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SCTA APPLICATION TO POLYMERS

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Abstract

In SCTA application to high polymers, we do not find remarkable and illustrative results such as found in multiple step dehydration processes of low molecular mass substances. However, some characteristic results were obtained in the application to high polymers, and three examples are described in this report.

Ceramic sintering process

In production of ceramic components for electronics devices, the ceramic powder is premixed with plasticizers, such as polymeric binder and other additives, and the mixture is molded into specific shape to form a so-called green body. The green body is, in general, heated at a constant rate for firing; burnout of the polymer and additives (or debinding) proceeds first and then sintering of the ceramic powder follows. However, in these processes crack formation sometimes happens and characteristics are not uniform in the components.

For producing high-quality products and optimizing the production lines, SCTG is applied [1, 2]. Because the mass loss rate is controlled, controlled rate burnout of polymeric binder, etc. can be made by SCTA. Thus, crack formation is avoided and uniform components are obtained without the formation of carbon residue. The temperature profile in the SCTG is applied to the temperature control in the production line, and high-quality products are produced.

Two examples were reported; one is alumina [1] (written in Japanese) and the other is tungsten carbide. In the former one alumina powder was premixed with poly(vinyl alcohol), poly(ethylene glycol) etc. In the latter one tungsten carbide with tantalum carbide and cobalt was mixed with polyvinyl resin etc. Optimum temperature profile for both the mixtures was obtained by SCTG. The burnout processes were examined also by TG-DTA-EGA (mass-spectrometry). The sintered products of the metal carbide are checked by electron microscopy.

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Estimation of activation energy

It was sometimes reported in the case of constant rate heating TG that the estimated activation energy is dependent on the sample mass. One example was reported for poly(butylene terephthalate) [3]. The estimated activation energy by Ozawa–Flynn–Wall method increases with increase of the sample mass from 160 to 200 kJ mol^{-1} for the sample mass from 1 to 16 mg. SCTG were made for 10 mg sample at different controlled mass loss rates and the activation energy estimated from the results by Friedman–Ozawa method is 163 kJ mol^{-1} [4], which corresponds to the activation energy obtained for the 1 mg sample by constant rate heating TG.

The authors of the paper [4] postulated that in SCTG the atmosphere is roughly controlled, because the mass loss rate is controlled, so that thermal decomposition process in SCTG proceeds under similar atmosphere to that for small mass sample by constant rate heating TG. The temperature gradient is also reduced in SCTG.

The decomposition mechanism reflects in the shape of the temperature profile in SCTG and SCTG data were further examined in detail for kinetic analysis by using the estimated activation energy. Similar kinetic analysis was made for SCTG data of poly(ethylene terephthalate). Thermal decomposition mechanism for the polymers is both random scission of main chain of the polymer.

Long term thermal endurance evaluation

For the thermal endurance evaluation, we should observe decomposition process in a low temperature range, because the decomposition processes in a practically applied low temperature range sometimes differ from those in high temperature range, and thus the constant rate heating TG is inappropriate for long term thermal endurance evaluation of polymeric materials. For this purpose we must find the low temperature range in which the mass loss rate can be measured within allowable error. By SCTG an instrument automatically searches for a suitable temperature range for a programmed mass loss rate. However, one of drawbacks of SCTA is the difficulty in estimating reduced time, and the reduced time is necessary for the thermal endurance evaluation. Therefore, the searched temperature range is used in the determination of a temperature range especially for repeated temperature scanning method [5].

References

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