Particle Deposition in Gas Turbine Engines: The Effect of Temperature on Mineral Particles’ Behavior

Undergraduate Thesis

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By

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Deposition of dust minerals inside gas turbine engines is a serious problem for engine manufacturers. This phenomenon leads to damages inside the engine’s components which raised an issue for maintenance teams and engine designers to account for the negative effects of dust mineral deposition. In this study, the effects of temperature on dust mineral mixes were investigated through a series of experiments. Quartz, Dolomite, Albite, Halite, Gypsum, and Hematite were milled and sized to approximately 0 – 10 μm in diameter. The effects of temperature on these pure minerals are known, however, the effect of temperature on mixes of pure minerals is not as clear. For this reason, the experiments consist of pure minerals combined to form different blends. These blends are then exposed to a defined temperature inside a kiln facility at The Ohio State University’s Aerospace Research Center. The results in this study suggest a relationship between chemical composition and melting temperatures. In fact, melting temperatures can decrease with the existence of certain elements inside the mineral blend giving the deposit a high probability of sticking and eroding engine components. This research will provide modelers with insight about changes in mineral mixes and their packing densities due to temperature in a static setting. Thus, the results will increase the precision and accuracy in model prediction of particle deposition inside gas turbine engines.
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Chapter 1. Introduction:

Gas turbine engine’s designs are usually modeled under the basis that they would operate in clean air conditions. With the amazing growth seen by commercial and military aviation, gas turbine engines are pushed to breath air from environments that would be considered as dusty or polluted. Furthermore, mineral deposition inside gas turbine engines becomes inevitable when performing in dusty areas. The deposits can form and lead to erosion on engine components’ such as the turbine and compressor blades decreasing the engine’s performance [1]. In some cases, deposits make their way through cooling passages on the turbine section’s blade causing extremely dangerous high temperatures more likely leading to the engine’s failure [1].
1.1 Background

Many of the theoretical and experimental studies use solid mechanics principles when addressing deposition which won’t be accurate when the minerals reach melting states. Very few studies consider the chemical composition of the minerals and how it changes with temperature. Chemical composition of dusts is detrimental in order to determine the reactions that occurred that led to the deposition. In fact, it has been shown that minerals can react and transform to new secondary phases. These secondary phases can include a melt that can infiltrate the thermal barrier coating of the engine’s components eventually leading to corrosion of the parts [4].

This research focuses on the effect of temperature on packing densities and the chemical composition of dust mineral mixes in a static setting. Though the previous work has a dynamic setting of experiments when studying deposition, this research attempts to compare and potentially explain the results of the previous work using the current study’s results as temperature is one of
the main factors for deposition. In Elms et al. [5], The study consisted of analysis of in-service engines and also the analysis of components in a turbofan engine damaged intentionally through dust ingestion tests. For the dust ingestion tests, a new dust was developed using data of minerals present in regions of the middle east. This dust was then ingested inside a turbofan engine to ensure realistic results. The results of the ingestion test dusts show the formation of new minerals (Melilite Ca$_2$MgSi$_2$O$_7$–Ca$_2$Al$_2$SiO$_7$, wollastonite Zn$_2$SiO$_4$, diopside CaMg(SiO$_3$)$_2$ and anorthite CaAl$_2$Si$_2$O$_8$) in the hot sections of the engine. This confirmed the dust deposits and compositions observed from the in-service engines operating in the field. These minerals are known to form in high temperatures and based on their concentration and composition it is understood that the dust ingested caused erosion, for instance the high concentration of aluminum. Calcium and Magnesium are sourced from the decomposition of Dolomite due to high temperatures. This confirms how temperature and chemical composition are detrimental for deposition.

In Crowe et al. [6], Pure minerals Quartz, Dolomite, Albite, Halite, and Gypsum were milled to a size distribution between 0 – 10 μm in diameter. A blend of these pure minerals was used to make AFRL02 a commercially available test dust. These different dusts were then injected in effusion test plates simulating a turbine’s blade cooling path geometry. The results in this study show that albite produces the highest blockage per gram (BPG) in the cooling holes, however dolomite and salt produced the lowest BPG. As for the AFRL02 the BPG was very close to the one of Dolomite’s. No obvious correlation was found to explain the results when looking at the mechanical properties, adhesion forces, and thermal behavior of the different dusts used.

In Song et al. [8], Volcanic ash samples were gathered from 9 different volcano sites around the world. These samples were sized to less than 63 μm then exposed to high temperatures in a furnace (up to 1600 C). Song et al posited 4 different temperatures at which the samples change
phases of the ash: shrinkage or sintering temperature (ST), deformation temperature (DT), hemisphere temperature (HT), and flow temperature (FT). Sticking of the ash starts at the deformation temperature, and the phenomenon can be seen throughout higher temperatures. It is understood that an ash sample with a low concentration of SiO₂ melts more than the one with a high concentration of SiO₂ which makes volcanic ash more prone to interaction with jet engines than quartz particles. Water concentration is expected to decrease from 600 °C and be completed by 1200 °C. When it comes to sand comparison to ash, it is found that sand’s melts thresholds are much higher than the ones of ash. This makes using sand to model ash deposition inaccurate as it was noticed that there was a melt temperature threshold between the ash samples themselves due to chemical compositions. Additionally, volcanic ash is an amorphous structure, but common dusts and sands are crystalline though they could have very similar composition.

In Libertowski et al.[9] two tests were conducted: impulse kiln tests (similar to the test conducted in the current research) and high temperature deposition tests conducted in the high temperature deposition facility (HTDF). ARD (Arizona road dust) was the dust used in this study which was sized to a range between 0 to 5 μm. In this study 0.5g was chosen to be immersed in the kiln at an assigned temperature for different durations. ARD with size distribution of 0 – 5 μm was used in this study. The results obtained from the Kiln tests suggest that there is a transition temperature between 1300K and 1500K where the ARD sample starts to sinter thus melt at higher temperatures after that. Additionally, the color of the ARD samples is darker after this temperature. Comparing those results to the HTDF results, before the transition temperature the samples have a smooth conical shape. However, at the transition temperature the HTDF samples don’t carry the same shape nor smoothness. Looking at the PF results in this study, the kiln samples show an increase in the PF to a maximum value of 0.6 and 0.35 at 1400K and 1500K respectively. The
results from this study also confirmed that particle size has a major effect on melting, where ARD samples of different sizes (0-5, 0-10, 10-20, and 40-80 µm) were immersed in the Kiln at a temperature of 1535K for 1 hour. The results affirmed that particles of smaller size are more prone to melting and depositing than bigger size particles. Additionally, immersion times are another factor for deposition, where the longer the duration is the more melting happens. Similarly, in Plewacki’s et al. [7] the impulse kiln test and the HTD tests were conducted following the same procedures. However, the ARD size distribution was between 0 – 10 µm and 0.05 g of ARD piles were placed on ceramic plates and exposed to assigned temperatures inside the kiln. The temperatures used in the kiln facility and the HTDF are the same for this experiment. The results of these impulse kiln tests suggest the presence of two temperatures, T_s and T_m which are the dust’s softening and melting temperatures respectively. Packing factor (PF) was used as a variable to evaluate the results, where PF VS. Temperature was graphed. The first inflection point of the graph represented T_s and the second inflection point represented T_m. The HTDF PF results showed no similarity to the Kiln test PF results, an explanation for this is that the particles impact nominally at the jet temperature throughout the test, and particles experience the temperature from the hot jet for different durations. In Plewacki’s thesis [14], the same series of experiments were conducted on ARD and AFRL02. AFRL02 is one of the blends used in this study, and from the impulse kiln data, Plewacki recorded that AFRL02, that there are two transition temperatures. The first is around 1387K where the slope of the curve increases dramatically and the second transition occurs close to 1487K, when the packing factor trend seems to level off. For the HTDF results obtained for AFRL02, the packing factor was observed to show a relatively constant trend similar to the one seen from the ARD packing factor results.
This study attempts to understand the effect of temperature on mineral dust particles which could be found in the air. The behavior of mineral dusts can be depicted by changes in chemical composition, color, or adhesive forces between the particles. Thus, the question this research is attempting to answer: What is the behavior of mineral dust particles when solely exposed to gas turbine temperatures?

Chapter 2. Experimental Procedure:

Tests were conducted using the Kiln facility in The Ohio State University’s Aerospace Research Center. Square ceramic plates with dimensions were used to hold the dust samples as shown below in Figure 2: Kiln Facility Experimental Setup. 0.05g of dust was placed on the ceramic plates while the kiln is turned on to achieve the desired test temperature. The ceramic plate’s mass is noted, and the total mass (dust + ceramic plate) is noted before and after the test. The pure mineral dusts are milled to a size distribution between 0 – 10 µm, then blended to form different mixes. A table showing the mixes and their pure mineral percent content is shown in Table 1: Dust blends and their percentage of pure mineral content. A heat transfer calculation (using numerical methods to solve for the differential equations assuming a 1D problem) through
the dust pile has been performed, and for a 0.05g sample, heat would reach the whole pile in about 70 min. This result is based on simplified assumptions which led to an inaccurate result. The equations used to solve for the time are shown below:

Assuming 1D problem, steady state

\[
\frac{dT}{dt} = \frac{k}{\rho C_p} \frac{\partial^2 T}{\partial y^2}
\]  \hspace{1cm} (1)

\[-k \frac{\partial T}{\partial y} = h (T_{Kiln} - T_{deposit}) \]  \hspace{1cm} (2)

Where \( k \) is the thermal conductivity, \( h \) is the enthalpy, \( \rho \) is the density, and \( C \) is the specific heat capacity.

Based on the impulse kiln tests conducted previously, it has been decided that the dust samples would be immersed for 10 minutes. Once the test temperature is achieved, four samples at a time are set on a slider and the window cover is removed. The slider is then fully extended, and the samples are inside the chamber, the insulation attached to the back of the slider lowers the heat losses from any potential openings. Once the duration of immersion has reached 10 minutes, the slider is pulled away from the kiln, and the window cover is placed back to the kiln to maintain temperature. The samples now are cooled down through natural convection.
Table 1: Dust blends and their percentage of pure mineral content

<table>
<thead>
<tr>
<th>Dust Blend Name</th>
<th>Weight Percent of Quartz</th>
<th>Weight Percent of Gypsum</th>
<th>Weight Percent of Albite</th>
<th>Weight Percent of Dolomite</th>
<th>Weight Percent of Halite</th>
<th>Weight Percent of Hematite</th>
</tr>
</thead>
<tbody>
<tr>
<td>AFRL</td>
<td>34.0</td>
<td>30.0</td>
<td>17.0</td>
<td>14.0</td>
<td>5.0</td>
<td>0.0</td>
</tr>
<tr>
<td>AFRL - Quartz</td>
<td>0.0</td>
<td>45.5</td>
<td>25.8</td>
<td>21.2</td>
<td>7.5</td>
<td>0.0</td>
</tr>
<tr>
<td>AFRL - Gypsum</td>
<td>48.6</td>
<td>0.0</td>
<td>24.3</td>
<td>20.0</td>
<td>7.1</td>
<td>0.0</td>
</tr>
<tr>
<td>AFRL - Albite</td>
<td>41.0</td>
<td>36.1</td>
<td>0.0</td>
<td>16.9</td>
<td>6.0</td>
<td>0.0</td>
</tr>
</tbody>
</table>
After each test, the sample masses are noted to understand whether any losses have occurred from dehydration or gas formations. The mass of the deposited dust is calculated by subtracting the ceramic plate’s mass from the total mass after the test (2). The samples are then taken to a high-resolution 3D scanner to measure the dust deposit volumes. Packing factors are then calculated using the formula (3). The theoretical particle density $\rho_{particle}$ is the mass-weighted average of the densities of the constituents of the dust being studied Table 2.

$$PF = \frac{\rho_{deposit}}{\rho_{particle}} = \frac{m_{deposit}}{V_{deposit}} \frac{V_{deposit}}{\rho_{particle}} \tag{3}$$

$$m_{deposit} = m_{Total} - m_{plate} \tag{4}$$

<table>
<thead>
<tr>
<th>Dust Blend Name</th>
<th>Density (kg/m$^3$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>AFRL - Halite</td>
<td>2570</td>
</tr>
<tr>
<td>AFRL - Gypsum</td>
<td>2650</td>
</tr>
<tr>
<td>AFRL - Albite</td>
<td>2540</td>
</tr>
<tr>
<td>AFRL - Dolomite</td>
<td>2500</td>
</tr>
<tr>
<td>AFRL</td>
<td>2536</td>
</tr>
<tr>
<td>AFRL - Quartz</td>
<td>2500</td>
</tr>
<tr>
<td>AFRL + Hematite</td>
<td>2610.5</td>
</tr>
</tbody>
</table>
The volume $V_{\text{deposit}}$ measured in the 3D scanner can carry some uncertainties. The main source of uncertainty would be the plane of reference (datum) at which is always considered the top of the ceramic plate. This plane of reference is set manually by the user for every sample, since the thickness of the ceramic plates can be different. The uncertainty calculated for the volume scanner measurement differ from lower temperatures to higher temperatures. In fact, the higher the temperature at which test was conducted the higher the uncertainty (mainly for blends that shrink in volume). Furthermore, for the temperatures ranging from 1255K to 1370K we get a volume measurement uncertainty of $\pm 3.11 \text{ mm}^3$. However, for the test temperatures between 1420K and 1500K, we get a volume measurement uncertainty of $\pm 7.09 \text{ mm}^3$. A potential explanation for this is that most of the dust samples have experienced melting, and it becomes challenging to set the reference plane, since the height of the samples at higher temperatures becomes very low. This uncertainty in volume measurements is noticed in blends that experience major changes.

The temperature of the kiln does oscillate since the relay opens the circuit and closes it each time it is above and lower respectively than the assigned temperature. Though the presence of temperature oscillations, the average temperature at which the test is conducted varies by less than 1.3%.

**Chapter 3: Results:**

In this portion of the study, packing factor trends are analyzed to understand how different minerals change in their morphology. The tests were conducted at six different temperatures in a range of 1255K to 1500K with 2 samples per temperatures for repeatability. Each blend’s PF data is shown in the figures 3-9, and a plot containing the average PF data of all mineral blends is shown.
in figure 10. The morphology of the significant dust minerals at temperatures between 1366K and 1500K is shown in figure 11.

Figure 3: Packing Factor data obtained for AFRL02 0 – 10 μm during the impulse kiln tests

Figure 4: Packing Factor data obtained for AFRL – Quartz 0 – 10 μm during the impulse kiln tests
Figure 5: Packing Factor data obtained for AFRL - Gypsum 0 – 10 µm during the impulse kiln tests

Figure 6: Packing Factor data obtained for AFRL – Albite 0 – 10 µm during the impulse kiln tests
Figure 7: Packing Factor data obtained for AFRL - Dolomite 0 – 10 µm during the impulse kiln tests

Figure 8: Packing Factor data obtained for AFRL - Halite 0 – 10 µm during the impulse kiln tests
Figure 9: Packing Factor data obtained for AFRL + Hematite 0 – 10 µm during the impulse kiln tests
Figure 10: Average PF data for impulse kiln tests for all mineral blends 0 – 10 µm
Figure 11: Photographs of mineral blends at (1366K, 1422K, 1450K, and 1500K)
Between 1255 K and 1366 K, packing factors for all dusts is relatively constant which is expected at this range since most of the pure minerals haven’t reached melting nor decomposition temperatures. Changes in PF trends is observed in temperatures above 1366K for most of the mineral mixes. For the AFRL02 blend (Figure 3), we notice a gradual increase of PF until it reaches a value of 0.87 at 1500K. This means that the density of the sample is 87% close to the theoretical density of AFRL02 calculated from a micro level as the mass-weighted average of the densities of the constituents (Theoretical densities are calculated using the average atomic weight, volume per cell). In other words, the adhesive forces between the particles that formed at 1500K would be considered cohesive as the molecules within the solid body are interacting. Similarly, AFRL – Dolomite (Figure 7) have the same PF behavior at 1500K with a value of 0.85, but PF starts rising at 1450K. Looking at the morphology of this mineral blend starting at 1422K, we notice a gradual shrinkage and deformation of the sample as seen in Figure 11 until it reaches melting at 1500K where the deposit becomes a hard glass. We can observe similar PF trends for AFRL – Halite (Figure 8) where the PF value at 1500K is 0.84. However, the PF doesn’t rise until it reaches 1450K. Similar to the two previous minerals, we notice the gradual change in the morphology of AFRL – Halite. Packing factors trends are very close for the last three mineral blends.

Looking at the AFRL – Gypsum results (Figure 5Figure 11), the PF trend for this mineral was relatively constant between 1266K and 1422K. However, we notice an increase in PF value at 1450K, the sample at this temperature experiences shrinkage. At 1500K the PF value reaches 0.69, sintering and some melting can be seen in Figure 11. AFRL + Hematite is the mineral blend that experienced all major transitions posited by Song et al [8]. This mineral blend experiences sintering and some melting at 1422K then fully melted at 1450K, at this temperature the sample is
a hard glass that cannot be broken. At 1500K, where the average PF value is 0.93 the volume of the sample is very low (10.5 mm$^3$), the mass of the deposit decreased by 52%. 

Despite the high temperatures experienced by the AFRL – Quartz sample, no major transitions or changes in morphology has been observed. The PF trend for this mineral blend is relatively constant (Figure 4), ranging between 0.2 and 0.27. When evaluated, AFRL – Quartz was easy to break but relatively dry showing signs of dehydrations. Similarly, Figure 6 shows that AFRL - Albite did not experience any major transitions, where the PF reached 0.34 at 1500K. However, there is very minor shrinking observed at 1500K explaining the small PF increase.
Chapter 4: Discussion

4.1 Introduction

The results showed that apart from AFRL – Quartz and AFRL – Albite the mineral blends used in this study experienced major packing factor increases. Quartz has the highest percent weight inside the blends used in this study, its melting temperatures is higher than the maximum temperature used in this experiment (1500K). Melting temperatures and decomposition temperatures of each pure mineral is shown in Table 3. The results highly suggest that the mineral blends experienced major changes in their behavior and their melting temperatures decreased significantly compared to some pure minerals’ theoretical melting temperatures present in the mixtures. This section attempts to investigate the behaviors of the mineral dusts at different temperatures.

Table 3: Mineral melting temperatures and chemical compositions [10]

<table>
<thead>
<tr>
<th>Mineral</th>
<th>Chemical Formula</th>
<th>Melting Temperature (K)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quartz</td>
<td>SiO₂</td>
<td>1986</td>
</tr>
<tr>
<td>Gypsum</td>
<td>CaSO₄·2H₂O</td>
<td>*</td>
</tr>
<tr>
<td>Albite</td>
<td>NaAlSi₅O₈</td>
<td>1391</td>
</tr>
<tr>
<td>Dolomite</td>
<td>CaMgC₂O₆</td>
<td>+</td>
</tr>
<tr>
<td>Halite</td>
<td>NaCl</td>
<td>1074</td>
</tr>
<tr>
<td>Hematite</td>
<td>Fe₂O₃</td>
<td>1811</td>
</tr>
</tbody>
</table>

*Gypsum dehydrates between 373K and 573K to form CaSO₄ (Anhydrite) then subsequently decomposes into CaO and SO₃ between 1073K and 1573K.
+ Dolomite decomposes to MgO and CaCO₃ between 573K and 773K, then CaCO₃ decomposes to CaO, and 2CO₂ between 1073K and 1273K
4.2 Chemical Composition and Temperature Dependence:

4.2.1 Mineral Phases

At different temperatures, each constituent can behave differently contributing to a major change within the whole sample. It was observed that Quartz (SiO$_2$) can transform from a solid to a fluid state at 1986K and can also dissolve in a melt between 1373K and 1873K [10]. It was also found that Albite melts at 1391K, Hematite (Fe$_2$O$_3$) at 1811K, and Halite at 1074K. Thus, at a temperature between 1373K and 1873K, Quartz can dissolve in the minerals’ (Albite, Hematite, and Halite) melts. Hematite (Fe$_2$O$_3$) can also dissolve in melt between 1373K and 1873K [10]. As described in Table 3, Gypsum dehydrates to become anhydrite between 373K and 573K, then decomposes to calcium oxide (CaO) and sulfur trioxide (SO$_3$) between 1073K and 1573K. Dolomite also decomposes to calcium oxide (CaO), magnesium oxide (MgO) and carbon dioxide (2CO$_2$) between 1073K and 1273K. With that, for the type of minerals used in this study, the temperature threshold for major changes to occur is valued at 1373K. The variety of oxides that is present in a blend like AFRL02 is significant. Opie [11] defined flux as an oxide compound that lowers the softening and melting temperatures of other constituents in a sample. With the introduction of a small amount of flux in a blend, the melting temperature can be reduced. Opie explained that an oxide like Na$_2$O can chemically break the Si and O bonds found in Quartz and Albite disrupting the ordered crystal. In fact, the greater the variety of oxides the lower the melting temperature in a mineral mixture [11].
4.2.2 AFRL02 Result Analysis:

At 1422K, AFRL02 wasn’t observed to experience any major melting despite the potential presence of a variety of oxides (CaO and SO$_3$ from the decomposition of Dolomite and Gypsum, MgO and CO$_2$ from Dolomite). The sample have experienced minor sintering observed in the hardness and the sticking of the particles together and to the ceramic plate. With that, elements like Albite and Halite have melted, this can be confirmed by the change of the color of the sample from very light red to a light white color. Albite mineral particles have a reddish color at room temperature but when blended with “white” minerals the mixture adapts a light red color. The time of exposure in the kiln is another factor that led to the change experienced at this temperature. In fact, the longer the time exposure of the sample inside the kiln the more changes the minerals will experience. With the described factors in place, at 1450K we notice major sintering and some melting. However, at 1500K the AFRL02 sample is fully melted and that is due to the presence of a variety of oxides that broke the silicon and oxygen bonds lowering the melting temperature of the mixture. Additionally, the dissolution of Quartz in the Albite and Halite melts contributed to the transformation of the blend from a solid to a fluid state at 1500K. The mass of the sample has decreased by 33%, this can be explained by the dehydration of the constituents mainly Gypsum, and gas formation from the decomposition of Dolomite. The circumstances described have all led to the melting AFRL02 at a temperature between 1450K and 1500K. Hence, the high packing factor value of 0.87 obtained at this temperature.
4.2.3 AFRL + Hematite Result Analysis

AFRL + Hematite, not like any other mixture experienced major packing factor increases starting at 1422K. At this temperature, the packing factor was valued 0.76 and from Figure 11, the volume of the sample decreased significantly where major sintering and noticeable melting occurred. At 1450K, the blend melted completely, this can be explained mainly by the presence of a variety of oxides seen in AFRL02 in addition to the small amount of Hematite (a pure oxide originally present in the mixture not a result from the decomposition of Dolomite or Gypsum). Additionally, as described in the Introduction of this section (4.1), Hematite can dissolve in a melt between 1373K and 1873K, where 1422 – 1500K falls within that range. At 1500K, the packing factor value reached 0.93 and the sample mass was reduced by 48%, this only indicates major gas formation and dehydration within the mixture. Since the kiln provides heat by radiation and compared to the other mixtures AFRL + Hematite has a darker color, this blend would have high heat absorptivity. In fact, it was observed that when this blend is inside the kiln the color of the sample is even darker which gives it higher heat absorptivity. Thus, this blend may be absorbing more heat than the other mixtures present. This is another potential explanation for the way AFRL+Hematite behaved compared to the other mixtures.

4.2.4 AFRL – Dolomite Result Analysis

AFRL – Dolomite at 1422K experienced shrinkage and some sintering but no obvious melting. Similar to AFRL02, Albite and Halite have already melted at this temperature (which is above the theoretical threshold of 1373K where decomposition, melting and dissolvement occur) this allows the potential dissolvement of the Quartz content in the existing melt though no obvious melting was observed until the test is ran at 1450K. At 1450K, minor melting and major sintering
can be seen in the morphology. As Gypsum (the main source of the oxides at high temperatures) constitutes 34.9% of this mixture, at 1450K, the oxides resulted from decomposition allowed the melting temperature of the mixture to decrease. We can observe melting at 1500K but not in the same degree experienced by AFRL02, with the reason being that the variety of oxides in AFRL02 is highly present as opposed to AFRL – Dolomite. This shows that the presence of multiple different oxides with silica minerals makes deposition/melting at lower temperatures inevitable.

4.2.5 AFRL – Halite Result Analysis

AFRL – Halite behaved approximately the same exact way as AFRL02, however, the packing factor for this mixture was relatively constant until reaching 1450K. Figure 11 shows that there was no obvious shrinking nor sintering of the sample at the temperatures before 1450K. In this case, Dolomite and Gypsum are both present and provided a variety of oxides, Albite melted which facilitated the dissolvement of Quartz but not in the same way as AFRL02 given that Halite is not present. The mixture did fully melt at 1500K the same way as AFRL02 did though the absence of Halite. Thus, AFRL – Halite experienced major changes between 1450 – 1500K. This result suggests that 6% of Halite in a mixture can only cause changes in a sample at a slightly higher temperature.

4.2.6 AFRL – Gypsum Result Analysis

AFRL – Gypsum (similar to AFRL – Halite) had a relatively constant packing factor until 1450K. The samples at 1450K experienced very low degrees of sintering as the minerals became harder and sticked to the ceramic plate. High degrees of sintering and very minor melting was observed at 1500K which can be explained by the dissolvement of Quartz in Albite and Halite
melts. Additionally, the presence of oxides (CaO, MgO, CO₂) from Dolomite lowered the melting temperature of the mixture. However, the packing factor value at 1500K is 0.69 which is lower compared to the previous mixtures. This shows how Gypsum can highly affect the behavior of the mixture by providing more oxides thus, decreasing the melting temperature of the sample.

4.2.7 AFRL – Albite and AFRL – Quartz Result Analysis

AFRL – Albite didn’t experience major changes compared to the previous mixtures where the PF value reached 0.34 at 1500K. Despite the presence of all AFR02 oxides and Quartz, there was no major changes observed in this mix. The blend contains 41% of Quartz and 6% of Halite, the results can be explained by the fact that the amount of Quartz didn’t fully dissolve in the Halite melt at 1500K. In the previous mixtures, the Albite melt present between 1422 – 1500K allowed for the Quartz dissolvement. Additionally, the amount of oxides and silica minerals present in the previous mixtures is comparable which allows the oxides to affect the silica minerals’ bonds. However, this mixture contains a high percentage of oxide minerals where Gypsum and Dolomite combined constitute 53% of the mixture. The main factors are the low presence of silica minerals in the mixture and the low presence of melted material necessary for Quartz to dissolve. AFRL – Quartz had a similar response to AFRL – Albite since the only change between the two mixtures is the type of silica mineral introduced. For similar reasons, the silica minerals present in AFRL – Quartz is low as opposed to the high presence of oxide sources (Gypsum and Dolomite) where Albite constitutes 25.8% of the mixture as Gypsum and Dolomite combined constitute 66.7%. This explains the relatively constant PF trends throughout the experiments’ temperature range.
4.3 Material Size Distribution Dependence:

It is known that size distribution plays an important role in facilitating the reactions between minerals within a sample. Figure 12 shows an experiment conducted by Libertowski et al. [9] showing the behavior of the material as it depends on its size. The blend used by Libertowski et al. is ARD which has different constituent than most of the blends used in the current study. However, ARD does contain silica minerals and a variety of oxides which would make the potential reactions in ARD at high temperature somewhat similar to most of the mixtures used in the current work. Libertowski et al. noted that some constituents of the dust can be highly present in a size distribution but show up in a smaller portion in a different size distribution, this can be a factor for the potential reactions happening in a sample. Comparing this result to the current study’s results, we understand that the size distribution of 0 – 10 μm of the mineral blends facilitated the reaction occurrence between the different compositions (when the right compositions are present). Furthermore, it would either take a longer time or a higher temperature for melting to occur if the blends used in the current study had larger a size distribution.

![Figure 12: Impulse Kiln tests immersed for 1 hour at 1535K performed with a gradual ramp rate [9]](image-url)
4.4 Comparison to Crowe et al. [6]:

Crowe conducted experiments by injecting dust minerals into effusion plates as described in the background section of this study (1.1). The temperature at which the tests were conducted is 1116K for the plate and the flow temperature is 950K which are both lower than the determined temperature threshold of 1373K where chemical changes are expected to occur in a pure mineral (4.2.1). Heat transfer occurs through convection when the particles are ingested in the coolant flow then through conduction when the dust particle attach to the effusion plate. The temperatures at which the tests are performed wouldn’t influence Quartz and Albite since their melting temperatures is much higher. However, Halite is expected to potentially melt, and Gypsum and Dolomite can potentially decompose at the test’s temperature into a variety of oxides. Figure 13 shows the blockage per gram (BPG) data obtained by Crowe et al. where the orange dots represent the BPG. The OSU – mixed AFRL02 (the same AFRL02 used in the current study) experienced below average blockage of the cooling paths. When the AFRL02 is injected in the cooling flow, dehydration of Gypsum and decomposition of Dolomite into MgO and CaCO3 can occur. When the AFRL02 reaches the effusion plate which is at 1116K, Dolomite and Gypsum can potentially decompose into the variety of oxides described in (4.2.1). Despite the presence of oxides, the silica minerals didn’t reach melting nor dissolvement temperatures. Halite is expected to melt at 1116K, but its low amount present wouldn’t affect quartz to dissolve therefore not facilitate melting. However, a potential hypothesis for the AFRL02 results observed in Figure 13, is that the Halite melt did contribute to the sticking of the different minerals together therefore show very minor signs of sintering.
A potential explanation for the low BPG resulted by Halite (Salt) is that at 1116K most of this sample transformed from a solid state to a fluid state. For the time of the test, as long as the salt is fluid no blockage should be expected until the test is over and the temperature drops. This would lead the salt to solidify or if there were any last particles injected, they wouldn’t experience any melting since at the end of every test the Kiln temperature would decrease in a fast way. Albite showed the highest BPG despite not reaching melting temperatures and Quartz resulted in approximately a third of the Albite’s BPG even though both dusts are silica minerals. Albite does contain Sodium and Aluminum in its chemical formula, which makes the mechanical properties of both dusts different.
4.5 Comparison to Song et al. [8]:

As described in the background section of this thesis (1.1), Song et al. conducted experiments similar to the experiments conducted in the current work but using volcanic ash. The size distribution of the ash particles was 0 - 63 µm and four characteristic temperatures were determined by the authors as shown in Figure 14.

![Figure 14: Geometrical definition of four characteristic temperatures in the volcanic ash melting process from Song et al. [8]](image)

The four characteristic temperatures will change with respect to the size distribution of the mineral particles based on the results obtained by Libertowski et al. [9]. The authors understood that a low concentrations of silicon dioxide melts more than the one with a high concentration of silicon dioxide which makes volcanic ash more prone to interaction with jet engines than quartz particles. Based on the results of the current study, we understand that melting temperatures decrease through the mixing of different minerals which is a characteristic of volcanic ash. This confirms the idea that volcanic ash more prone to sticking and interacting with jet engines than Quartz particles. In fact, blends of minerals are more prone to interacting with jet engines than pure Quartz particles. However, quartz or any silica mineral is a major constituent that allows for melting temperatures to decrease and lead to deposition at a lower temperature. This is confirmed in the results seen in AFRL – Quartz and AFRL – Albite (4.2.7) where very minor changes in the packing factor occurred (No changes at all for AFRL – Albite). The concentrations of SiO₂ in a
blend plays a major role in melting but at the same time the presence of oxides and minerals with low melting temperatures has a major contribution to melting of a sample. It evident that there is a potential ratio of silica minerals to base oxides in a mixture that would result in lowering a mixture’s melting temperature.

4.6 Comparison to Plewacki [14]

The experiments conducted by Plewacki et al. [14] consisted of tests in the HTDF (high temperature deposition facility) and in the impulse kiln facility. Plewacki use ARD and AFRL02 both with a size distribution of 0 – 10 µm, the same as the one use in the current study. The ARD PF results from both test is shown in Figure 15: Packing Factor data for 0-10 µm ARD in the Impulse Kiln tests and HTDF tests (150 m/s) from Plewacki et al. [14].

![Figure 15: Packing Factor data for 0-10 µm ARD in the Impulse Kiln tests and HTDF tests (150 m/s) from Plewacki et al. [14]](image-url)
The PF data from the impulse kiln tests show two transition temperatures represented in the inflection points observed at 1375K and 1437K. The first inflection point represents the start of sintering and shrinking of volumes until it reaches melting with higher PF values. The second inflection point represent the fall of PF values due to the highly porous nature of the deposit. Plewacki explained that this porosity is caused by either trapped air or outgassing that is contained within the outer shell of the dust as it rapidly melts during the quenching process. The PF values obtained from the kiln tests for ARD are close to the PF values observed in the AFRL – Gypsum sample. Table 1,Table 4 show the composition of AFRL – Gypsum and ARD respectively. A potential explanation for this is that the ratio between the amount of oxides and silica minerals for ARD is similar to the ratio between oxides and silica minerals found in AFRL – Gypsum despite the major differences in bulk chemistries. The percentage weight of silica minerals contained within AFRL – Gypsum is about 72.9% and the one found in ARD is between 68 – 76%. However, the PF changes for ARD occur at a lower temperature compared to the changes noticed in AFRL – Gypsum. This can be explained by the additional constituents of ARD that contributed to melting differently than the ones present in AFRL – Gypsum.

Table 4: ARD chemical composition

<table>
<thead>
<tr>
<th>Chemical</th>
<th>Percent by Weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO₂</td>
<td>68-76 %</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>10-15 %</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>2-5 %</td>
</tr>
<tr>
<td>Na₂O</td>
<td>2-4 %</td>
</tr>
<tr>
<td>CaO</td>
<td>2-5 %</td>
</tr>
<tr>
<td>MgO</td>
<td>1-2 %</td>
</tr>
<tr>
<td>TiO₂</td>
<td>0.5-1.0 %</td>
</tr>
<tr>
<td>K₂O</td>
<td>2-5 %</td>
</tr>
</tbody>
</table>
Figure 16: Comparison of top view photographs for 0-5 µm ARD obtained from the 30-minute HTDF facility from Plewacki et al. [7]

Figure 16 shows different ARD morphologies a different temperature from the HTDF and impulse kiln tests. At 1500K, the kiln results showed a fully molten ARD sample. Differently, the ARD results show particles that are still mainly solid, but we can still observe very minor melting on the top of the sample. It can be observed that the color change in the Kiln is from brown to approximately black as opposed to the HTDF where the ARD samples stayed in the brownish spectrum. A potential explanation for this is the heat mechanism used the two experiments. The main heat mechanism found in the Kiln tests is radiation, differently, the one found in the HTDF tests is convection. Radiation heat would make the particles surely behave differently than convection heat since variables such as absorptivity and emissivity are in place in the heat transfer process inside the Kiln.
Plewacki conducted the same series of tests for AFRL02. The HTDF and Kiln results obtained for AFRL02 are shown in Figure 17.

![Figure 17: PF data for 0-10 \(\mu\)m AFRL02 from the impulse kiln and HTDF from Plewacki et al. [14]](image)

The PF data for AFRL02 obtained by Plewacki are close to the ones obtained in this study (shown in Figure 3) which is expected since the same mineral blend is tested. The current study’s results for AFRL02 confirms what has been resulted in Plewacki et al [14]. However, in Figure 17 smaller temperature increments are used in the kiln tests which showed the trend levelling off between 1475 – 1500K, this result should then be expected in further kiln tests of AFRL02. The HTDF packing factor trend for AFRL02 is relatively constant (similar to what has been observed in ARD) with an average PF value of 0.53 and ARD showing an average PF value of 0.40. This difference is surely due to the material composition of the samples. A potential explanation for the
constant aspect of the PF seen in the HTDF testing is that the heat mechanism at which heat is transferred to the particles is convection. But the kiln heat transfer type is radiation and variables such as absorptivity are in place which would make the particles behave differently than the ones in the HTDF facility.

4.7 Comparison to Impingement Deposition Experiments [15]

Eric Nied conducted experiments in the CoR rig facility. This facility simulates deposition on the cool side of a turbine air foil or combustor liner of gas turbine engine. The experiment consists of a pressurized feed box where dust is injected at a constant rate, the dust is then pushed down into a funnel which then drives the dust into a jet flow (main line) with a velocity of 57 m/s. Before reaching the main line, the dust is passed through a device that breaks any clumps formed in the sample to keep a constant size distribution. The dust minerals with a size distribution of 0-10 µm exits the main line and impinges normally upon the center of a nickel base alloy plate (Hastelloy X). The plate is heated from behind using a methane-oxygen torch at the same time as it is cooled from the impingement flow. A schematic of the experimental set up is shown in Figure 18. The mineral blends used in this series of experiments are the same as the ones used in the current study. The nomenclature for the blends is somewhat different, where for instance, Mix 2: No Gypsum would be the same blend as AFRL – Gypsum and Mix 7: w/ Hematite would be the same as AFRL + Hematite.
Figure 18: Schematic of Impingement Deposition Rig.

For this series of experiments, capture efficiencies (CE) were calculated which is the ratio of the mass of the deposit to the mass delivered to the plate (5).

\[
\text{Capture Efficiency} = CE = \frac{\text{Mass of Deposit on Target}}{\text{Mass Delivered}} \quad (5)
\]
Figure 19: Capture efficiency for pure minerals
The results of the impingement deposition experiments shown in Figure 19 and Figure 20 where the blue, orange, and grey charts represent tests conducted at 1033K, 1144K, and 1255K respectively. The capture efficiency obtained from the different blends does increase with temperature. However, the blends that show higher capture efficiency is not consistent with the packing factor result obtained for that particular sample. For instance, AFRL – Quartz (Mix 6: No Quartz) show one of the highest capture efficiencies at 1255K. In fact, Mix 1, Mix 2, Mix 4, Mix 5, and Mix 7 are all known to have a significant increase in packing factor, differently, we notice higher capture efficiencies for pure minerals like Hematite (Which melting temperature is 1811K) and Albite. The impingement deposition experiments showed that the plate can also reach very
high temperatures as close to 1500K. Despite that, the results obtained from the impulse kiln tests are different than the results obtained from the impingement deposition experiments. As mentioned in section 4.6, the heat mechanism would play a major role in the behavior of the samples, the kiln heat transfer process is radiation, but the CoR rig’s heat mechanism would mainly be convection which would make the results different.
Chapter 4: Conclusion and Future Work Recommendations

The goal of this research has been to understand the behavior of dust mineral blends at higher temperatures. Impulse kiln experiments were conducted on mineral mixtures constituted of pure minerals to understand how blends behave at higher temperatures. Packing factors which is the density of the deposit (mass of the deposit over its volume) over the theoretical density of the blend was calculated. Packing factors is used as a metric to understand the morphology of the mixture samples. Blend mineral compositions play a major role in melting of the samples, the test results showed that changing one of the constituents in a blend can significantly affect its behavior. Size distribution is a major factor for the particles to react to the assigned temperatures and interact with each other to form new minerals. Percentages of silica minerals and percentages of oxides (or oxide sources) play a major role for melting or any significant behavior to occur within a dust sample.

Six pure minerals were combined to form several blends that were tested in this study. AFRL + Hematite is a mixture that was seen to show major changes with temperature. Despite the presence of minerals that can only melt at temperatures higher than the ones used in the series of tests, melting was observed in most of the samples containing silica minerals (Quartz and Albite) and at least an oxide source (Gypsum or Dolomite). This was explained by dissolution of minerals like Quartz in a melt already existing an assigned temperature. Additionally, the presence of oxides contributed to breaking the SiO$_2$ bonds leading to a decrease of melting temperature of the blend by changing the structure of the molecules, thus the eutectic point.
To understand the deposition phenomenon and how temperature affects mineral particles, the current study’s results were compared to results from other deposition experiments such as the effusion plate tests by Crowe [6], HTDF tests by Pelwacki [14], and the Impingement jet tests. From this comparison, it is understood that the behavior of the blends inside the kiln is significantly different than the behavior of the particles in any of the dynamic experiments since the heat mechanisms are different.

There are additional tests that can be conducted in order to gain a deeper understanding of deposition. In terms of the chemical compositions relating to melting temperatures, more impulse kiln tests can potentially be performed with different composition percentages. With this information, modelers can have a good idea about melting temperatures and how they can change with the percent of mineral constituents or chemical compositions. At the same time, x-ray diffraction (XRD) would be a valuable tool to determine which constituents reacted with one another in each blend. For deposition experiments inside an internal cooling geometry, chemical compositions will result in different mechanical properties, a high BPG would require a mineral that would not leave any air trapped between particles in the cooling paths. It would be valuable to determine how does the amount of air trapped between particles blocking a hole change with different minerals at the same test temperatures 1116K. In other words, determine packing factors or some type of metric that would relate the volume blocked to the volume of the cooling path. From this, understand how this packing metric of different minerals and blends changes considering chemical compositions and how they could possibly relate to the mechanical behavior observed in the effusion plate testing.
Bibliography


[15] Eric Nied IGTI paper. Accepted for presentation in the IGTI 2022 conference in Rotterdam, Netherlands