Design of a New DSC Cell with Tzero™ Technology

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ABSTRACT

A new DSC cell has been designed with Tzero™ technology and innovative integral cooling, to ensure maximum performance. The new cell design is balanced for thermal resistance, thermal capacitance, mass and heating rates between the sample and reference sides. In addition, thermal resistance between sensor and pans and pan mass are also addressed. This new cell allows a more fundamentally accurate determination of heat flow, provides better baselines, and excellent resolution, while supplying improved cooling.

INTRODUCTION

Heat Flux DSC was first commercialized in 1968 by TA Instruments (then part of The DuPont Company). Over the years, Heat Flux DSC has become the most popular type of DSC, with every manufacturer having a Heat Flux DSC in its product line. A heat flux DSC can be represented schematically as shown below:

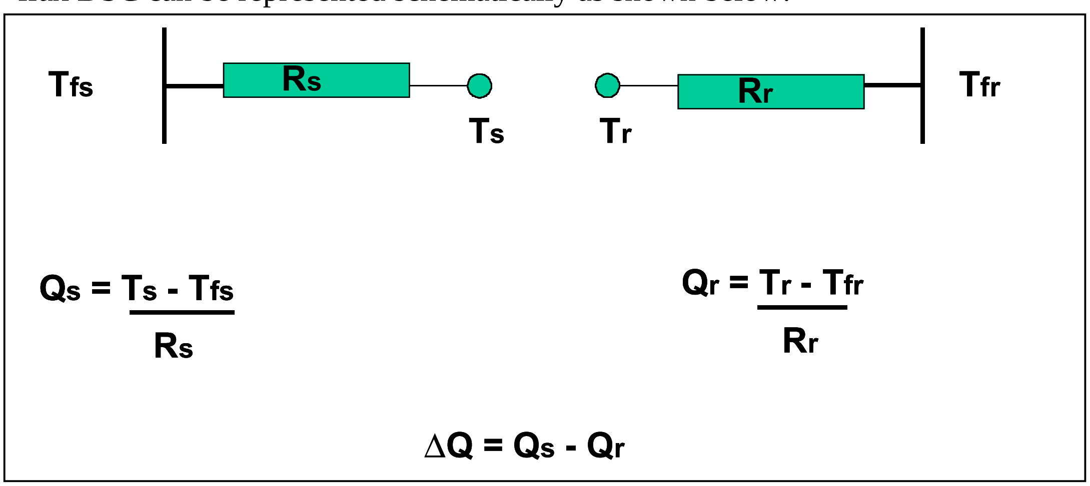

Figure 1 A simple schematic of a Heat Flux DSC

Where: Tfs= Temperature of the furnace on the sample side
Tfr= Temperature of the furnace on the reference side
Ts = Temperature of sample sensor

Tr = Temperature of reference sensor

Rs = Resistance between sample sensor and furnace

Rr = Resistance between reference sensor and furnace

Q = Heat flow

If the equation is rearranged, one gets:

$$\Delta Q = Qs - Qr = \frac{Ts - Tfs}{Rs} = \frac{Tr - Tfr}{Rr}$$

If the temperature of the furnace on both the sample side and the reference side are the same, and that the resistance of the sample to the furnace is equal to the resistance of the reference to the furnace, the equation becomes: $\Delta Q = \frac{Ts - Tr}{R} = \frac{\Delta T}{R}$

All DSC's on the market today, use some form of this basic equation.

This equation assumes some relationships instead of measuring them. An expanded view of the Heat Flux DSC Cell can be showed as below:

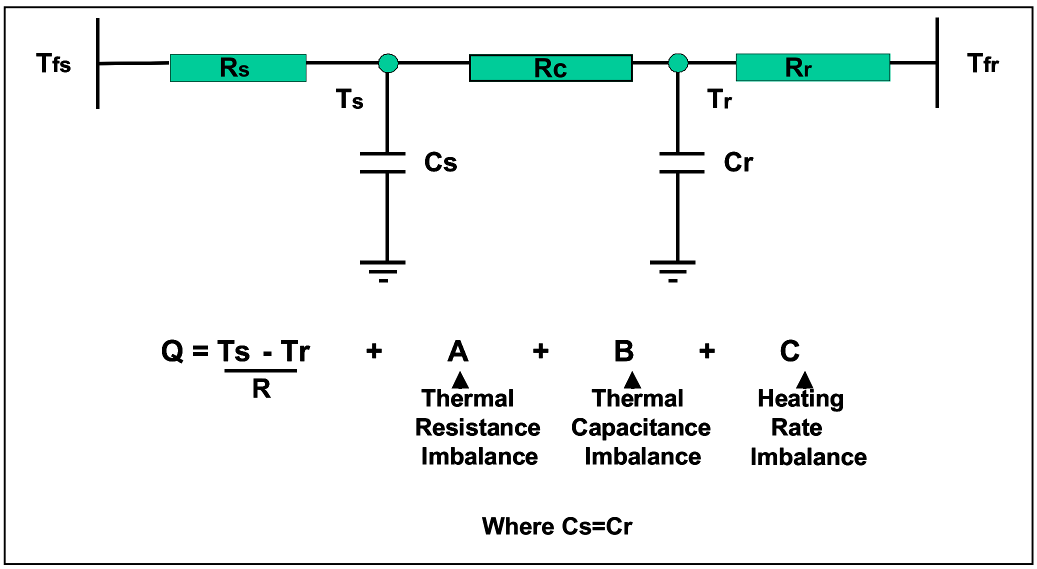

Figure 2 Expanded View of Heat Flux DSC

Figure 2, along with the corresponding equation, takes into consideration the resistance, capacitance, and heating rate difference that the basic equation makes assumptions about.

To make the measurements required for the equations in Figure 2, a new DSC cell was developed. A schematic of this DSC cell is shown in Figure 3.

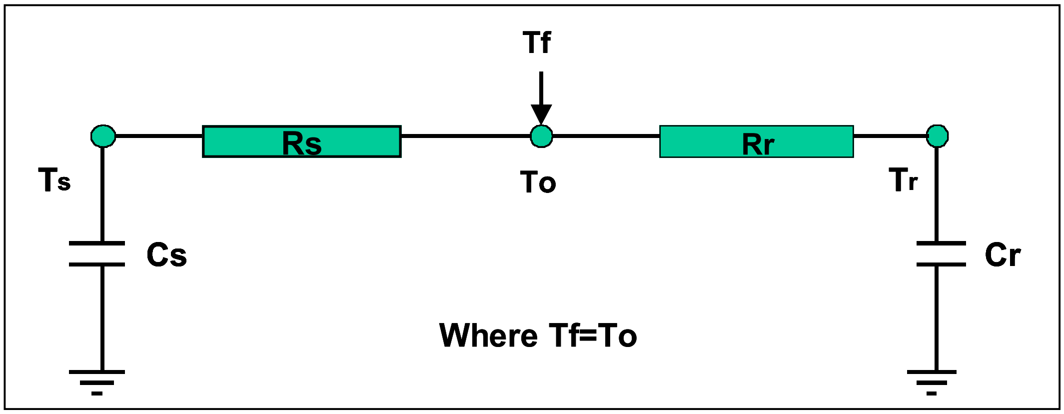

Figure 3 Schematic of Tzero™ DSC Cell

This new cell design permits the measurement of the actual values for resistance and capacitance of each sensor. This new cell has been redesigned in three of the most important parts of any DSC cells. These are the sensor, the furnace, and the integrated cooler.

SENSOR

While every part of a DSC cell is important, the sensor is the actual measurement device. In the Tzero™ cell the sensor is made of constantan, a copper-nickel alloy. Constantan is used because when it is paired with chromel to form a thermocouple, it generates more potential per change in temperature (µv/°C) than any other thermocouple over the same temperature range. This sensor is machined to exact dimensions ensuring that the resistances on both the sample and reference sides are as close as possible. The sample and reference platforms are machined to ensure flatness to provide minimum thermal resistance between the sensor and the pans. In the center of the sensor is a Tzero™ thermocouple. This thermocouple controls the furnace temperature, and provides precise temperature control due to the proximity to the sample location. Underneath the sensor on both the sample and the reference sides are Chromel Area Detectors. These chromel detectors are matched by weight and size. This keeps the mass balanced on each side of the cell. They are welded to the bottom of the sensor using sophisticated alignment fixtures, with sixteen welds per detector. This overall sensor is attached to a silver block to ensure even heating. A diagram of this is shown in figure 4.

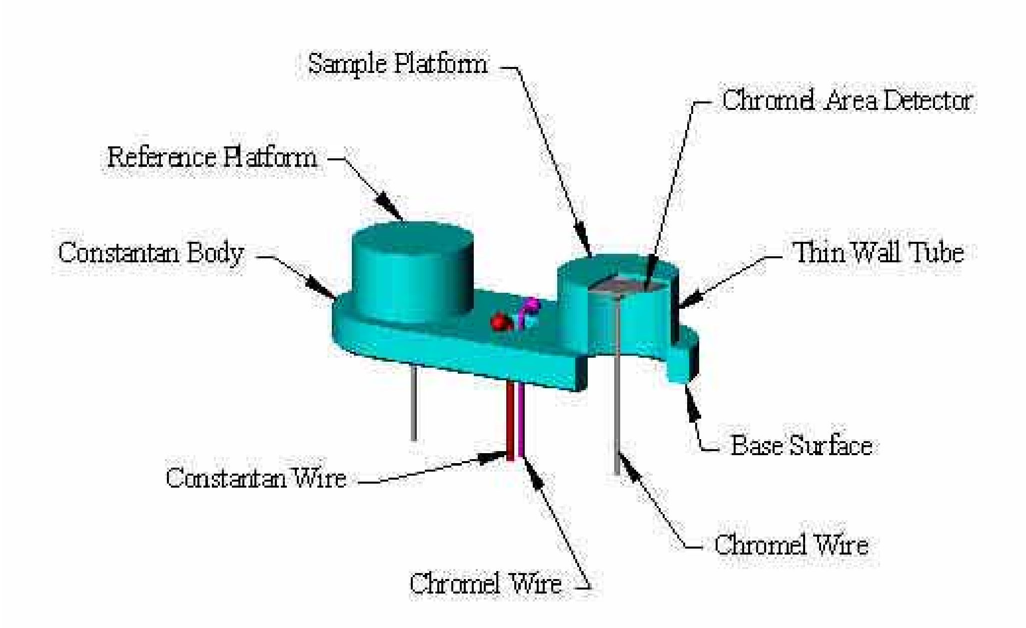

Figure 4 Tzero™ Sensor

FURNACE

One of the major differences between this new Tzero™ DSC and older Heat Flux DSC's is the location of the furnace. In the older Heat Flux DSC's the furnace surrounded the measurement chamber. In this configuration the heat flowed from the outside to the inside. In the Tzero™ DSC cell; the furnace is located below the measurement chamber. (See Figure 5). The heat flows through the silver block up to the sensor. Since the Tzero™ thermocouple controls the furnace temperature, the heat flows from the inside to the outside through the Tzero™ thermocouple.

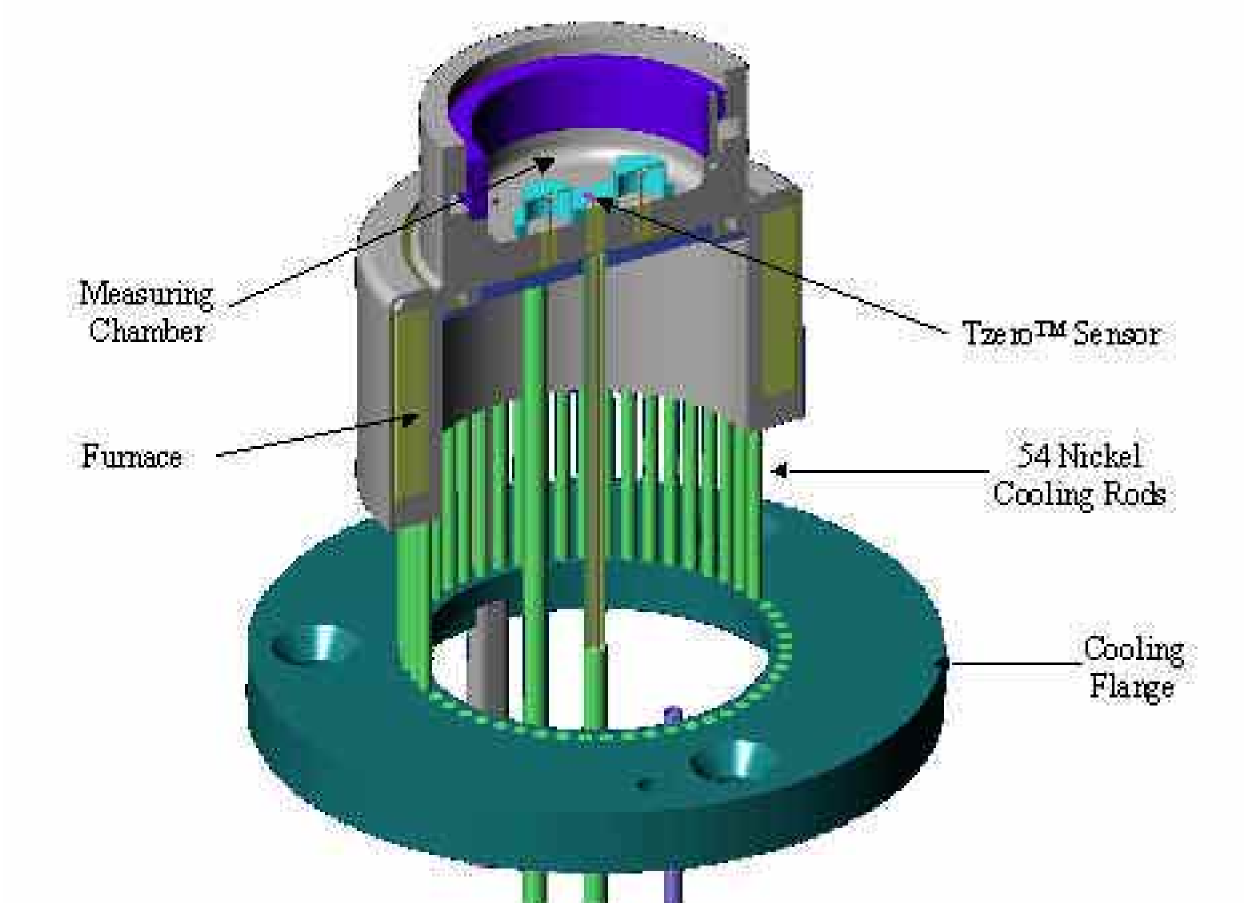

Figure 5 Cut-a-way of Tzero™ Cell

Integrated Cooler

In most DSC systems, cooling has always been added to the system as an afterthought. With the Tzero™ DSC Cell, cooling is designed integral into the system. As seen in Figure 5, this new cell is designed with a cooling flange, which is connected to the silver block with a symmetrical array of 54-nickel rods. On this cooling flange sits one of three different cooling units (see Figure #6). These cooling units have the following temperature ranges.

Liquid Nitrogen Cooling System	-180°C - 550°C
Refrigerated Cooling System	-90°C - 600°C
Finned Air Cooling System	25°C - 725°C

The cooling flange is used as a heat sink to guarantee rapid cooling rates, and stable baselines.

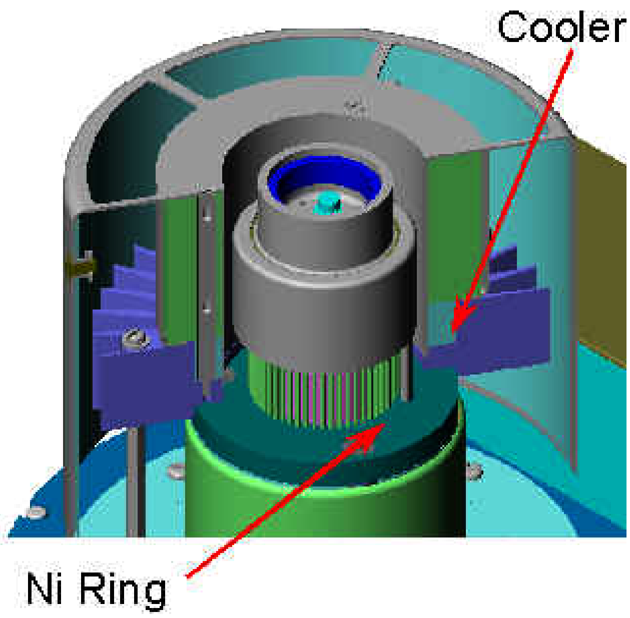

Figure 6 Integrated Cooler

RESULTS

Below are results that show the benefit of this technology. Compared are baselines of a conventional Heat Flux DSC to a Q1000 DSC using Tzero™ technology.

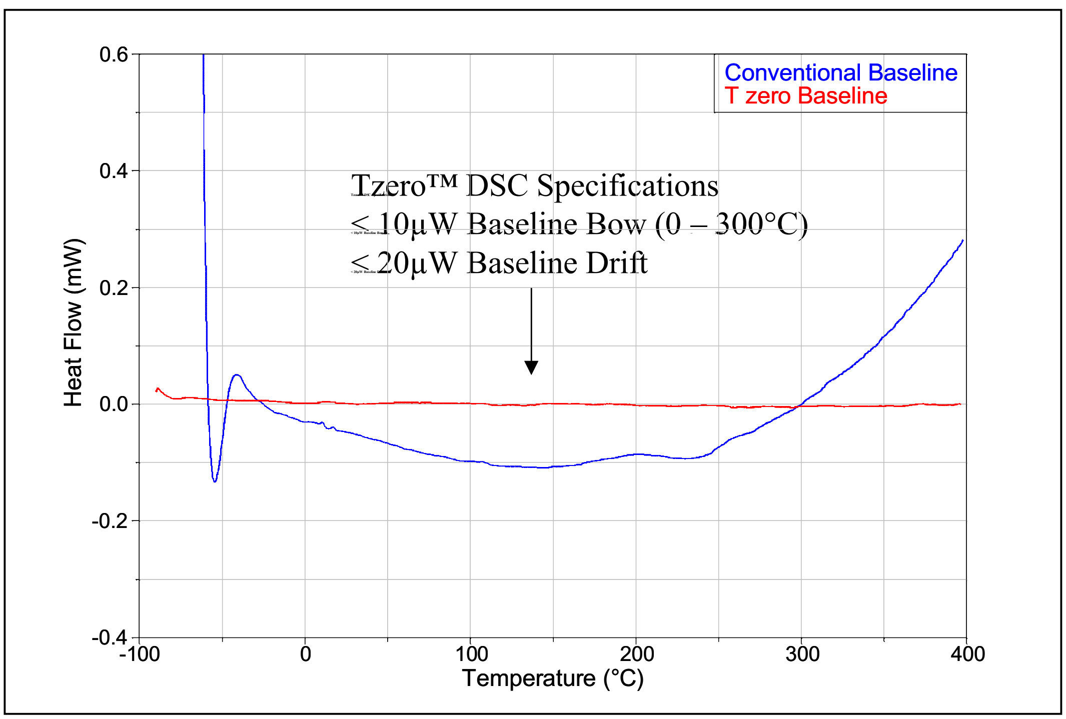

Figure 7 - Standard Heat Flux DSC vs. Tzero™ DSC

CONCLUSIONS

A new DSC Cell has been designed with Tzero™ technology. This DSC utilizes a new, innovative design that improves performance by measuring resistance and capacitance, instead of making assumptions like other DSC units. This technology is available in the Q100 and Q1000 DSC's.