Research regarding behavior simulation when heating metallic materials

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Keywords

Metallic material, heating, behavior simulation, DSC

1. Introduction

Obtaining information regarding the behavior of metal materials when heated, reheated, heated then cooled, including maintaining them at a high temperature is very important when designing equipment that will be exploited in harsh, even hazardous conditions. Results of the calorimetric analysis combined with creep testing can be used to identify inadmissible issues when used in real exploitation conditions.

Thermal analysis is important for a wide range of industries such as polymer and composite production and processing, pharmaceutical products, eatables, oil, organic and mineral chemical products; it offers information regarding weight loss, on the possibility of dimension or mechanical properties change function to temperature.

Characterized proprieties include melting, crystallization, transitions and reticular phases, oxidation, decomposition, volatilize, thermal expansion coefficient and module. [1]

Calorimeter (DSC) NETZSCH works on the principle of thermal flux, equipments are characterized by a symmetrical tridimensional design with homogeneous heating. Sensors have a high calorimetric sensibility, a short time response and a measurement enclosure without condensation that guarantees a high sensitivity signal. [2]

DSC is the general term for the following measurement methods:

DSC heat flow

A technique in which the sample heat, made out of the sample and reference material, varies in a specified program, and the heat difference between the sample and the reference material is measured function to the temperature.

DSC with power compensation

A technique in which the heat difference is applied on the sample and reference material on a time unit; it is measured as a temperature function to equalize it, while the samples hear, made out of sample and reference material, varies in a specified program [3].

Figure 1 presents the DSC block diagram; heat flow comprises the sample and reference support, heat tolerance, radiator and heater. In the sample we introduce heat from the heater and the reference heat through the radiator and resistance. Heat flow is proportional with the difference of heat between the radiator and the supports. The radiator has sufficient heating power compared to the sample. In the case in which in the sample take place endothermic or exothermic, such as transition or reaction, this phenomenon is compensated by the radiator. Thus the heat difference between the sample and the reference material is maintained constant. The difference between the heat provided to the sample and reference material is proportional to the heat difference of both supports. By calibrating standard material, quantity measurement of the unknown sample is achieved [2].

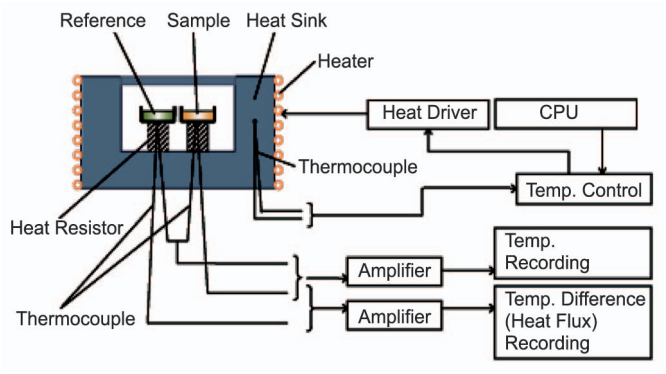

Figure 1. Block diagram DSC heat flow.

The technical TG application in which the sample mass is monitories function to time and temperature, while the sample heat, in a specified atmosphere, is programmed.

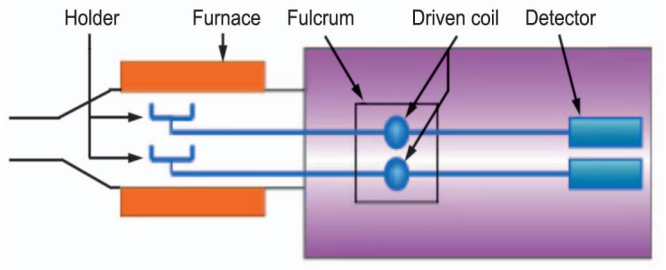

Figure 2. Block scheme of the horizontal differential of TG / DTA.

Figure 2 presents the balance guides for the sample and reference material, these being situated in the heater. Sample and reference mass are measured separately on the drive coils, sensitively calibrated. Mass difference is transmitted as TG signal. By differential mass measuring, effects of the beam expansion, convection flux and floating force are canceled. Thus we accomplish the highly sensitive thermogravimetry measurement. Measuring the sample and reference mass by the independent coils allow a slight adjustment of the initial

electric TG deviation. Also each support has a thermocouple that allows the simultaneous exit of the DTA signal.

TG can be used to analyze thermal decomposition, oxidation, dehydration, heat resistance and kinetic analysis. By combining the two measurement techniques we can obtain a various amount of information from a single sample. [3]

2. Experiments

The calorimetric method, above mentioned, was used to develop and accomplish two, new, intelligent brazing materials that deposit different functional roles at a single melt, namely coated rods for brazing VAg25SnSiPR and VAg40SnR [4].

2.1. Experimental program

Thermal analysis of the studied samples was made using the Simultaneous Thermal Analysis – STA, namely equipment STA 449 F3 made by Netzsch, Germany. The simultaneity of these analyses is achieved due to the DSC-TG (Differential Scanning Calorimetry-ThermoGravimetry) applications with which the equipment is equipped.

2.2. Work plan

From each type of electrode, a sample of the core (uncoated electrode) was taken, the VI Ag40SnR electrodes have been marked with white and the electrodes VI Ag25SnSiPR with yellow.

Each sample was heated at 1100 °C and cooled to 50 °C in an inert gas / argon atmosphere in two steps:

• in the first stage, any non-metallic components of the core alloy were removed (by combustion, evaporation, etc.);

• in the second step we operated on the clean sample from the first stage.

2.3. Equipment, work parameters and results

Differential Scanning Calorimetry (DSC), namely Differential Scanning Calorimetry (CSD) - is based on the measurement of enthalpy changes due to changes in the physical and chemical properties of the material, depending on temperature or time, in which the measured parameter is the flow rate, to or from the sample within a pre-set temperature program.

Elements of interest - among other things - are the melting behavior/ crystallization, phase transition detection, etc. The parameter of interest is temperature.

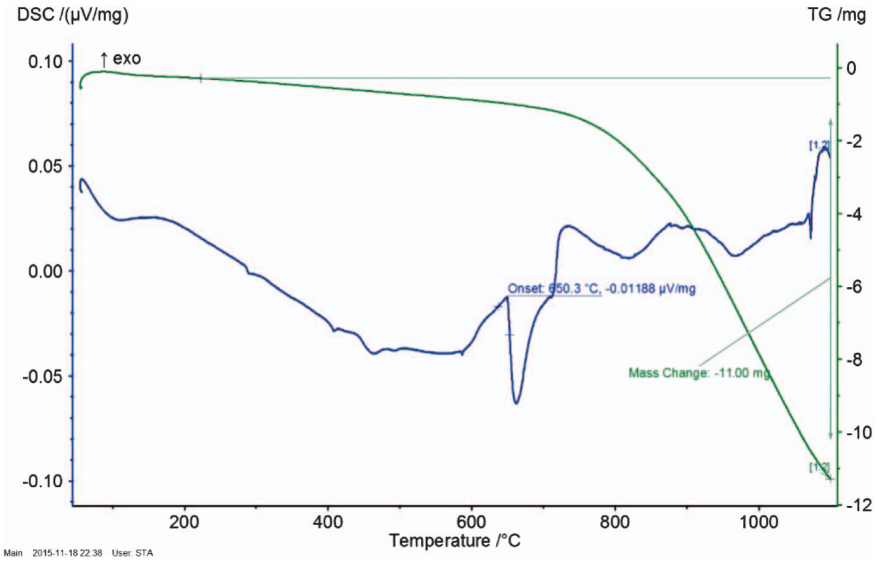

Figure 3. DSC-TG graphic for the white sample (VI Ag40SnR) when heating in the first stage.

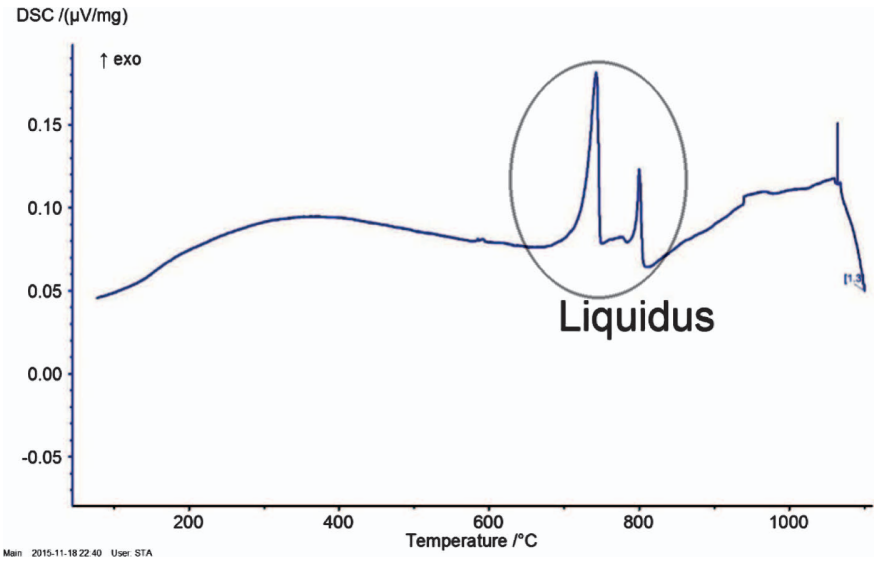

Figure 4. DSC-TG graphic for the white sample (VI Ag40SnR) when cooling in the first stage.

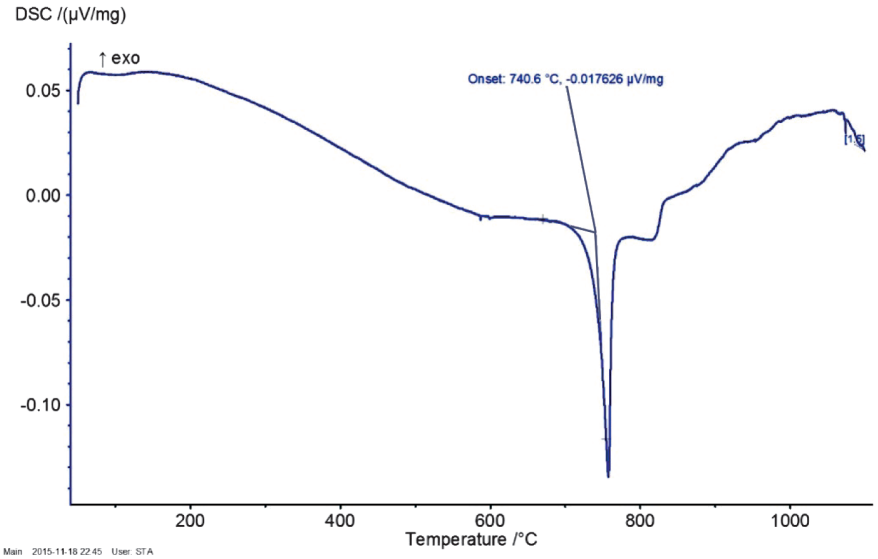

Figure 5. DSC-TG graphic for the white sample (VI Ag40SnR) when heating in the second stage.

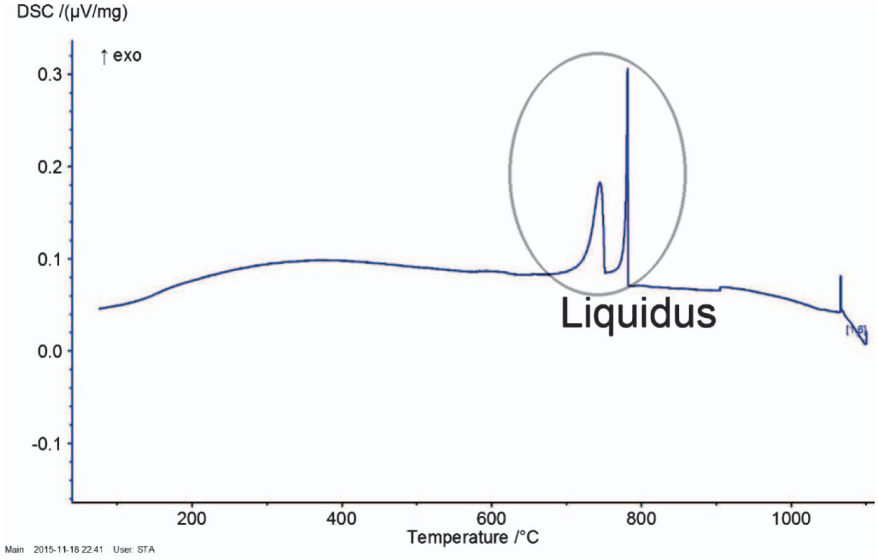

Figure 6. DSC-TG graphic for the white sample (VI Ag40SnR) when cooling in the second stage.

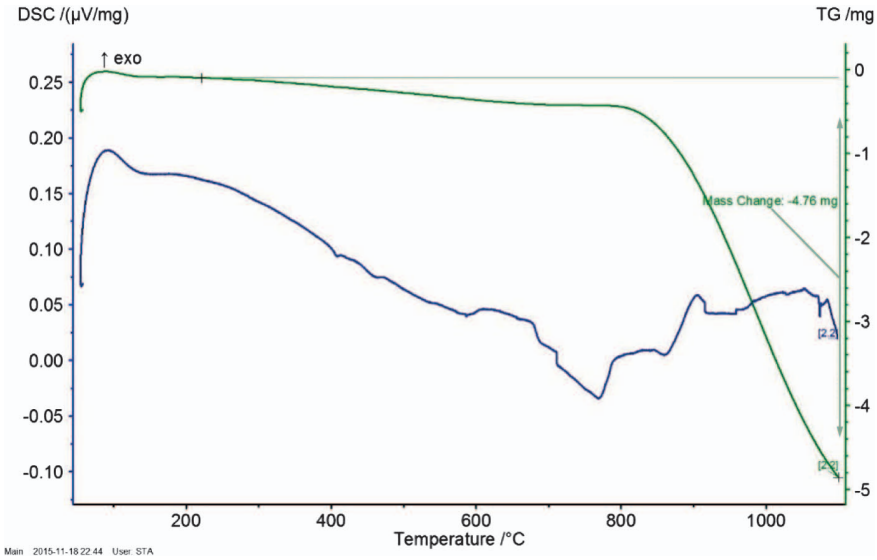

Figure 7. DSC-TG graphic for the yellow sample (VI Ag25SnSiPR) when heating in the first stage.

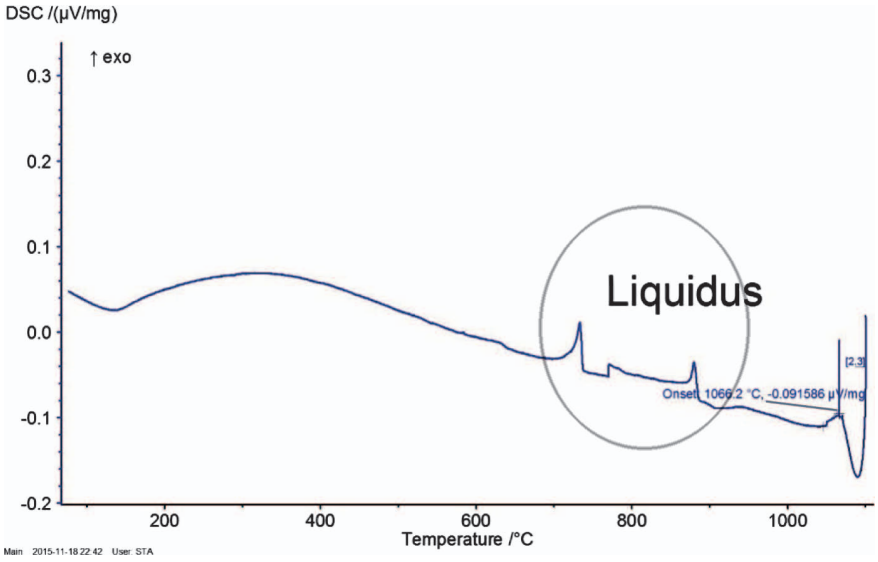

Figure 8. DSC-TG graphic for the yellow sample (VI Ag25SnSiPR) when cooling in the first stage.

The ThermoGravimetry (TG)application, namely the Thermal Gravimetry (GT) - is based on the continuous recording of changes in the mass of the material sample, based on a combination of temperature and time; and, in addition, to the pressure and composition of the gas. The parameter of interest is mass change.

Calibration of sample temperatures is achieved by melting standard samples of Sn, Ag, Au in alumina crucibles.

Simultaneous thermal analysis was performed by heating at a rate of 10 °C/min, 5 (five) minutes of isothermal maintenance at 50 °C and zero minutes of maintenance at 1100 °C, then immediately cooling to 50 °C; the cooling parameters were identical to the heating ones. The entire heating cycle - heating-cooling - is performed in a controlled atmosphere enclosure, respectively an Ar stream with a flow rate of 50 ml/min. A 50 mg sample was used in all thermal analysis experiments.

After the completion of the first step, the second stage of the experiment was carried out in identical conditions, namely without lifting the furnace lid and without disturbing the initial position of the sample.

The analysis performed was in accordance with specifications in ISO 11357, ISO 11358, AST M E967, ASTM E 968, ASTM E 793, ASTM D 3895, DIN 51004, DIN 51006, DIN 51007, where structural changes and material losses can be observed. [5]

3. Result analysis

First step. For both samples, a multitude of exo and endothermic events are observed. These are the result of "evaporations" of materials as well as multiple phase transformations. Mass loss at over 780 °C represents an order of magnitude of about 20%.

It is observed that melting begins at 650 °C in the white sample (VI Ag40SnR), (figure 3) and 711 °C in the yellow sample (VI Ag25SnSiPR), (figure 7).

Stage two. For both samples it is observed that the alloy has a fairly wide liquids range: 700-860 °C for the yellow sample (VI Ag25SnSiPR), (figure 8), and 650-800 °C for the white sample (VI Ag40SnR), (figure 6).

The melting point and material losses are approximately the same order of magnitude: 740.6 °C / 0.017626 mg for the white sample (VI Ag40SnR), (figure 5) and 730.5 °C / 0.011101 mg for the yellow sample (VI Ag25SnSiPR), (figure 9).

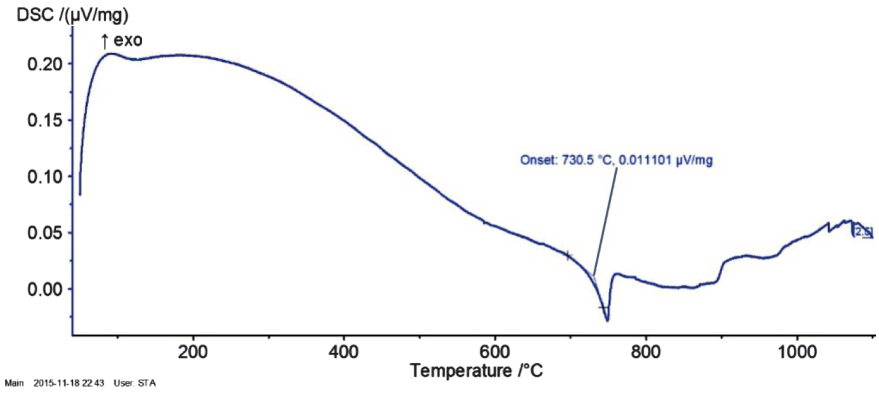

Figure 9. DSC-TG graphic for the yellow sample (VI Ag25SnSiPR) when heating in the second stage.

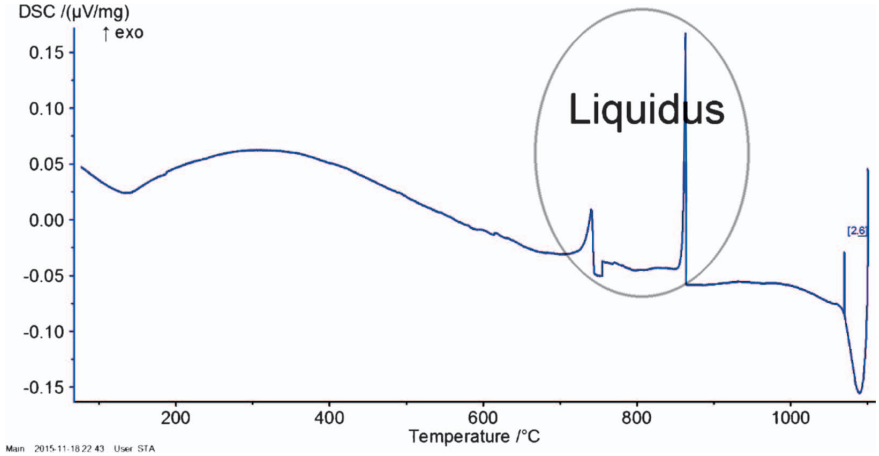

Figure 10. DSC-TG graphic for the yellow sample (VI Ag25SnSiPR) when cooling in the second stage.

Conclusion

Experiments performed had the effect of modifying the coating recipes for the brazing rods VI Ag40SnR and VI Ag25SnSiPR, demonstrating the viability and applicability of the analysis for a new process of obtaining coated rods for brazing.

Loss compensation in chemical elements discovered by analysis was compensated by additional alloying deposits from the coat.

The melting temperature of the verge, experimentally determined, was used to prepare the coating recipe on the principle of melting it with approx. 500 °C, under the melting temperature of the verge.

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