

of the apparatus allowed for the X-ray detection and observation of the two events at the same temperature and elucidated the phenomena that the thermal transition at 185° C. was associated with two events of opposite heat flow (i.e., exo- and endotherm).

Example 5

A multicomponent product containing a blend of inorganics, organics and polymers was analyzed by the apparatus of the invention. In addition, the experiment was run so that the temperatures, atmospheric environment and heating times and rates simulated those of the commercial process. The sample was heated rapidly and cooled rapidly in a cycle from 23°-300° C. The entire experiment took 90 minutes. The DSC data show 3 events. Prior art comparisons of the multicomponent product to standards of the individual materials comprising the product could only identify the glass transition of the polymer. The other two events, an exotherm and an endotherm, could not be identified by comparison to standards. The DSC/XRD experiment showed that the exotherm was a crystallization of an organic in the polymer matrix. The endothermic transition was shown by the X-ray diffraction data to be the dissolution of the organic in the sample matrix. The dissolution of the organic in the product occurred 70° C. below the melting point of the pure organic. When the experiment was conducted at either a different heating rate or under a different atmosphere, the exothermic and endothermic transitions were shifted by as much as 40° C. Therefore, to identify the structural nature of the thermal transition, both X-ray diffraction and calorimetric data had to be acquired simultaneously. Prior art instruments could not simulate either the speed or the temperature control of the apparatus of the invention. The experiments also showed how complex mixtures could be analyzed and how the chemical interactions among the components of the mixture (i.e., the in situ crystallization and dissolution 70° C. melting point) can be elucidated by the apparatus of the invention. Once again this analysis was critical since other experiments have shown that the impact strength of the product is affected by how the components blend in the mixture.

Example 6

Several copper compounds and copper compounds blended with additives were analyzed by the DSC/XRD apparatus of the invention for potential catalytic uses. The experiments usually consist of three parts: first, a careful preheating of the material in a controlled atmosphere (sometimes N₂, sometimes oxidative gas mixtures), second, reduction in a mixed H₂/N₂ atmosphere and finally, a catalyst regeneration program which involves both oxidation and reduction.

The DSC/XRD instrument provides careful temperature control in all phases of the experiment. In catalytic studies, this control can prevent unwanted runaway exothermic reaction (as in the reduction of metal catalysts). In the preheating stage, the DSC/XRD instrument provides precise measurement of thermal decomposition by correlating the DSC data with the observed X-ray diffraction patterns. On a multicomponent mixture, the correlated data identifies which material is being thermally changed and the magnitude and rate of that transition. Catalysts are commonly composed of the active material, a multicomponent substrate and other materials such as binders and pelletizing lubricants.

In the reduction experiments, which may be run isothermally, the DSC data indicate the start and the completion of the reductive exotherm. This is important since the X-ray diffraction data are a result of bulk transitions and are not sensitive to small changes which can be seen in the DSC data (i.e., the initiation of the reduction and the very last steps of the completion of the reduction). In general, X-ray diffraction methods are sensitive to crystalline changes of one percent by total weight. The DSC data can detect noncrystalline changes in the material and some changes below one percent. The XRD data are used to determine which material or materials are being reduced. As in Example 3, experiments have been run where the reductive exotherm at an elevated temperature has been a combination of the simultaneous reduction of CuO, Cu₂O and a copper salt to Cu (metal) all in one step. Experiments have been run where > 50 percent of the total reducible (or oxidizable) materials have been reduced (or oxidized) in less than 5 seconds. Therefore, the speed of the apparatus of the invention results in measured reaction rates with thermal-structural material identifications which have not been previously identified or measured.

In all phases of the catalytic cycles (oxidation, reduction, regeneration), times and temperatures can be optimized by the use of the invention. For example, if a high surface area catalyst is desired, the apparatus of the invention can be used to optimize the aforementioned cycles to get the desired physical properties in the shortest preparation time or in the best cost effective manner.

What is claimed is:

1. An instrument for studying structure of a sample material in a controlled environment and simultaneously studying energy changes in the sample material as a function of controlled temperature or atmospheric change, the instrument comprising:

a differential analyzer having means for determining energy changes in a sample material in comparison to a thermally heated reference contained in said analyzer and as a function of controlled temperature or atmospheric change, the analyzer having a sample holder assembly forming an enclosure relative to an external atmosphere which contains a thermally heated sample holder and which provides an environment in which the sample material is physically isolated from the external atmosphere so that a controlled temperature and atmospheric environment can exist about the sample material, a path defined through the sample holder assembly to and from the sample holder which is substantially transparent to X-rays to allow a beam of X-rays outside of the sample holder assembly to be impinged on the sample material within the sample holder assembly and to be diffracted effective for detecting the diffracted radiation, and

an X-ray diffractometer including a source of an X-ray beam to impinge on the sample material through said sample holder assembly, and a position sensitive detector arranged for receiving diffracted radiation from the sample material.

2. An instrument according to claim 1 in which a monochromator is arranged with the X-ray beam source to provide a focused monochromatic beam.

3. An instrument according to claim 2 in which the X-ray source is a line source equipped with a Guinier diffraction system and a curved focusing crystal monochromator.