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<td>Abstract</td>
<td>This deliverable presents the small scale validation of high temperature protection solutions, radio performance in hot pellets environments, and positioning estimation performance. All validations have been performed in industrial environments. The high temperature protection was evaluated in a 1200°C environment. The performance agree very well with previous simulations, and show that the developed concept can be used for future DISIRE sensors in hot environment. Using the developed protection, a radio test was performed with the system embedded in 900°C hot pellets. The test showed that the usage of a low radio frequency (433 MHz or below) is to be preferred to reduce attenuation. The position measurement validation verified the findings in the radio test on the need for a low frequency for data transmission. Magnetic field beacon signals were however clearly readable, and position estimates of 1 m was achieved in free air.</td>
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1 Introduction

This report presents the small scale validation that has been performed as part of the ongoing wireless sensor development for continuous processes within WP3. The status for the work with batch sensor design is separately reported in D3.3. The validation for wireless sensors has been performed in order to investigate the functionality in the following areas:

- **High temperature protection.** A sensor protection principle has been developed. Its behaviour in a temperature of 1200°C has experimentally been investigated.
- **Radio performance in high temperature pellets environment.** Using a prototype temperature sensor enclosed in the protection above, a range of available radio frequencies have been tested to investigate performance limitations.
- **Positioning performance.** Range and precision of a positioning solution has been evaluated in air and enclosed in iron ore pellets.

Respective area is presented in its own section below, followed by a brief conclusion.

2 High temperature protection

2.1 Introduction

The wireless sensor devices of DISIRE will need to be deployed in processes where temperatures exceed 1200°C for measurement of e.g. temperature or gas properties. To reduce development time and cost, it is desired to be able to use standard electronics where the upper temperature limit is 125°C. This upper limit will also, for short operating times, allow batteries normally specified for lower operating temperatures (e.g. silver oxide batteries) to be used.

Even though the device is not, in most cases, expected to be recovered from the process, achieving as long operating time as possible is important. Furthermore, some processes set stringent size requirements on the devices. Thus, high demands are put on the construction of the heat insulation. One approach to achieve size effective heat protection is to combine an outer shell of thermal insulation with an inner volume consisting mainly of material or fluid that will consume large amounts of energy when heated through the insulation [1], [2], [3].

This section presents the design and experimental validation of an encapsulation concept for operation in temperatures up to 1200°C. Simulations show achievable performance for a number of insulation material choices when water is used as the encapsulated heat absorbent. Experiments performed in industrial environment verify the simulations and the feasibility of the concept. Subsection 2.2 introduces the overall concept as well as a discussion on insulation materials and heat absorbents. Simulations of achievable performance are presented in subsection 2.3. The final device is verified by measurements presented in subsection 2.4. Parts of the design, material choices, and simulated results were presented in the DISIRE deliverable D3.1. They are herein included for completeness, and also extended. The results in this section has also been presented at [4].

2.2 Design

2.2.1 Principle

To keep the internal temperature of the device with constrained outer dimensions low for as long as possible, it's desirable to minimize the thermal conductivity from the outside and into the device, while at the same time maximizing the amount of energy that is required to heat the device to it's maximum operating temperature. One effective way to achieve this is to combine a
layer of thermal insulation with the encapsulation of a material or fluid that will consume energy when heated inside the insulation [1].

The design of the device presented in this paper is shown in Fig. 1. A round (ball shaped) design is chosen as this maximizes the volume for a given surface area. The outer insulation shell is cast in two halves with a matching finger and groove that keeps the halves aligned while they are being cemented together before use. As further elaborated below, one of the best materials in terms of required energy in the temperature span normal electronics can manage (−40..125°C) appears to be water (specific heat per volume and heat of evaporation, possibly combined also with heat of fusion), but it has the disadvantage of being liquid at room temperature. To benefit from the large energy required for evaporation the water must be evaporated within the thermal insulation. To this end, the inner part of the device is composed of wet floral foam (Oasis®), whose purpose is to hold the fluid to be used for energy absorption. To minimize fluid loss to the porous high temperature insulation, the floral foam is wrapped in aluminum foil. Shown in the figure is also the possible inclusion of an electronics printed circuit board (PCB), which holds sensors and battery. The PCB will be cast in epoxy to protect it from the fluid in the floral foam and may be sandwiched between sheets of aluminium to avoid local hot-spots.

As the internal temperature of the device rises when subjected to high outside temperatures, the specific heat of the fluid absorbs the bulk of the thermal energy until the boiling point is reached. Furthermore, as the temperature reaches the boiling point of the fluid, energy is absorbed as the fluid changes phase from fluid to gaseous. During this phase change, the internal temperature of the device is held constant at the boiling temperature of the fluid, allowing electronics and batteries to function. When the fluid changes phase to gaseous, the internal pressure in the device will increase. Thus, to avoid cracking, it is important to ascertain that escape paths are available from the inside through the insulation material. For the experiment described in this paper, the escape path was provided adjacent to the temperature probe that was inserted into the device.

When used in a high temperature environment, e.g. 1200°C, the floral foam and the electronics will burn or turn to char shortly after all the fluid has evaporated. The outer insulation will survive, but will be hard to reuse as the two halves are cemented together before usage.
2.2.2 Insulation materials

To find an upper bound on the feasible life-time of enclosed units a survey of commercially available insulating materials was performed. Investigated materials include:

- **Carbon black** [5]: survives very high temperatures (3000°C in reducing atmospheres), thermal conductivity comparable to or lower than the best solid materials found that support temperatures of 1200°C or more. However, it is a loose granular material that would need to be enclosed in some form of ceramic shell (typically alumina).

- **Alumina fiber boards** [6]: useable up to about 1700°C, slightly higher thermal conductivity than carbon black. The material is delivered in the form of rigid boards rather than a loose granular material.

- **High-temperature moldable materials** [7] exist, but do not insulate as efficiently as the best solid products, and in the authors experience also exhibited significant shrinkage during drying, which made the manufacturing of shapes with high precision difficult.

- **Moldable materials** [8]: This material supports up to 1260°C with a moderate increase in thermal conductivity. The material is delivered as a paste that can be cast to desired shape.

- **Microporous materials** [9] exhibit far superior insulating properties, but withstand only up to about 950°C. This very fragile material is delivered as boards.

There is a clear trend that insulating materials that withstand higher temperature exhibit higher thermal conductivity at all temperatures. Some improvement could be achieved by combining materials, but since the thermal conductivity is higher for materials that support higher temperatures
2.2.3 Heat absorbent

The following assumes that temperatures much higher than 100°C are not practical, especially as commercially available batteries appear to be limited to 125°C. Lower operating temperatures would extend the range of available batteries significantly.

Any insulating material will transmit some energy. This energy is absorbed as temperature increase and/or phase change of the materials enclosed within the material. The most promising method of absorbing heat known to us at this point is the evaporation of water. This requires 2260 J/g. If, on the other hand, one starts from ice (heat of fusion: 334 J/g) and the energy required for heating water 100°C, about 420 J/g, a total of about 3 kJ/g (or close to 3 KJ/cm³) can be absorbed. Using the evaporation of water is however not without problems: filling the core with ice/water could interfere significantly with antennas, and it could be challenging to ensure that the water only exits the core once it has evaporated, not in liquid form.

For lower operating temperatures alcohols would be one alternative at the cost of significantly lower performance. For example, methanol boils at 65°C and absorbs about 1 KJ/cm³ from 0°C to vapor at 1 Atm. Solid-liquid phase changes are easier to use because a hermetically sealed container can be used but to the authors knowledge they perform worse still: fatty acids such as Lauric acid has a heat of fusion of about 200 J/cm³, and hydrated salts such as NaSO₄·10H₂O of about 350 J/cm³. Further investigations into available salts may yield some improvements, but a relatively small compartment of water that is allowed to evaporate would nevertheless serve the same function as a significantly larger volume of any other known practically useable material.

Figure 3: Simulated operating time before core exceeds 100°C at an ambient temperature of 1200°C.

(especially at high temperatures), the fraction of the thickness where the lower-temperature insulating material can be used is expected to be small, with a corresponding modest improvement in performance. This would also increase the mechanical complexity of the device significantly.
2.3 Simulations

For a given outer geometry, a trade-off exists between the thickness of the insulating material and the volume left for the material that absorbs the conducted heat. Whereas increasing the thickness of the insulation will reduce the thermal conductivity (approximately, depending on shape) linearly, the volume inside the insulation (the core) changes with the cube of its linear dimension. For a ball-shaped device, FEM simulations were performed using piecewise linear models of the thermal conductivity as a function of temperature for the various materials. The thermal conductivity was interpolated at a constant level below the lowest temperature specified by the manufacturer and linearly above the highest temperature for “AX Moldable” where data was only available up to 800°C. Simulations over a range of core and outer diameters show that for all materials relevant here, the optimum diameter for a core completely filling an insulating shell would be about 67% of the outer diameter, independent of the actual outer diameter.

From this data the time required for an ice filled core at 0°C to be completely converted to steam at 100°C was calculated for various values of outer radius. The temperature gradient across the insulation was assumed to be static with an inner surface temperature of 100°C, thus underestimating the conducted energy somewhat when the core is at 0°C. The resulting maximum operating time for devices insulated with four different materials when the ambient temperature is 950°C is shown in Fig 2, with a corresponding plot for three materials at an ambient temperature of 1200°C shown in Fig 3.

As expected, the simulations show that the achievable operating time is highly dependent on outer diameter as well as on ambient temperature. As an example for a sphere built by moldable material [8], the resulting simulated maximum operating time before the core temperature exceeds 100°C at an ambient temperature of 1200°C is 21 minutes for an outer radius of 4 cm. If the outer radius is increased to 10 cm the time increases to 125 minutes. At the lower temperature of 900°C the 4 cm radius gives an operating time of 32 minutes while a 10 cm radius achieves 208 minutes.
2.4 Measurements

Measurements were performed to verify the simulations. The measurements were performed using a Nabertherm N87/H oven at Mefos premises in Luleå, Sweden. Spheres with an outer radius of 4 cm were fabricated using the moldable insulation material [8] with a water filled core. Two devices were fabricated and measured; one was mounted using a pre-freezed core where the majority of the water was frozen, while the second core was saturated with water at room temperature. The internal temperature of the devices was measured using a Type K temperature sensor inserted through the insulation into the core as shown in Fig. 13.

The devices were placed in the oven as shown in Fig. 5, and the internal temperature of the devices was recorded. Figure 6 shows the internal temperature for both devices when subjected to an oven temperature of 1200°C. The device filled with fluid water held the core temperature at or below 101°C for a total of 25 minutes. The time to reach the boiling point of the water was 9 minutes. Thereafter, the temperature was held constant at 100 +/- 1°C for an additional 16 minutes whereafter a rapid rise in temperature took place once all water had evaporated. The device with initially frozen water performs less well. The operational time is shorter than for the device with fluid water, and the temperature fluctuates both approaching the boiling point and at the boiling point. We speculate that the instability is due to an uneven temperature distribution in the semi-frozen core. As parts of the core close to the outer insulating shell melts this section absorbs heat at a faster rate than the parts that are still frozen. Local boiling and evaporation occurs, causing temperature fluctuations when this happens close to the inserted temperature probe.

The measurements were aborted when the internal temperature exceeded 300°C, and the device was recovered from the oven. Figure 7 shows the device cooling off after a completed measurement. At this time the floral foam has been partially pyrolyzed.
Figure 6: Data from temperature test of two devices; one with initially frozen water core and one with initially fluid water core at room temperature. The oven temperature was 1200°C.

Figure 7: Device cooling after withdrawal from oven. Note the glowing support ring whereas the outer layer of the insulation cooled down much more rapidly and stopped glowing visibly almost immediately after the device was taken out of the oven.
3 Radio performance in high temperature pellets environment

3.1 Introduction

One unknown parameter in the design of the high temperature sensors was the behavior of radio transmissions in the presence of hot iron ore pellets. Previous measurements as reported in the DISIRE deliverable D3.1 investigated mainly magnetic coupling in cold materials up to 100 MHz. Although these measurements showed good performance in cold sintered pellets, the size limitations in the DISIRE project makes it highly interesting to investigate the behavior of higher frequencies giving propagating waves, as this allows for smaller antennas both at receive and transmit position. Also, at higher temperatures the conductivity of the material may change.

The remainder of this section describes the design and experimental validation of a wireless measurement system with temperature measurement (internal/external) and transmitter that utilizes free RF bands from 433 MHz up to 5775 MHz. The measurement system was enclosed in the high temperature protection presented in the previous section, and experimentally validated covered in 900 °C hot iron ore pellets.

3.2 Measurement system design

3.2.1 Electronics

The system was built to fit into the enclosure presented in section 2 above. With this size limitation the electronics was designed using a two sided four layer PCB measuring 18x38 mm. The core of the system is a MAX2871PLL chip that is used to generate the selected radio frequencies. The PLL is controlled from an Atmel Xmega 32E5 microcontroller and the output is amplified by MGA-81563 monolithic amplifiers. The PLL has two outputs that are driving each two amplifiers used to drive two monopole antennas in anti-phase. The two sets of two monopoles are thereafter combined into two dipoles mounted on the PCB. The double set of dipoles is used to achieve the possibility to transmit on two polarizations to reduce rotational sensitivity for the transmitter. Internal temperature information is sampled from an ADT7410 digital temperature sensor chip. External temperature data are collected using the microcontroller internal ADC to sample a type K thermocouple temperature probe that is designed to protrude out of the insulation shell. Also coarse battery voltage level measurements was performed using 8 levels (3 bits) during the test.

The transmission requires high current, over 200 mA. To be able to supply this from the used silver oxide batteries, they are buffered by a large capacitor and the transmission is made in short bursts of up to 6 ms each to conserve battery energy. Transmission is initiated by a short period with carrier only on the selected frequency. Thereafter 127 bits of FSK modulated (+/- 50 kHz deviation) data are sent at 32768 bit/s. The initial 64 bits are a synchronization word, thereafter follows data from internal temperature sensor, ADC data from external temperature sensor, power supply indication and polarization id information. In total the data uses 31 bits, which are expanded to 63 bits using BCH code for error correction. The sequence is repeated for both polarizations and for the frequencies 433, 868, 1458, 2450, 3753, and 5775 MHz. The software on the system is to the largest extent run on the 32768 Hz interrupt handler. The frequency of messages as well as the selected radio frequencies are all software adjustable.

The PCB is mounted with components on both sides, as shown in figures 8 and 9. Thereafter, external components such as dipole antennas, storage capacitor, and temperature probe are mounted, and the system is covered with protective lacquer as shown in figure 10. The final step of the assembly of the electronics for the measurement system is to mount 1 mm thick aluminum heat distribution plates on both sides using thermally conductive silicon glue. The plates are used to avoid local thermal heating of the electronics. The assembled electronics unit is shown in figure 11.
3.2.2 Integration in heat protection

The electronics unit of the measurement system was integrated into the heat protection described in section 2. A hollow area was prepared in the floral foam holding the water, large enough for the electronics to be fitted. The floral foam was also removed adjacent to the dipole antennas to get better radio environment, as seen in figure 12. As shown in figure 13, the batteries are now mounted on the lower side of the electronics unit. The figure also shows the protrusion of the external temperature sensor through the temperature protection. As a preparation for the final step of integration the floral foam is wrapped in aluminum foil. It is thereafter soaked with water and mounted into the heat protection, which has its halves glued together with non-hardened insulation material as shown in figure 14. The completed measurement device, ready to use, is shown in figure 15.

3.3 Measurements

Measurements were performed at Mefos premises in Luleå, Sweden. A steel container containing an approximately 30 cm thick layer of iron ore pellets was pre-heated to approximately 900°C, whereafter it was removed from the oven as shown in figure 16. Before heating of the pellets, a cylindrical steel tobe was placed in the pellets. The steel tube was used to be able to place
Figure 10: PCB with components and mounted dipole antennas, capacitor, and external temperature probe. The PCB has been coated with protective lacquer.

Figure 11: Completed electronics unit of the measurement system. Red and black cables are for connection to batteries which will be mounted on the underside as the electronics is integrated in the heat protection and activated for operation.

As described in section 3.2.1 above, the measurement system collected data on internal temperature, external temperature, and battery status during the test. All the data was transmitted using all investigated frequencies. As seen in figures 22, 23, and 24, the measurement system was transmitting data during approximately 30 minutes before operation ceased. Again, this time is well in accordance with estimates simulated and presented in section 2.3 above, where an estimate of 32 minutes is done at 950°C. The slightly lower time achieved at a slightly lower temperature is likely attributed to the fact that some of the internal space for water was occupied.
by the electronics, thus giving a lower energy consumed for the phase change of the water.

The external temperature sensor was calibrated using the knowledge of the initial temperature in the pre-heated pellets container. The resulting measured external temperature data is shown in figure 22. Seen is the exponential decay in temperature that takes place once the container with pellets has been removed from the oven. The internal temperature measurement in figure 23 shows, as expected, a temperature plateau at, and slightly above, 100°C. Once all water has changed phase at around 1700 s into the test, the temperature rises sharply. At 145°C transmission of data stops. Battery data presented in figure 24 shows a stable battery voltage level up to the approximate point where the rapid temperature rise takes place. Thereafter the voltage level drops, and the last indicated level is half of the initial value. A likely explanation is a failure of one of the series connected battery cells, with subsequent system operation failure.

The overlying goal of the measurement was the evaluation of attenuation of radio signals on different frequencies. For this purpose, the signals transmitted from the measurement system were received and recorded using an Agilent E4440A spectrum analyzer. The resulting data is shown in figure 25 for all used frequencies. Here, 0 dB indicates the received signal level for each frequency before the measurement system is inserted into the hot pellets. Insertion starts at about -50 s, and the time is set to 0 s where the insertion tube is removed from the pellets and the measurement system is covered in the pellets. The data clearly shows that the
higher frequencies attenuate more than the lower. The frequencies of 3753 MHz and 5775 MHz completely disappear, and the 2450 MHz is noticeably weakened. Also at lower frequencies a time dependence can be seen. The reason for this is unclear, it may relate to internal temperature changes or movement of the antennas inside the measurement system.
A clear conclusion from the measurement is that a frequency as low as possible should be used for radio transmissions in the environment. One factor that may need to be taken into account is that the attenuation most likely will increase with an increasing thickness of the layer of pellets. This may be a limiting factor for deployment into large silos, as also discussed in section 4.2.1 below.
Figure 17: Placement of measurement system into the heated pellets using the pre-placed hollow tube.
Figure 18: Removal of the hollow tube to let the measurement system be completely encapsulated by the heated pellets.
Figure 19: Measurement system dug out of the heated pellets after completed measurement.

Figure 20: Measurement system cooling off.
Figure 21: System opened after retrieval from the hot pellets.

Figure 22: Data from external temperature measurement during testing in hot pellets.
Figure 23: Data from internal temperature measurement during testing in hot pellets.

Figure 24: Data from battery monitor during testing in hot pellets.
Figure 25: Relative received signal strength at respective frequency during testing in hot pellets. The level of 0 dB indicates the signal level received before the measurement system was inserted into the hot pellets.
4 Position determination

4.1 Position measurement principle

As also briefly discussed in the DISIRE deliverable D3.1, one viable technology for the determination of position is the use of low frequency magnetic field "beacons". The technique is based on measurement of the strength of the received magnetic field(s) in a transponder. The further the distance from a magnetic field transmitter, the lower the signal received in the transponder. For DISIRE, the system is designed based on three or more beacons that transmit ranging pulses at 125 kHz. The beacons are placed as orthogonally as possible in relation to the intended (foreseen) position of the transponder. The transponder is equipped with a commercially available three dimensional receiver antenna, as shown in figure 26. The beacons will send at discrete time slots, with identification. The used beacon is the Electrotech Craycom Najad, sized 190x140x30 mm, shown in figure 27. At reception of the beacon signal(s), the transponder measures the signal strength received in each axis by each beacon, and transmits the data back to overlying control system using traditional radio at 868 MHz. The control system, in turn, calculates the position of the transponder based on received signal strengths from each transmitter.

4.2 Measurements

Two sets of measurements have been performed; one at Mefos in Luleå where the transponder was embedded in pellets, and the other in free air at Electrotech premises in Kalix.

4.2.1 Positioning measurement in pellets

A measurement was made where the transponder was embedded into approximately 2 m of iron ore pellets as shown in figure 28 (VILKEN STORLEK UNGEFÅR). The primary purpose of this measurement was to verify system functionality in this type of environment, with not much regard to actual position performance. The main challenge in this measurement was the return signal attenuation for the frequency of 868 MHz. As also discussed above in section 3.3, the attenuation increases with increasing frequency and also with increasing thickness of the layer of surrounding pellets. The measurements in section 3.3 showed 10-20 dB of attenuation for the 868 MHz signal, which would be acceptable. However, the positioning measurements had a much thicker layer of pellets (2 m vs. 30 cm), and showed attenuation values of 20-40 dB, making it hard to receive the return signal. Thus, we foresee a need to shift the return signal to 433 MHz at the highest. This band showed very low attenuation in section 3.3, which is promising for the positioning application. Also, the possible use of even lower frequencies down to 169 MHz will be investigated. Here, the challenge lies in the protection of frequency bands; the intended 169 MHz band is for now open for remote reading of appliances such as electrical and water meters. It’s use in the positioning application will need to be verified.

The beacon signal at 125 kHz, on the other hand, shows good penetration, which is also consistent with measurements previously presented in the DISIRE deliverable D3.1. Thus, although the foreseen application is in silos with sizes of 10’s of meters, we do not foresee that the beacon signal will pose a problem. Furthermore, in the silo application, the size of transmitter antennas can be enlarged to counteract possible attenuation for large distances. Tests in free air has shown a possible reception distance of 20 m using a beacon antenna of 80 cm.

4.2.2 Positioning measurement in free air

A measurement was made where the transponder was placed in free air, as shown in figure 29. The used area was approximately 7x12 m. Four Najad beacons were used, and the range for the 125 kHz beacon signal showed no limitations for the experiment size. Also the reception signal at 868 MHz was clearly readable, which was expected as there was no attenuating iron ore pellets in
Figure 26: The transponder used for position measurements. The transponder size is 43x27x7 mm. Seen is the three dimensional orthogonal antenna, which measures 12x12x3 mm.

Figure 27: The Craycom RFID reader used as position beacon. The reader size is 190x140x30 mm.

the path. The resulting estimated positional resolution was in the range of 0.4 m, with an accuracy of about 1 m. These results are fairly modest. One possible cause is an uneven reception in the three orthogonal antennas in the transponder. This results in an apparent position shift when the transponder is rotated around one of its three axes. The underlying cause of the uneven reception has been identified as an uneven coupling between the three orthogonal antennas and the surrounding magnetic field. Even though the reception antennas are designed for equal inductance, they have different areas. It is believed that this is the cause of the uneven reception. One remedy is to perform a more accurate calibration in preparation for future measurements.
Figure 28: Pellets storage area used for position measurements. The size of the pocket is approximately 4x4x2 m.

Figure 29: Experimental area for free air testing of positional accuracy.
5 Conclusion

The evaluation of the thermal insulation concept clearly has shown its feasibility to be used in the intended application. The use of liquid water as a phase change media proved to be highly efficient, and will be kept as the principle for the forthcoming sensor development. As the results presented in this report was achieved with relatively small outer diameters, we see high potential for long operating times in environments where larger sizes can be accepted, e.g. in the walking beam furnace. The radio transmission in pellets environment showed to be highly sensitive to the chosen radio frequency. Thus, for future sensor development, low RF frequencies such as 433 MHz or below will be prioritized. This is relevant for hot side sensors that measure process parameters, as well as for hot/cold side position sensors that need to transmit beacon strengths to overlying control systems. The positioning work itself shows that thorough calibration of reception coils is needed. Once this is done, we foresee a well functioning system.

6 References

References


