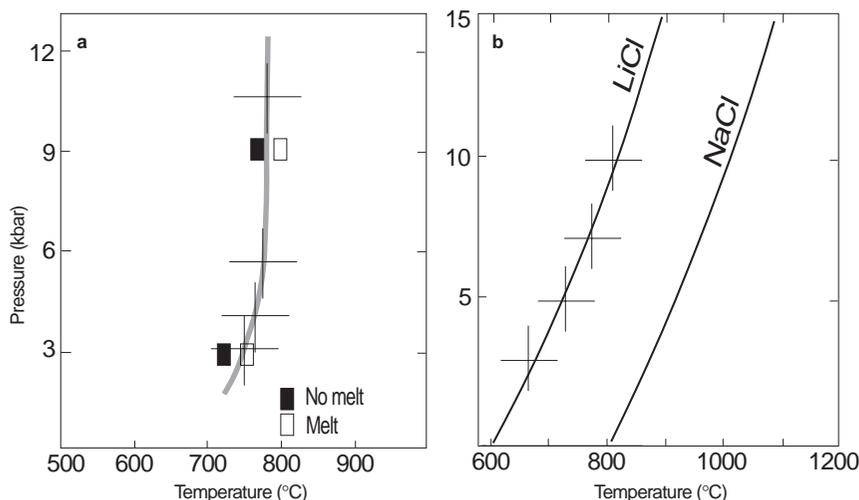
FIGURE 4. *P-T* diagrams showing the results of the incremental heating technique. (a) The fluid-absent solidus curve of the Ollo de Sapo gneiss (Castro et al. 1999) and (b) the melting curve of LiCl determined by Clark (1959) (cf. Bohlen 1984). Crosses represent to the *P-T* values obtained in this work. Boxes show the *P-T* conditions at which conventional experiments were carried out for the pelitic gneiss.

Solidus curves for the pelitic gneiss and LiCl

The *P-T* solidus determinations for the gneiss and LiCl can be used to locate the solidus curves in *P-T* space (Fig. 4). The curve for the Ollo de Sapo gneiss is compared with previous experimental results, in which the solidus temperature was determined by the classical technique observing the polished experimental runs with an SEM. In this classical technique the accuracy of the solidus temperature determination depends on the number of experiments necessary to bracket the melting point over a narrow interval. With the incremental heating technique described in this paper the determination is accurate to ~10 °C. In the pelitic gneiss used in this study the real solidus curve is closer to the no-melt point at 10 kbar and closer to the melt-point at 6 kbar (Fig. 4). The technique described here is more precise because it depends on a mechanical effect induced by the beginning of melting within the capsule. Based on observations of the experimental runs performed at near-solidus conditions, only about 1 vol% melt is required to produce the observed mechanical effects.

The technique also closely reproduces the solidus curve for LiCl determined by Clark (1959) (cf. Bohlen 1984). The behavior of this material differs considerably from silicates. The compaction effects are detected more readily in the pelitic gneiss. However, we note that the solidus points obtained by applying the technique to LiCl melting are very close to the previously determined solidus curve (Fig. 4). We conclude that the incremental heating technique is an accurate method for measuring fluid-absent solidii. The method permits the solidus *P-T* conditions of any silicate rock to be determined by performing a single, short-duration (2 h) piston-cylinder experiment.

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