MEASUREMENTS OF BUILDING MATERIALS BY TRANSIENT METHODS

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Abstract

This paper studies three contact transient methods for the measurements of the thermophysical properties of porous building materials – autoclaved aerated concrete and calcium silicate boards reinforced by cellulose fibers. The methods used were the pulse transient, transient plane source and the hot strip method. These methods measure three thermophysical parameters – thermal conductivity, thermal diffusivity and specific heat – in one single measurement. Data from these methods are analyzed and compared with results obtained by classical methods.

KEYWORDS: building materials, transient methods, specific heat, thermal diffusivity, thermal conductivity

1 Introduction

A general need of modern technology is testing methods that give reliable data of thermophysical parameters, preferably on small specimen in a short time, especially for materials used in building construction. Modern materials of this kind possess significant heterogeneity that could range from microns up to several tens of millimeters and extreme porosity that is the reason for high thermal insulation or low thermal conductivity that require long stabilization time. When studying such materials the variation of a broad range of specimen sizes and also the controlled variation of the moisture content in surrounding atmosphere to obtain reliable data is necessary.

Recently, it has been shown that a group of techniques known as Contact Transient Methods (CTM) should be suitable for this class of materials [1].In comparison with stationary or steady state methods the advantage of the transient methods is that some of them give a full set of thermophysical parameters within a single measurement, namely specific heat, thermal diffusivity, thermal conductivity or effusivity. Measurement regime, data evaluation as well as specimen geometry has to be optimized for these transient methods to obtain stable results differing only a few percents even when comparing the different measuring methods to each other [2,3,4,5].

The present contribution shows the application of the Pulse Transient method [6], the Transient Plane Source method [7] and the Hot Strip method [8] for the investigation of the thermal transport in building materials, namely in autoclaved aerated concrete (AAC - HEBEL) and calcium silicate board reinforced by cellulose fibers of industrial name

CalSil250 (CS). The measurements were compared with data from the Guarded Hot Plate method and the method of Regular Heating Regime [9,10].

For calcium silicate materials the dependence of thermophysical properties on moisture is very high even for the value of volume moisture that corresponds to the saturated sorption by the surrounding air of a given relative humidity.

2 Theory

2.1 Pulse Transient method

The principle of the Pulse Transient method and the standard specimen setup are shown in Figure 1. The heat pulse Q is generated by the passing of the electrical current I through the plane electrical resistor made of a 20 micron thick nickel foil. The temperature response is measured by a thermocouple. The thermophysical parameters are calculated from the parameters of the temperature response to the heat pulse (Figure 1).

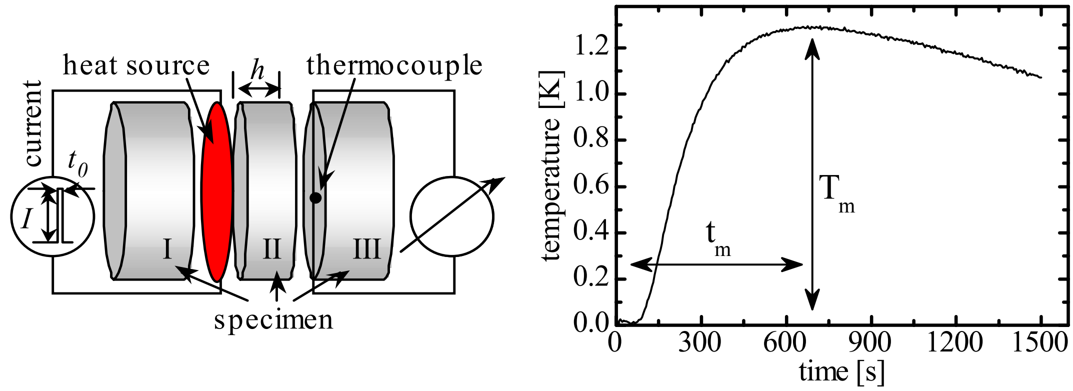

Figure 1. Principle of the Pulse Transient method and an example of the temperature response (CS 20mm thick).

The use of an ideal model gives the relations for thermal diffusivity

$$a = h^2/2t_m \,, \tag{1}$$

for the specific heat

$$c = \frac{Q}{\sqrt{2\pi e}\rho h T_m} \tag{2}$$

and for the thermal conductivity

$$\lambda = a\rho\, c = \frac{hQ}{2\sqrt{2\pi e}\; t_m T_m}\,, \tag{3}$$

where h is the thickness of the specimen, t_m is the time to reach the maximum of the temperature response, e is exp(1) = 2.718282, ρ is the density, T_m is the maximum of the temperature response, $Q = RI^2t_0$ is the pulse energy, and R is the electrical resistance of the heat source.

Optimal geometry of the specimen setup considering heat loss from free specimen surfaces and contact thermal resistance are taken into account. Then the change of the distance between the heat source and the thermometer h and the diameter of the sample gives the optimal set up. Then by the optimization of the specimen and sensor configuration using modified set up illustrated in Figure 2, the surface effects as well as the contact effects can be suppressed during measurement. This setup does not influence the principle of the ideal physical model, as the heat flux is symmetrical around the specimen axial direction. Compared to the standard setup, contact effects between the second and third part of the specimen setup can be avoided. With this modified specimen setup, measurements of various specimens thicknesses are easy to perform just by changing the thermocouple distance from the heat source. Only slight statistical differences in data between standard and modified specimen setup were previously found [7]. In the present modification, the ideal model of the pulse transient method in addition was modified to include the effect of heat losses from the free specimen surfaces (Figure 2, right). Theoretical background and the experimental details have been published in [6,2].

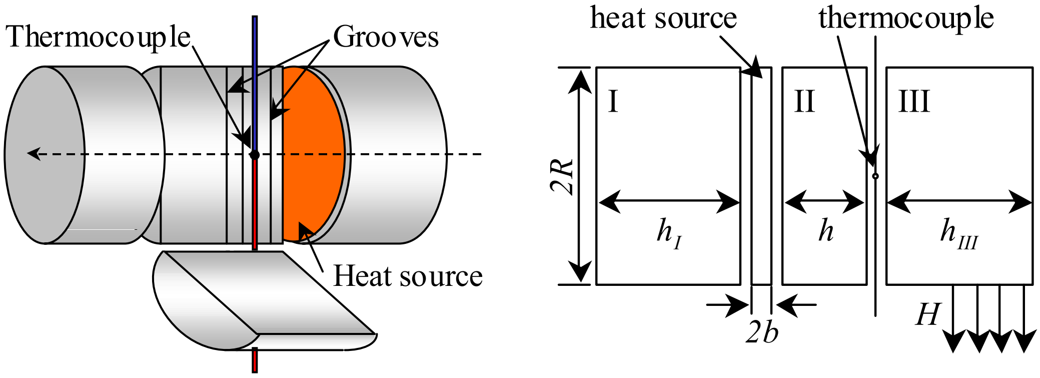

Figure 2. Modified specimen setup for measurements using various thicknesses of the specimen (left). Model for heat losses from the specimen free surfaces (right).

2.2 Transient Plane Source method

In the Transient Plane Source technique [7,11,12], a 12 micron thick double-spiral sensor made of a nickel foil is sandwiched between two identical pieces of sample material, *cf.* Figure 3. The sample material must completely cover the sensor probe. A step-wise heating power is applied to this sensor configuration, resulting in a transient temperature response that is simultaneously recorded by the same spiral heating elements. The double spiral thus gives a constant heating rate per unit spiral length, and simultaneously operates as a resistance thermometer, to record the average temperature response of the sensor.

Assuming the thermally heated region (which grows into the surrounding sample with a time-dependent thermal penetration depth (depth of probing) of $d_p = 2\sqrt{a \cdot t}$) never reaching the lateral or outer surfaces of the sample material, the average temperature response can be modeled as [3, 12]:

$$\Delta T = A + \frac{P_0}{\lambda \pi^{3/2} r} D(\tau), \qquad (4)$$

where $\tau = \sqrt{\frac{t - t_{corr}}{\theta}}$ is a dimensionless time, $\theta = \frac{r^2}{a}$ is the characteristic time of the experiment, t_{corr} is the time correction, P_0 is the output of power in the sensor, r is the sensor radius, λ is the thermal conductivity and a is the thermal diffusivity of the sample. Parameter A incorporates the imperfect thermal contact conditions in a measurement between sensor probe and the sample surface, making it possible to eliminate the influence of any thermal contact effects when performing analysis [12]. From this model, the thermal conductivity is obtained by plotting the temperature increase ΔT vs. $D(\tau)$ and extracting the conductivity from the slope of the best line-fit. The thermal diffusivity a and time correction t_{corr} are obtained by an iterative procedure producing the best model fit of Equation 4 to the experimental data points.

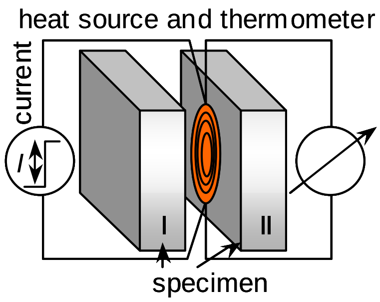

Figure 3 The Transient Plane Source technique utilizes a sandwich-sample setup configuration, completely covering the thin double spiral sensor probe.

2.3 *Hot Strip method*

The present Transient Hot Strip [8] configuration is based on the same principles as the Transient Plane Source technique. The present Hot Strip configuration consists of an array of 12 micron thick Nickel strips comprising a Hot Strip-shaped (9 x 60 mm^2) area that is capable of producing an essentially constant heating rate. In this technique, a slightly different model is used, in accordance with the original Transient Hot Strip technique theory (a single strip [8]):

$$\Delta T = A + \frac{P_0}{4\lambda\pi^{1/2}h} f(\tau), \qquad (4)$$

where $\tau = \sqrt{\frac{t - t_{corr}}{\theta}}$ is a dimensionless time, $\theta = \frac{d^2}{a}$ is the characteristic time of the experiment. Here, h represents the half length, and d represents the half width of the Hot Strip sensor.

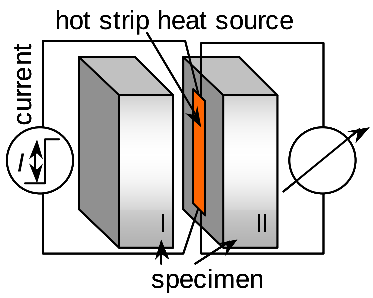

Figure 4 The Transient Hot Strip technique utilizes a sandwich-sample setup configuration, completely covering the thin strip sensor probe.

3 Experiments

Autoclaved aerated concrete (density 586.1 kg m^{-3}, porosity 0.8) and calcium silicate boards reinforced by cellulose fibers (density 280 kg m^{-3}, porosity 0.9) are building construction materials with porous structure. Specimen dimensions were 150 x 150 x 40 mm.

For the pulse transient method the heat source MINCO having the thickness 0.25 mm and electrical resistance of 11 Ω was used. A Chromel – Alumel thermocouple (100 microns in diameter) measured the temperature response. The thermocouple was placed into a groove made at various distances from the heat source. The contact effect for small thickness *h* and the heat losses from the specimen surface for large *h* were suppressed by optimization of the specimen geometry [3,2]. Stable data – not influenced by these mentioned effects – were found in the range of optimized thicknesses between 15 and 22.5 mm [2]. The pulse transient measurements were performed by instrument RTB 1.01 (Institute of Physics, SAS) at room temperature conditions, 25°C, where the relative humidity randomly varied in the range 20% to 85%.

The Transient Plane Source [7] (known as Gustafsson probe) uses the set of concentric rings for heating. In this case we used sources having outer diameter 12 and 30 mm. The probe resistance was 12 and 13 Ω. The used hot strip probe [8] has the dimensions 90 x 60 mm and a resistance of 10 Ω. In both the transient plane source and the hot strip techniques the same blocks of sample materials as in the case of pulse method were used. The probes were placed between two blocks of a given material, sufficiently deep inside the specimen in order to avoid heat reaching the outer sample surfaces (which would otherwise disturb the transient temperature response). The measurements were performed by the Transient Plane Source instrument (Chalmers University of Technology), which is also adapted for the hot strip technique.

4 Results

The data of thermal diffusivity, specific heat and thermal conductivity measured on AAC by the listed transient methods, Guarded Hot Plate method (GHP) and the method of Regular Heating Regime (RHR) are compared in Figure 5. Avg denotes mean averaged data measured by transient techniques. The statistical errors calculated for transient techniques include at least 5 measurements and are always below 3%. The statistical errors of average values (Avg) evaluated in % for all investigated transient methods are 5% for thermal diffusivity, 4% for specific heat and 3% for thermal conductivity. The corresponding results available in the literature [10] are included for comparison.

The measurements by GHP and RHR methods were performed on the sample of different batch of production and at relative air humidity 50% – corresponding to 2.7% of the mass to mass moisture content [10]. The recorded difference between the transient measurements and data from literature for specific heat and thermal conductivity is about 7 % and for the thermal diffusivity about 1%. This difference is probably caused by the fact that the GHP measurements were not performed on the same batch of materials. In addition, the moisture content was not precisely controlled when performing the transient measurements – the value of the relative humidity of the surrounding atmosphere was between 20% and 85 %.

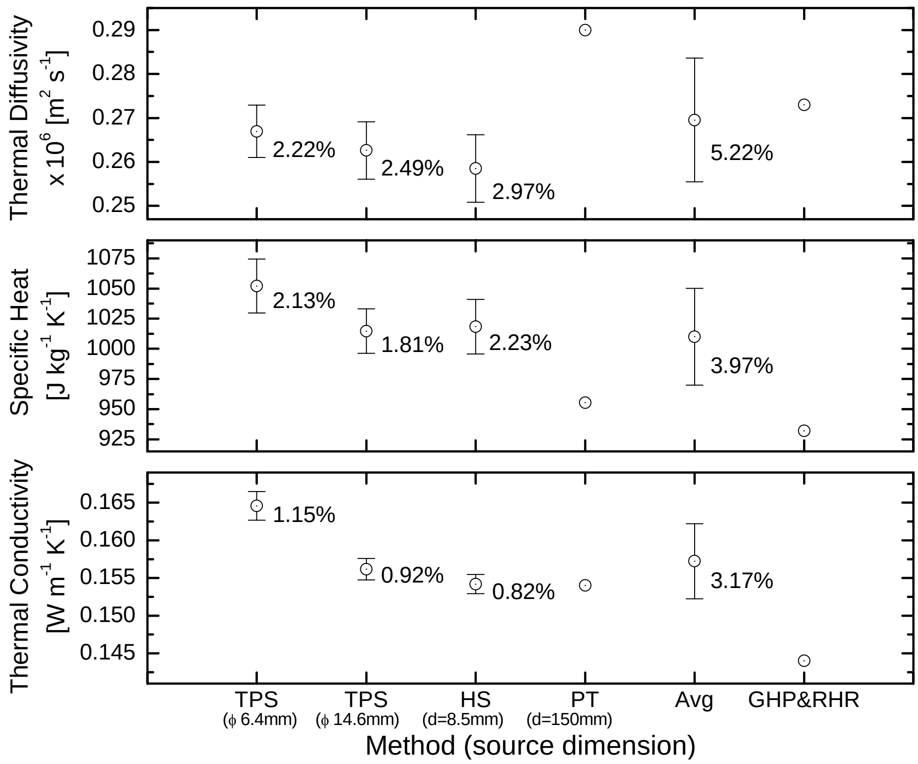

Figure 5. Thermophysical properties of autoclaved aerated concrete for various measurement methods.

For the calcium silicate board specimen, atmosphere humidity significantly influences the heat transport through the porous structure, and thus the macroscopic thermophysical properties. A significant difference was found in our measurements for two specimens - the virgin sample (Figure 6) and another one that was treated in a vacuum and subsequently aerated with atmosphere (Figure 7). In this case it appears that the moisture content in pores decreased rapidly by vacuum treatment. Environmental atmosphere and especially the relative humidity of surrounding air significantly influence the heat transport that appears to be a complex function of the material skeleton property and the gas in the pores [9]. The values of standard deviation calculated from at least 5 subsequent measurements are in Figures 6 and 7 represented by error bars. The corresponding statistical errors given in percentage are typed apart. The mean values of thermophysical parameters measured by all three transient methods and corresponding statistics are marked as Avg. For comparison the data of thermal conductivity measured by Guarded Hot Plate method – unfortunately on a dry sample of different batch – is shown. Data of thermal diffusivity and specific heat for vacuum treated specimen were not available (NA). The difference in the value of measured thermal conductivity for virgin specimen of CS could possibly be explained by the fact that GHP measurements were performed on well-dried material. Much closer are the values of thermal conductivity of material that was exposed to vacuum. Data for thermal conductivity measured after vacuum treatment (and thus with various moisture content) are shown in Figure 8. where thermal conductivity as a function of moisture content is plotted. Data were obtained by Guarded Hot Plate method.

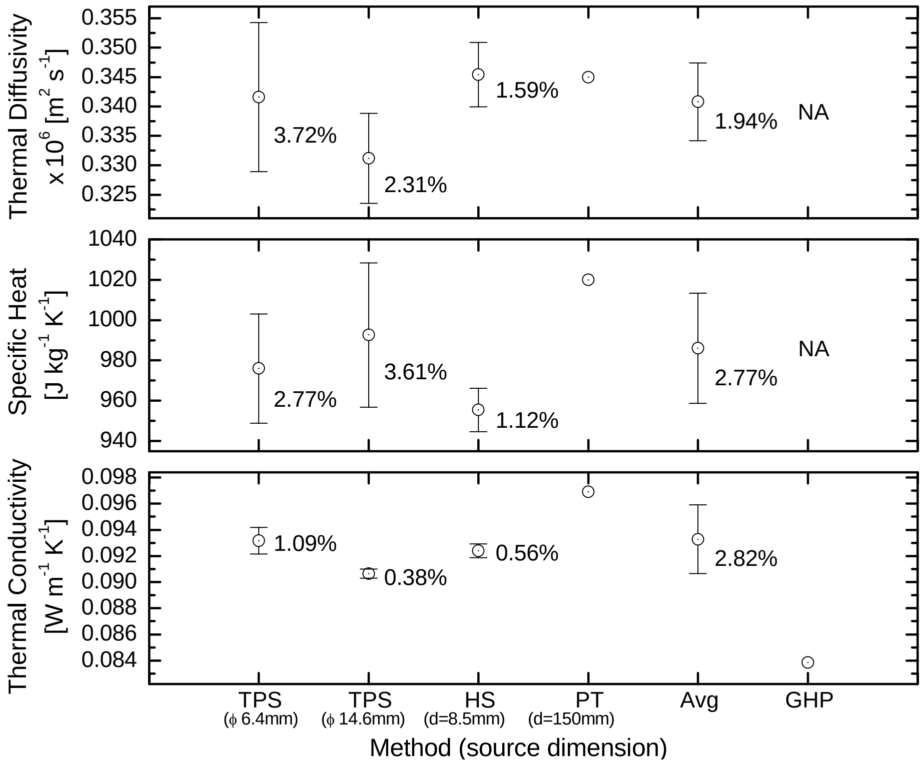

Figure 6. Thermophysical parameters of calcium silicate board reinforced by cellulose fibers measured by different techniques. (NA means Not Available)

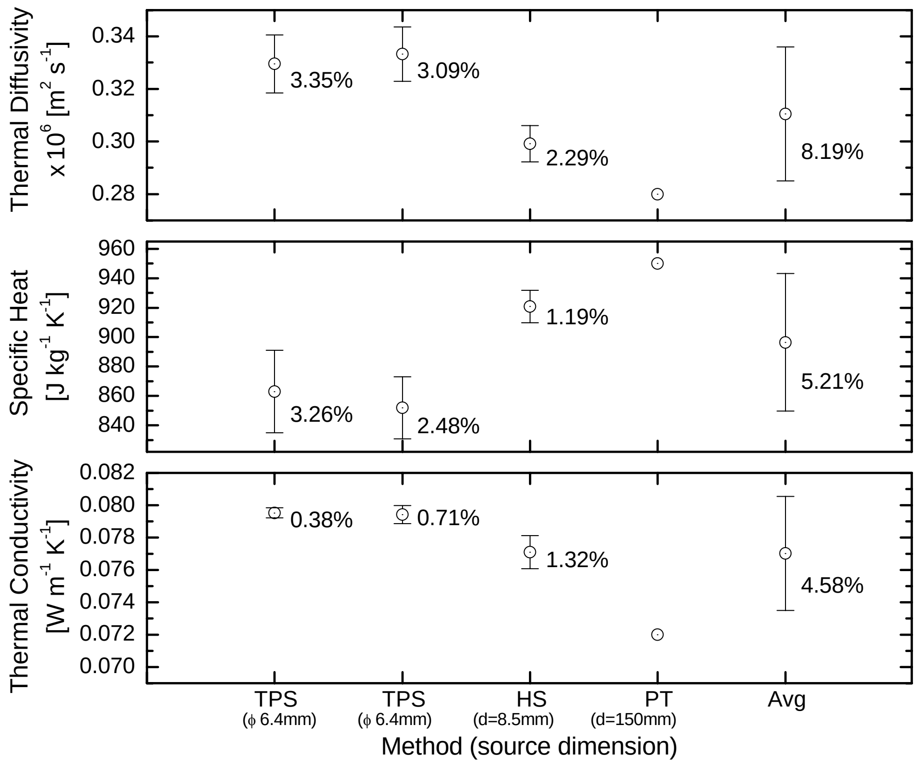

Figure 7. Thermophysical parameters of calcium silicate board reinforced by cellulose fibers measured by different techniques. Avg denotes mean averaged data measured by transient techniques. Material was dried in a vacuum and then aerated to a relative humidity of approximately 40-50%.

Avg in Figures 6 and 7 denotes mean averaged data measured by transient techniques. For a comparison the data measured by guarded hot plate, unfortunately on dry sample of different batch are shown.

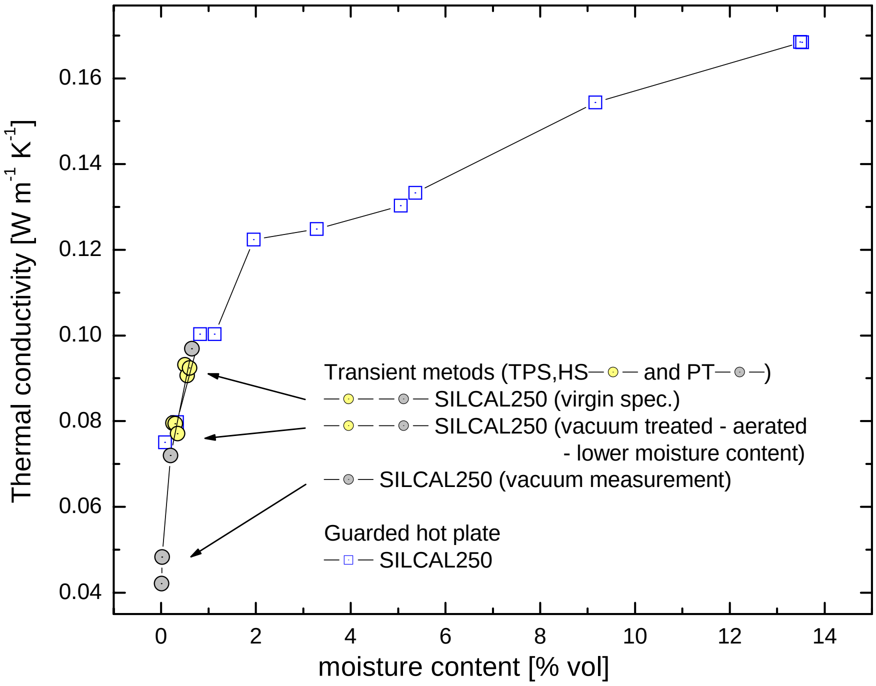

Figure 8. Comparison of thermal conductivity versus moisture content of Calcium silicate measured by guarded hot plate method [9] and by transient techniques.

5 Conclusions

The thermophysical properties of building materials – aerated concrete and calcium silicate board reinforced by cellulose fibers – were investigated by pulse transient method, transient plane source and hot strip method. Difference analysis methodology of data evaluation and optimized specimen geometry was used for the measurements. The free surface effects and contact effects were suppressed to improve data reliability.

Influence of the moisture content in surrounding atmosphere on the measured data was found. The data of thermal conductivity in Figure 8 confirm the coincidence of moisture effect with that one measured by guarded hot plate method. Clearly the mechanism of the heat transport is influenced by contribution of gas and moisture content in the pores.

Good agreement was found among data given by transient methods for these two inhomogeneous building materials. This confirms that published methodology of evaluation is a prerequisite for successful intercomparison measurements.

The difference between data measured by transient methods and data from literature (measured by Guarded Hot Plate and method of Regular Heating Regime) is small, regarding the fact that measurements were performed on different sample batches.

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