Activation Energy Determination of High-Test Peroxide Decomposition in Inconel 718 Crucibles

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Abstract

In this work, investigations on the applicability of the "green propellant" high-test peroxide (HTP) as coolant in regeneratively cooled space propulsion systems were conducted. For this purpose, the activation energy of the exothermic decomposition reaction of 97.2 wt% HTP was determined in passivated high-pressure crucibles made of *Inconel* 718 by differential scanning calorimetry (DSC). Based on the *Kissinger method*, the DSC measurements were analyzed and an activation energy of $E_a = (79.5 + 9.6) \text{ kJ/mol was}$ calculated. This activation energy corresponds to literature values known for the decomposition reaction of HTP without a catalytic effect. It can therefore be assumed that passivated *Inconel* 718 has no significant catalytic impact on the decomposition reaction of HTP.

1. Introduction

Since the 1960s, storable liquid propellant combinations based on hydrazine or its derivatives monomethylhydrazine (MMH) or unsymmetrical dimethylhydrazine (UDMH) as fuel and nitrogen tetroxide (NTO) as oxidizer have been used in many different bipropellant space propulsion systems of upper stages, manned or unmanned spacecrafts and satellites [1–3]. However, these conventional storable propellants have a considerable hazard potential for humans and the environment. On the fuel side, hydrazine and its derivatives are extremely toxic and carcinogenic substances [4]. Due to its carcinogenic properties, hydrazine was even added to the *candidate list of substances of very high concern* under the European REACH (registration, evaluation, authorization and restriction of chemicals) regulation in 2011 [5]. This makes it possible that the use of hydrazine (and perhaps also its derivatives) will be banned in Europe in the near future and will no longer be available as fuel for space propulsion systems. On the oxidizer side, NTO-based propellants are also extremely toxic and have a very high vapor pressure (1000 mbar at 20 °C [6]), so that these substances pose a particularly high risk of inhalation exposure [4]. Therefore, when handling these conventional storable fuels and oxidizers, time-consuming and expensive safety procedures are necessary. For example, during the fueling of a spacecraft, all employees involved must wear special protective clothing with an external air supply, known as SCAPE (self-contained atmospheric protective ensemble) suits. To enable a more environmentally friendly, safer and cost-reduced space transport in the future, "green" storable propellants have therefore been investigated worldwide for several years. Compared to the conventional storable propellants, their green alternatives have a significantly reduced hazard potential. As a result, only much simplified safety procedures are necessary when handling these propellants, which drastically reduces time and costs.

Highly concentrated hydrogen peroxide, also referred to as high-test peroxide (HTP), is currently considered one of the most promising green substitutes for the conventional storable oxidizers in bipropellant space propulsion systems. At a comparable level of performance, HTP has a lower toxicity and a significantly reduced vapor pressure (1.9 mbar at 20 °C [7]) than NTO, which simplifies the handling procedures of this substance enormously. Also the *European Space Agency* (ESA) has HTP identified as a suitable green oxidizer. Based on a propellant trade-off analysis, ESA selected 98 wt% HTP as oxidizer for its future green storable kick stage *LunaNova*, which is currently being developed within the *Future Launcher Preparatory Programme* (FLPP) [8, 9]. Space propulsion systems which use HTP propellant combinations operate typically at a high oxidizer-to-fuel mixture ratio (ROF) for an operating point in the range of maximum specific impulse (approx. ROF = 5 - 8 [10]). This provides a significantly higher oxidizer mass flow compared to the fuel. In addition, HTP has a high specific heat capacity (2.63 kJ/(kg K) [11]). Considered together, these two properties make HTP very interesting for an application as coolant in regeneratively cooled space propulsion

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systems beyond its use as an oxidizer. In the thrust chamber of a space propulsion system, the combustion temperature of the propellants is much higher than the melting temperatures of most chamber wall materials, so cooling is necessary. One of the most commonly used cooling methods is regenerative cooling, where the fuel or oxidizer (sometimes both propellants) is passed through cooling passages located in the thrust chamber wall before being injected into the combustion chamber. In these cooling passages, the propellant absorbs thermal energy from the thrust chamber wall and preventing its temperature from exceeding a critical value that would cause the thrust chamber to fail. The most challenging part of using HTP as coolant in such a regenerative cooling is its exothermic decomposition [11]:

$$H_2O_2(l) \longrightarrow H_2O(l) + \frac{1}{2}O_2(g); \qquad \Delta_r H^0 = -98.1 \text{ kJ/mol.}$$
 (1)

Under normal ambient conditions, the rate of this decomposition is very slow (approx. 0.5 wt%/year [12]). However, if the HTP absorbs thermal energy in the cooling passages of a regeneratively cooled space propulsion system, above a certain amount of absorbed energy, a self-accelerating decomposition begins. A large heat release in the cooling passages is the result. This so-called *thermal runaway* leads to local hotspots, so that the thrust chamber is no longer cooled sufficiently and will be destroyed. For this reason, such a thermal runaway must be avoided absolutely. The amount of absorbed energy at which the self-accelerating decomposition begins can be estimated with the activation energy of the decomposition reaction of HTP shown in Equation (1). The activation energy is the minimum amount of energy which is necessary to start a chemical reaction. For HTP, this activation energy depends on the HTP itself (e.g. concentration, purity or stabilizers) and the surface in contact with it (e.g. material compatibility, surface finish or surface to volume ratio) [12, 13]. For a safe and reliable application of HTP as coolant in a regeneratively cooled space propulsion system, the determination of its activation energy is a fundamental condition. This work focuses on the activation energy determination of the decomposition reaction of HTP which is in contact with conventionally manufactured and passivated surfaces of the nickel-based alloy *Inconel 718*. This is a material commonly used for thrust chambers due to its high strength, corrosion resistance and good weldability.

2. Materials and Methods

2.1 Differential Scanning Calorimetry

Differential scanning calorimetry (DSC) is a widely used thermal analysis technique that quantifies the amount of heat absorbed or released from a sample during a heating process [14]. DSC is characterized in particular by the fact that only very small sample quantities of a few milligrams are required for the measurements [15]. This reduces the hazard potential for the user when preparing and carrying out the measurements, especially in the case of energetic substances such as HTP. In this work, the measurements were conducted by *heat-flux differential scanning calorimetry*, one of two common used DSC methods, and were used as data base for the activation energy determination of the decomposition reaction of HTP.

2.1.1 Basic Principle

As shown schematically in Figure 1, a heat-flux differential scanning calorimeter consists of a cylindrical furnace that completely encloses a sample carrier at its center. This sample carrier has two platforms arranged symmetrically to its center axis, where a crucible is placed on each of them. It is very important that these two crucibles have the same design and are made of the same material. During a measurement, a purge gas flows around the sample carrier and the crucibles. The sample to be analyzed is placed in one of the two crucibles (*sample crucible*). The other crucible (*reference crucible*) contains an inert reference substance. During the DSC measurement, the furnace heats the crucibles according to a previously defined heating rate β , so that both crucibles are equally subjected to the temperature profile T(t). The temperature of the sample and reference crucibles is measured by two temperature sensors mounted on each of the two sample carrier platforms. From these two measured temperatures the temperature difference $\Delta T(t) = T_s(t) - T_r(t)$ is calculated. If an endothermic or exothermic process takes place in the sample, the temperature difference has a value unequal to zero (endothermic process: $\Delta T < 0$, exothermic process: $\Delta T > 0$). This temperature difference is proportional to the heat flow $\dot{Q}_s(t)$ which passes into or out of the sample. This heat flow can be determined from the measured temperature difference using a proportionality factor K(T(t)) as follows [16]:

$$\dot{Q}_s(t) = K(T(t)) \cdot \Delta T(t).$$
⁽²⁾

The heat flow related to the sample mass is usually referred to as *DSC signal*. If this DSC signal is plotted against temperature, it is very simple to identify the temperature range in which a endothermic or exothermic process has occurred in the sample.



Figure 1: Schematic structure of a heat-flux differential scanning calorimeter [17].

2.1.2 Differential Scanning Calorimeter

In this work, the DSC measurement campain was carried out with the differential scanning calorimeter *NETZSCH STA* 449 F3 Jupiter[®], which operates according to the basic principle described in Chapter 2.1.1. The platinum furnace of this instrument was used to heat the sample crucible and reference crucible alike from room temperature (approx. 25 °C) to 250 °C. This was done at five different heating rates ($\beta = 2, 4, 6, 8, 10 °C/min$), at each of which five measurements were performed. A sample carrier of type *HTP4000A72.010* was used, in which type S thermocouples were installed to measure the temperature of the crucibles. Nitrogen 5.0 was applied as purge gas with a flow rate of 50 ml/min.





Figure 2: Differential scanning calorimeter NETZSCH STA 449 F3 Jupiter[®] (left) and its sample carrier with two crucibles placed on it (right).

2.1.3 Crucible

In order to avoid evaporation of the HTP sample due to the heating process, so-called *high-pressure crucibles* were used during the conducted DSC measurements. These are pressure-tight sealable containers and consist of a crucible, lid and sealing disc. They have a volumetric capacity of $100 \ \mu$ l and are reusable. To screw down the high-pressure crucibles, a special sealing tool is required. Commercially available high-pressure crucibles are typically made of

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stainless steel (with or without a gold-plated surface) and have gold sealing discs. However, for the activation energy determination of the decomposition reaction of HTP which is in contact with surfaces of Inconel 718, it is necessary to use a high-pressure crucible made of this material. Therefore, high-pressure crucibles made of *Inconel* 718 were conventionally manufactured especially for the investigations in this work (see Figure 3). Their design is based on that of commercially available high-pressure crucibles. Since HTP has a poor compatibility with gold (compatibility class: 4 [18]), it was assumed that sealing discs made of this material would affect the decomposition reaction of HTP. For this reason, the sealing discs were also newly manufactured. The material used was Aluminium 1060, which has an excellent compatibility with HTP (compatibility class: 1 [18]). The influence of this material on the decomposition reaction of HTP can therefore be considered negligible. Before the DSC measurement campaign, the crucibles, lids and sealing discs were subjected to a passivation procedure to further increase their compatibility with HTP. In this passivation procedure, the parts were first placed in a trisodium phosphate sodium metasilicate solution for two hours, then in a 5 wt% sodium hydroxide solution for one hour and afterwards for one hour at a temperature of 60 °C in a CitriSurf[®] 3050 dilution (one part CitriSurf[®] 3050, three parts water). After a drying period of 45 minutes, the parts were finally placed in 30 wt% hydrogen peroxide for eight hours. The parts were rinsed with water after each passivation step. Such a passivation procedure is a common approach for materials that will have contact with HTP and can in principle also be applied in cooling passages of regeneratively cooled space propulsion systems. The five measurements of one heating rate were carried out with a high-pressure crucible which was used exclusively for the corresponding heating rate. These high pressure crucibles were unused except for the passivation procedure conducted on them, so the same surface conditions existed for all high pressure crucibles prior to the DSC measurements.



Figure 3: High-pressure crucible made of Inconel 718 in the open (left) and closed (right) state.

2.1.4 Sample

For the conducted investigations HTP from *Jakusz SpaceTech* was used. Its concentration was determined before and after the DSC measurement campaign. For this purpose, three density measurements were carried out in each case at a temperature of 25 °C using the *Easy D40* density meter from *Mettler-Toledo*. With the two resulting mean values of these measurements and using reference [19], the concentration of the HTP before and after the measurement campaign was then determined to be 97.2 wt%. During the measurement campaign, the HTP was stored in a refrigerator and was only removed from it briefly for filling the high-pressure crucibles. Before each measurement, 7.0 μ l of HTP was measured with a pipette and filled into a high-pressure crucible. The sample quantity was based on a recommendation of the *International Confederation for Thermal Analysis and Calorimetry (ICTAC)* [20]. Accordingly, the product of sample mass and maximum heating rate should not exceed the value of 100 mg °C/min. In order to obtain the strongest possible DSC signal in the measurements, the sample quantity of 7.0 μ l was chosen so that the value of the resulting product (99.8 mg °C/min) was just below this limit. To exclude a possible influence of the atmospheric oxygen on the decomposition reaction of HTP, the filling and sealing of the high-pressure crucibles took place in a glovebox filled with the inert gas argon. Also the reference crucible was sealed in this glovebox. The tightness of the high-pressure crucibles was controlled during the measurements by a high-precision balance installed in the differential scanning calorimeter. No leaks were detected by any of the conducted DSC measurements.

2.2 Kissinger Method

The *Kissinger method* was used in this work to determine the activation energy of the decomposition reaction of HTP from the DSC measurement data. This method was published in the 1950s by *H. E. Kissinger* [21, 22] and is one of the most popular approaches for determining activation energies by DSC due to its simple and rapid applicability. In order to apply the Kissinger method, four conditions must be fulfilled, which relate to the measurement data and the reaction to be investigated:

• DSC measurements with different heating rates

The data must be from DSC measurements, which were carried out typically at five or six different heating rates, each held constant. The DSC measurements in this work were performed under the mentioned conditions with five different heating rates.

• Relative (local) minimum or maximum

The data must have a relative (local) minimum or maximum in each measurement. Such a peak typically results from an endothermic or exothermic reaction that occurs in the sample during the heating process of a DSC measurement. In this work, HTP was used as sample whose decomposition is exothermic, thus generating a significant exothermic peak in the DSC signal.

• First-order reaction

The reaction under investigation must be a first-order reaction. First-order reactions are characterized by a linear correlation between the reaction rate d[A]/dt and the concentration of the reacting substance [A] according to the following equation:

$$\frac{\mathrm{d}[A]}{\mathrm{d}t} = -k \cdot [A]. \tag{3}$$

Here, k is called the reaction constant. The decomposition reaction of HTP studied here is such a first-order reaction [23].

• Single-step reaction

If the Kissinger method is to be used for an exact determination of the activation energy, the reaction to be investigated must be a single-step reaction. This is because the Kissinger method can be used to determine only a single activation energy for a reaction. Such a single activation energy is only available for single-step reactions, which take place in one step without the formation of intermediates. However, often the reaction under investigation is a multi-step reaction that has a complex reaction mechanism which consists of a sequence of elementary reactions. Here, each of these elementary reactions has its own activation energy. In principle, the Kissinger method can also be used for a multi-step reaction. But the activation energy resulting from such an application is then equal to that of their simplified global reaction [24]. Also the decomposition reaction of HTP which is studied in this work is a multi-step reaction [25]. Therefore, the Kissinger method can only be used to determine the activation energy of the global reaction of HTP given in Equation (1).

The Kissinger method is based on the fact that the endothermic or exothermic peak observed in the DSC signal shift to higher temperatures when the heating rate is increased. The extent of this peak shift depends, among other things, on the activation energy of the investigated reaction and can be described by the following *Kissinger equation*:

$$\ln\left(\frac{\beta}{T_p^2}\right) = \ln\left(\frac{A \cdot R}{E_a}\right) - \frac{E_a}{R \cdot T_p}.$$
(4)

Where β is the heating rate, T_p is the temperature of the peak maximum (named peak temperature), A is the preexponential factor, R is the gas constant (R = 8.314 J/mol K) und E_a is the activation energy.

For the activation energy determination by the Kissinger method, the conducted DSC measurements must first be analyzed with respect to their peak temperature. These peak temperatures are then plotted as data points on the *Kissinger plot*, which shows $1/T_p$ on the x-axis and $\ln(\beta/T_p^2)$ on the y-axis. This is followed by a linear regression analysis to determine a straight line that best describes the linear correlation between the data points. The equation of this resulting regression line corresponds to the presented Kissinger equation (Eq. (4)), so that the activation energy can be finally determined from the slope of this line according to the following equation:

$$E_a = -R \cdot \frac{\mathrm{d}\left(\ln\left(\frac{\beta}{T_p^2}\right)\right)}{\mathrm{d}\left(\frac{1}{T_p}\right)}.$$
(5)

2.3 Temperature Calibrations

For a higher accuracy in the determination of the peak temperatures, temperature calibrations were conducted with the differential scanning calorimeter prior to the DSC measurement campaign. For this purpose, calibration measurements with four pure calibration substances (see Table 1) whose melting temperatures (T_m) are well known were carried out under the same measuring conditions as in the later DSC measurement campaign (same high-pressure crucibles, heating rates, atmosphere, etc.). The selection of these calibration substances was made to ensure that they are well compatible with the *Inconel 718* material of the high-pressure crucibles. An influence of this material on the melting process and therefore a falsification of the temperature calibration could thus be excluded.

Table 1: Calibration substances used in the temperature calibrations and their accurate melting temperatures [26].

Calibration substance	T_m
Indium	156.6 °C
Tin	231.9 °C
Bismuth	271.4 °C
Lead	327.5 °C

For performing the calibration measurements, a sample quantity of about 10 mg was first measured from each of the four calibration substances. These four samples were then each placed in a separate high-pressure crucible. Analogous to the procedure for the later DSC measurement campaign, this filling and the subsequent sealing of the high-pressure crucibles took place in a glovebox filled with the inert gas argon. Because a separate temperature calibration was necessary for each of the five heating rates used ($\beta = 2, 4, 6, 8, 10 \,^{\circ}C/min$), five calibration measurements were made with each of the four calibration substances, in which the heating rate was varied accordingly. During a calibration measurement, the respective calibration substance was heated in the differential scanning calorimeter from room temperature (approx. 25 $\,^{\circ}C$) to a temperature of about 100 $\,^{\circ}C$ above its melting temperature. This temperature range ensured that the entire melting process of the calibration substance was captured in the calibration measurement. After completion of a calibration measurement, a cooling phase took place in which the temperature of the calibration substance returned to room temperature. Then the next calibration measurement could be started.

After all calibration measurements had been carried out, these had to be analyzed with regard to the measured melting temperatures. In principle, a melting process is represented in the DSC signal by an endothermic peak. The melting temperature is then defined as the temperature at which this endothermic peak begins. A good estimate of this melting temperature can be made by determining the so-called *onset temperature*. This onset temperature corresponds to the temperature of the intersection of two tangents applied to the baseline and the descending slope of the endothermic peak, respectively. For the determination of these onset temperatures from the conducted calibration measurements, the analysis software *NETZSCH Proteus*[®] (version 6.1.0) was used. This analysis software was then also used to generate the calibration curves from the determined onset temperatures, which were used in the later DSC measurement campaign. Therefore, the software calculated the deviations between the accurate melting temperature of the calibration substance (see Table 1) and the melting temperatures which were determined for this calibration substance in the calibration measurements. Based on these deviations, the software generated a separate calibration curve for each of the heating rates used, which corrected the measured peak temperatures during the later DSC measurements.

Finally, in order to determine the quality of the temperature calibrations, the calibration measurements for indium were repeated using the generated calibration curves. As shown in Table 2, the application of the calibration curves further increased the already quite high accuracy in the determination of the melting temperature. In particular, a

notable improvement was achieved in the minimum and maximum heating rate. The temperature difference ΔT given in Table 2 in each case represents the deviation of the determined melting temperature from the accurate melting temperature of indium (156.6 °C).

β	$T_{m, cc off}$	ΔT	T _{m,ccon}	ΔT
2 °C/min	155.8 °C	-0.8 °C	156.4 °C	-0.2 °C
4 °C/min	156.3 °C	-0,3 °C	156.4 °C	-0.2 °C
6 °C/min	156.8 °C	+0.2 °C	156.2 °C	-0.4 °C
8 °C/min	157.0 °C	+0.4 °C	156.3 °C	-0.3 °C
10 °C/min	157.4 °C	+0.8 °C	156.4 °C	-0.2 °C

Table 2: Measured melting temperatures of indium without $(T_{m, cc off})$ and with $(T_{m, cc on})$ the calibration curves.

3. Results and Discussion

3.1 Peak Temperatures

The conducted measurement campaign consisted of a total of 25 DSC measurements. In all of these measurements, a significant exothermic peak was observed in the DSC signal which results from a decomposition of the HTP heated in the crucible. Figure 4 shows exemplary the exothermic peaks of the first measurement, which was carried out for each heating rate. Here, the peak shift to higher temperatures when the heating rate is increased, as described in Chapter 2.2, can be clearly seen. Such a shift could also be observed in all other measurements performed.



Figure 4: Exothermic peaks of the first measurement of each heating rate.

In order to be able to apply the Kissinger method for the activation energy determination, the recorded exothermic peaks were first analyzed with respect to their peak temperatures. Therefore, the analysis software *NETZSCH Proteus*[®] (version 6.1.0), which was already used for the temperature calibrations, was applied again. The resulting peak temperatures of all performed DSC measurements and the average peak temperature calculated for each heating rate are listed in Table 3.

β	$T_{p,1}$	$T_{p,2}$	$T_{p,3}$	$T_{p,4}$	$T_{p,5}$	\bar{T}_p
2 °C/min	118.5 °C	116.3 °C	116.8 °C	118.6 °C	119.5 °C	117.9 °C
4 °C/min	128.1 °C	130.7 °C	127.9 °C	125.9 °C	126.1 °C	127.7 °C
6 °C/min	134.2 °C	133.3 °C	133.5 °C	134.3 °C	135.4 °C	134.1 °C
8 °C/min	138.3 °C	138.6 °C	140.4 °C	141.0 °C	140.3 °C	139.7 °C
10 °C/min	140.7 °C	143.1 °C	142.9 °C	143.7 °C	143.8 °C	142.8 °C

Table 3: Peak temperatures determined in the DSC measurement campaign.

3.2 Activation Energy

According to the Kissinger method introduced in Chapter 2.2, the peak temperatures averaged for each heating rate were first plotted as data points on the Kissinger plot (see Figure 5). A linear regression analysis was then conducted to determine by the *method of least squares* a straight line that best describes the linear correlation between the plotted data points. The regression line resulting from this analysis has a coefficient of determination of $R^2 = 0.998$, which indicates that the fit of the line is obtained very well. It has the following equation:

$$y = -9567.9 \cdot x + 13.24. \tag{6}$$

In accordance with Equation (5), the activation energy can be determined from the gas constant and the slope of this regression line $(d(\ln(\beta/T_p^2))/d(1/T_p) = -9567.9 \text{ K})$. The resulting activation energy is $E_a = 79.5 \text{ kJ/mol}$. To determine the error range of this activation energy, the sample standard deviation was first calculated for each of the averaged peak temperatures and then plotted as error bars on the Kissinger plot. After that, the straight lines were identified that have the minimum and maximum possible slope to be just within all error bars. From the slopes of these two straight lines, the minimum and maximum possible value for the activation energy was then calculated ($E_{a,min} = 73.3 \text{ kJ/mol}$, $E_{a,max} = 89.2 \text{ kJ/mol}$). These values limit the error range of the determined activation energy. The activation energy of the decomposition reaction of HTP which is in contact with passivated *Inconel 718* can therefore be given as follows:

$$E_a = (79.5 ^{+9.6}_{-6.3}) \,\text{kJ/mol.} \tag{7}$$



Figure 5: Kissinger plot for activation energy determination.

In order to assess whether and, if so, to what extent the decomposition reaction of HTP was catalytically affected by the material *Inconel 718*, the activation energy determined in this work was compared with literature values known for the decomposition reaction of liquid HTP without a catalytic effect (see Table 4). It was found that these literature values are very similar to the value obtained in this work and that they are even all within its error range. Since materials with a high catalytic activity toward HTP typically result in a substantial reduction of the activation energy by several 10 kJ/mol [27], it can be assumed that passivated *Inconel 718* has no significant catalytic impact on the decomposition reaction of HTP.

Table 4: Selection of activation energies known for the decomposition reaction of HTP without a catalytic effect.

E_a	Reference
79.3 kJ/mol	[23]
76.0 kJ/mol	[28]
75.3 kJ/mol	[29]

3.3 Scanning Electron Microscope Images

Before and after the DSC measurement campaign, the scanning electron microscope (SEM) *JEOL JSM-IT200 InTouch-Scope*[®] was applied to take images of the bottom from one of the high-pressure crucibles. The crucible which was used to perform the DSC measurements at a heating rate of 2 °C/min was selected for this purpose because it had the longest exposure time to the HTP compared to the other crucibles (more than 9 hours in total). It was assumed that if any changes in the surface topography had occurred due to the influence of the HTP, the changes had to be most pronounced at this crucible.

Figure 6 shows two SEM images from the same location of the bottom taken before and after the DSC measurement campaign. Both images show small dark spots, which are assumed to be a type of pitting corrosion that may be caused by HTP [30]. However, a comparison of the two images shows that the number and diameter of these dark spots did not change during the DSC measurement campaign. Since no changes in the surface topography were visible in any of the other SEM images taken of the bottom, this suggests that no extensive interactions between the HTP and the passivated *Inconel 718* occurred during the DSC measurement campaign. Thus, the SEM images confirm the result of the conducted activation energy determination. It was not possible to clarify if the dark spots were possibly formed during the passivation procedure or if they were already on the surface before, because no SEM images of the bottom of the spots that appear differently bright on the SEM images are only the result of different lighting conditions when the images were taken and are not due to an influence of the HTP.



Figure 6: SEM images of the bottom from one of the high-pressure crucibles before (left) and after (right) the DSC measurement campaign.

4. Conclusion

In this work, differential scanning calorimetry (DSC) was used to determine the activation energy of the exothermic decomposition reaction of high-test peroxide (HTP) which is in contact with passivated *Inconel 718*. For the DSC measurements, samples of 97.2 wt% HTP were placed under an inert atmosphere in high-pressure crucibles made of passivated *Inconel 718*, which were then heated at a constant heating rate during the measurements. Five different heating rates were used in the DSC measurements ($\beta = 2$, 4, 6, 8, 10 °C/min), with five measurements taken at each rate. In all conducted measurements, a significant exothermic peak was observed in the DSC signal resulting from the decomposition of the HTP. After analyzing these exothermic peaks with respect to their peak temperatures, an activation energy of $E_a = (79.5 + 9.6) \text{ kJ/mol}$ was determined by the Kissinger method. A comparison of this determined activation energy with literature values known for the decomposition reaction of HTP without a catalytic effect suggests that passivated *Inconel 718* has no significant catalytic impact on the decomposition reaction of HTP. Investigations of the surface topography of the bottom from one of the high-pressure crucibles by a scanning electron microscope showed no changes that would indicate extensive interactions between the HTP and the passivated *Inconel 718*, so that these investigations confirm the result of the conducted activation energy determination.

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