to the energy storage (real) and energy loss (imaginary) portions of said at least one parameter.

The invention further comprises a method of analyzing a sample using a differential analysis apparatus comprising (i) subjecting a sample and reference to an externally applied disturbance, such as temperature change, in accord with a prescribed function comprising the sum of a linearly changing part and a periodically changing part, (ii) detecting a differential signal representative of at least one characteristic parameter of the sample, and (iii) processing said signal into components relating to the energy storage and energy loss portions of said at least one characteristic parameter.

DETAILED DESCRIPTION OF THE INVENTION

The present invention finds application in differential analysis methods and apparatus including, but not limited to, DTA, DSC, DDA, and differential photocalorimetry (DPC). While the following detailed description is provided with reference to DSC and DTA, the present invention is not limited to an apparatus comprising, or method employing, DSC and DTA. Further, a sinusoidal periodic function is adopted for the following description. However, the invention is not so limited, and any periodic function can be substituted for the sinusoidal function. The term "prescribed function" as used herein means any function which comprises the sum of a linearly changing part and a periodically changing part. The periodically changing part includes, but is not limited to, a sinusoidal function, a saw tooth function, a square wave function, or a pulse wave. While all periodic functions can be characterized by a period or frequency, certain periodic functions, such as sinusoidal functions, are also characterized, in addition, by an amplitude.

The linearly changing part can have a positive (heating), negative (cooling), or zero slope. In the case where the slope is zero, the prescribed function includes "isothermal functions" in which the sample and reference are subjected to a periodic temperature variation such that during the scan the average temperature of the sample and reference remains essentially constant.

FIG. **1** illustrates a portion **10** of a DSC, for example, a Perkin-Elmer Corporation DSC-7 type of calorimeter, which can be used to implement the present invention. The user's manual to the DSC-7 calorimeter, entitled "Users Manual, 7 Series/Unix DSC7, Differential Scanning Calorimeter," is herein incorporated by reference. This instrument measures the differential power required to keep both sample and reference sensors **44**, **54** at the same temperature throughout the DSC experiment. The apparatus, as illustrated, is described and explained in basic terms in E. S. Watson et al., "A Differential Scanning Calorimeter for Quantitative Differential Analysis, " Anal Chem. 36(7), 1233–1238 (1964), which is herein incorporated by reference.

In FIG. **1**, reference and sample containers **56** and **58**, respectively, are mounted on platforms **52** and **46**. The reference generally is a standard (or simply the empty container) and the sample is a material having some characteristic parameter to be compared with that of the standard. As used herein, the term "characteristic parameter" means any property representative of the sample which is measured differentially with respect to the reference. Characteristic parameters include, but are not limited to, differential power input, differential heat flow, differential temperature, dielectric constant, and differential weight loss.

The reference **56** and sample **58** are subjected to a programmed heating or cooling program, in accord with a prescribed function, through a process of programmed and balanced heating. The programmed heating or cooling run subjects the sample and reference to an externally applied disturbance. The term "applied disturbance", as used herein, means a physical stress applied to both the sample and reference which permits, in accord with the differential technique used, the measurement of a characteristic parameter of the sample. In DSC and DTA, the applied disturbance is heat which induces a change in temperature (though not a change in average temperature if an isothermal function is used). In DDA, the applied disturbance is an electrical field. In DPC, the applied disturbance is a lightwave.

Both the programmed heating and the balanced heating are performed through the reference heater **50** and the sample heater **48** in the reference and sample bases **52**, **46**. The heaters are powered with alternating current, and on one half-cycle the power supplied to both heaters is controlled by the temperature programmer **60**. On the other half-cycle, however, a different power is supplied to each heater to nullify any temperature differential between the sample and the reference as sensed with the platinum resistance thermometers **54**, **44** in the container bases. Thus, the heating system has two control loops, one responding to the temperature program and the other responding to the different energy requirements of the sample and reference. The average temperature amplifier **62**, average temperature computer **64**, recorder **68** and differential temperature amplifier **66** interact to maintain the two control loops as explained with respect to FIG. **1** in the following paragraph. The instrument responds very rapidly so that the deviation of the sample temperature from the reference temperature is negligible, and therefore the sample temperature follows the predetermined program even though it may undergo a thermal event (such as a phase or glass transition).

The system of FIG. **1** can be divided into two separate control loops, one loop for average temperature control and the other for differential temperature control. In the average temperature control loop, the programmer **60** provides a signal which is proportional to the desired temperature of the sample holder **58** and the reference holder **56**. The programmer information is also relayed to the recorder **68** and appears as the abscissa scale marking. The programmer signal reaches the average temperature amplifier **62** and is compared with signals received from resistance thermometers **54**, **44** via the average temperature computer **64**. If the average temperature is greater than the temperature called for by the programmer **60**, then the power supplied to the sample and reference heaters **48**, **50** is decreased, and vice versa if the average temperature is less than that called for by the programmer **60**.

In the differential temperature control loop, temperature signals received from the resistance thermometers **44**, **54** are relayed to the differential temperature amplifier **66** via a comparator circuit (not shown) which determines whether the sample or reference temperature signal is greater. The differential temperature amplifier **66** responds to a disparity in the sample and reference temperature signals by adjusting the differential power increment fed to the sample and reference heaters **48**, **50** to correct the temperature difference. A signal proportional to the differential power is sent to the recorder **68**. The recorder **68** relays the differential signal to computing device **69** which processes the signal to provide the user with the characteristic parameter of the sample. Such computing devices include any appropriate commercially-available device, including desktop personal computers, such as the Perkin-Elmer Unix 7-Series data station.