ACTIVATION ENERGIES OF SPARK PLASMA SINTERED
OXIDE DISPERSION STRENGTHENED STEELS

by

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Spark plasma sintering (SPS) was used to consolidate powders to create oxide dispersion strengthened (ODS) steel samples. Three different compositions of powder were used. These were Fe-16Cr-3Al (wt.%), Fe-16Cr-3Al-0.5Y2O3, Fe-16Cr-3Al-0.5Y2O3-1Ti powders. These materials are of interest for their potential application as cladding of nuclear fuel rods. Hardness and density of the samples are presented. The effects of composition, milling, and heating rate were explored. Heating rate has little effect on density and hardness. The Young and Cutler method was used to find the grain-boundary diffusion and volume diffusion activation energies. These values are reported for the different compositions and different mill times.
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Chapter 1

Introduction

1.1 Sintering

Sintering is a thermal treatment for bonding particles into a coherent predominantly solid structure. A powder is taken, which is made up of small particles, and heated below the melting temperature. When the powder is heated, the particles bond together and become one solid piece.

The process of sintering is described by different stages. Before any sintering takes place, the particles are in contact with one another. In the initial stage of sintering, mass is transported in the system and the particles start to bond together, creating grain boundaries and pores. The connections between the particles are often referred to as necks. Very little densification takes place during the initial stage of sintering. The particles start to connect, but the space between the particles, and therefore the total volume, stays the same. The initial stage ends when the ratio of neck length to particle diameter reaches approximately 0.3 [1].

The intermediate stage is the next stage of sintering. This is the most important stage for densification. During this stage the properties of the solid are determined.
Chapter 1 Introduction

The pores start shrinking due to diffusion of particles and vacancies. In the final stage of sintering, the pores become isolated from one another. When compared with the initial and intermediate stages of sintering, the final stage is a very slow process [1]. Figure 1.1 illustrates the stages of sintering.

Figure 1.1 The stages of sintering are represented here. The spheres start out in point contact. The particles develop necks where they are touching, creating grain boundaries where they are touching [1].

1.2 Diffusion

The driving force for sintering is lowering the system energy. When the particles are separate as in the powder, a large surface area exists. A large amount of surface energy is associated with this large surface area. It is energetically favorable for the particles to combine into a solid, thus lowering the surface energy. A chemical potential difference exists between a flat surface and a sphere. This leads to a stress that directs mass flow during sintering [1]. The powder is approximated by spheres which become flat as sintering occurs.

The system lowers its energy by means of diffusion. When the temperature of the powder is increased, the stress in the system directs the flow of atoms causing
1.3 Spark Plasma Sintering

Diffusion. Volume diffusion is the motion of particles or vacancies through a crystalline structure. Sometimes this involves motions from a surface site on the particle to another surface. When this occurs, there is no densification of the sample. Another path that the particles can follow is from the neck to the grain boundaries between the particles. This leads to densification and shrinkage of the sample [1].

Another diffusion mechanism that takes place during sintering is grain boundary diffusion. Atoms or groups of atoms are transported along the grain boundary to the necks. This diffusion process also leads to densification and shrinkage [1]. An activation energy is associated with both the volume diffusion and the grain boundary diffusion.

Several different methods for sintering exist. Some of these methods use pressure and others do not. Temperature is a necessary element for sintering. There are many different methods for raising the temperature of the powder. This work used the spark plasma sintering (SPS) method for the research. Therefore, the focus throughout this text is on SPS.

1.3 Spark Plasma Sintering

Spark plasma sintering may actually be a misnomer. Some think that there is a creation of plasma in SPS. However, it has been shown that plasma may not exist during the sintering process [2]. Regardless of whether a plasma is actually formed or not, this text will use SPS for the name.

SPS uses a high amperage, low voltage direct current to raise the temperature of the powder. The current is pulsed. The powder is placed in a die with two punches. The current goes through the punches (and the powder if it is conductive) and heats the powder by Joule (or resistive) heating. Figure 1.2 is a schematic of an SPS
machine.

**Figure 1.2** A schematic of the SPS machine. This is a side view as if everything were cut in half. The powder is placed in a graphite die in between two graphite punches. Uniaxial pressure is applied. A pulsed current provides the necessary energy for sintering to take place.

SPS has several advantages over other sintering methods. One of the major advantages of SPS is sintering time. SPS can sinter a sample in minutes, which would take other methods hours or even days to finish. The required temperature for full densification is also significantly lower compared to other processes [2]. These advantages have led to an interest in SPS.
1.4 Understanding Sintering

As sintering has become more common, people have tried to develop methods for understanding the sintering process. One of the concepts that has been explored is the activation energy required for sintering. Johnson [3] developed the following equation for studying grain boundary and volume diffusion.

\[ y^2 \frac{dy}{dt} \simeq \frac{2.63 \gamma \Omega D_V y}{kT a^3} + \frac{0.7 \gamma \Omega b D_B}{kT a^4} \] (1.1)

Here \( y \) is the fractional shrinkage (that is \( \Delta L/L_0 \) where \( \Delta L \) is the shrinkage and \( L_0 \) is the original length), \( \gamma \) is the surface tension, \( \Omega \) is the volume of vacancy, \( D_V \) is the volume diffusion coefficient, \( b \) is the grain boundary width, \( D_B \) is the grain boundary diffusion coefficient, \( k \) is Boltzmann’s constant, \( T \) is the temperature (K), \( a \) is the particle radius, and \( t \) is the time.

To separate the volume diffusion and grain boundary diffusion, Young and Cutler [4] modified equation 1.1 to

\[ \ln \left( yT \frac{dy}{dT} \right) = \ln \left( \frac{2.63 \gamma \Omega D_V y}{k a^3 c} \right) - \frac{Q_V}{RT} \] (1.2)

\[ \ln \left( y^2 T \frac{dy}{dT} \right) = \ln \left( \frac{0.7 \gamma \Omega b D_B y}{k a^4 c} \right) - \frac{Q_B}{RT} \] (1.3)

where \( c \) is the heating rate, \( Q_V \) is the volume diffusion activation energy, and \( Q_B \) is the grain boundary diffusion activation energy. By plotting \( \ln(yT \frac{dy}{dT}) \) and \( \ln(y^2 T \frac{dy}{dT}) \) vs. \( 1/T \), the activation energies can be found by fitting a line and finding the slope. The literature shows that this method works for Mo-30W (wt.%) [5]. This is the method that will be used in this work.

In order to understand more about sintering, this work also finds the density and hardness of sintered materials. The materials looked at in this work are oxide
dispersed strengthened (ODS) steels. Samples of Fe-16Cr-3Al (wt.%) (FCA), Fe-16Cr-3Al-0.5Y2O3 (wt.%) (FCAY), and Fe-16Cr-3Al-0.5Y2O3-1Ti (wt.%) (FCAYT) are sintered. FCAY and FCAYT are ODS steels while FCA is not. Research is done on ODS steels for its possible use as cladding in nuclear reactors [6].
Chapter 2

Procedure

2.1 Milling

In this work, we studied the effects of milling. Milling can change the properties of different powders. In planetary ball milling, the powder is added to a grinding jar along with some metal balls. The jar rotates around the center of the sun wheel and it also rotates about its own axis in the opposite direction. A schematic of a planetary ball mill is shown in figure 2.1. Milling is different than mixing. If you mix two different powders, you could still separate them out with enough patience and small enough tweezers. After two powders are milled, they are still a powder, but they cannot be separated.

Samples were milled with a Retsch PM100 planetary ball mill. To prepare for milling, we put the balls that we planned on using into the grinding jar we would use. We added acetone to the grinding jar and set it to mill for approximately 10 minutes at 380 RPM. This was to clean the jar and the balls. After the 10 minutes were up, we cleaned off the balls, rinsed the jar, added more acetone, and set it to mill again. We did this a total of three times. After the jar and balls were clean, we put them in
Chapter 2 Procedure

Figure 2.1 A schematic of the planetary ball mill. The large outside wheel (the sun wheel) rotates in one direction. The grinding jar rotates in the opposite direction at a ratio of 1:-2.

We used a glove box to protect the powders before they were sintered or milled. A glove box is a large plastic box that is sealed from the outside air with sealed gloves inserted through holes. The gloves are present to be able to prepare samples and powders without putting them in the atmosphere. The glove box was kept at a nitrogen atmosphere so that the powders do not oxidize. We put the jar and the balls in the glove box. Next we measured 30 grams of the powder and added it to the jar with the balls. We placed a special lid on the jar, which sealed the jar and also had two valve stems. After removing the jar from the glove box, we used an argon tank to fill the jar with argon. After waiting for the powder to settle, we filled with argon again. We wanted to purge the nitrogen from the jar. We did this about ten
times to remove as much nitrogen as possible. If nitrogen remained in the jar during milling, it would combine with the powder to create nitrides. This was undesirable, so we replaced the nitrogen with a noble gas.

Next, we placed the jar in the mill. We tightened the clamp to make sure the jar did not move during the milling. The counterweight was adjusted to the appropriate level. We set the mill to run at 380 RPM for the desired mill time. The mill shut off if the counterweight was not in the appropriate spot. When this happened, we adjusted the counterweight and started the mill again.

After the mill finished running the desired time, we placed the jar back in the glove box. We removed the lid and separated the balls out with forceps. We carefully poured the powder into a tray. Next, we replaced the balls and the lid and shook the jar to loosen more powder. We did not scrape the sides of the jar as this had the potential to introduce material from the jar that would change the powder we milled. After removing as much powder as possible, we poured the powder into a vial, which we kept in the glove box.

2.2 Sample Preparation

In preparation for the sintering, all of the components were cleaned. The die and punches were wiped clean with acetone and paper towels. A paper towel was wrapped around a spatula and covered in acetone to clean the inside of the die. Care was taken to not use the sharp end of the spatula on the graphite die. This could lead to grooves and scrapes in the die which would be undesirable. The flat edge of the spatula was pushed against the sides with the paper towel.

We cut some graphite foil to line the edge of the die and place over the punches where the powder would touch. This was necessary to ensure that the powder would
not sinter to the inside of the die or to the punches. After lining the die with graphite foil, we put the die and punches into the glove box by means of a load lock. After placing these in the glove box, we measured out approximately 3 grams of metal powder. The powders were kept in a glove box to make sure they did not oxidize. The glove box had a nitrogen atmosphere with less than 400 ppm oxygen. The bottom punch was placed in the die so that the powder would not fall out. After weighing the powder, we put the powder in the die and placed the top punch. The punches fit just right in the die so that the powder does not fall out and very little oxygen will be able to enter. Next, we removed the die and pressed the powder to 2050 pounds force. Since the inner diameter of the die is 12 mm this worked out to be 80 MPa pressure. This was to compact the powder at the pressure we would use in the SPS.

### 2.3 Sintering

The Dr. Sinter 1500 Spark Plasma Sintering System is the machine used to complete the sintering for this research. It is a high current low voltage machine. Dr. Sinter controls the temperature by reading a thermocouple. It tracks where the temperature needs to be, where it is, and where it has been and makes the necessary adjustments to follow the temperature profile. Dr. Sinter also gives a readout to a computer of displacement, pressure, current, and voltage.

After the powders were compacted, the die was ready to be placed in the chamber of the Dr. Sinter system in preparation for sintering. Several graphite cylinders are stacked on top of each other to conduct electricity through the die. Care must be taken to ensure that the cylinders are all placed concentrically in the center of the stage of the SPS. A continuous upward force is exerted during the sintering. If the cylinders are not placed correctly, the whole thing will fall down when the stage rises.
After everything was in the appropriate position, pressing the two green buttons when the switch is in the up position raised the stage. There are two buttons as a safety to make sure the operator does not put his or her hands in the chamber when the stage is moving. The stage rises until the top of the highest cylinder is against the upper plate of the chamber. At this point we placed the thermocouple in a hole in the center of the die. Next we closed the chamber by turning a switch to the side marked chamber and pressing the two buttons.

Next, we evacuated the chamber to about 12 Pa and then filled the chamber with argon gas. We then evacuated the chamber again. The chamber was purged three times to lower the oxygen content in the chamber. The sintering takes place in a reduced pressure to ensure that the graphite does not burn. The vacuum pump stays on the whole time the sintering process takes place.

The upward pressure can be controlled automatically or by the operator. For the samples studied, we chose to control the pressure. After the sintering starts, we first paid close attention to the current and voltage gauges to make sure the current didn’t spike. If the current were to spike in the beginning, we would know that something went wrong and have to abort the run. Anything out of the ordinary was a sign that something was not working correctly. After making sure that everything started as expected, we increased the upward force by turning a dial on the SPS. We kept a force of 9 kN throughout the sintering. As the powders sintered, the force changed, so we kept an eye on the force readout and made adjustments as needed.

The Young and Cutler [4] method uses the displacement during sintering to find the activation energy. To account for thermal expansion, we did a few runs at different heating rates with no powder. The steps in the run were the exact same as the runs we had with the powder. By this method, we measured the thermal expansion of the graphite so that we could account for it in the data. Figure 2.2 shows the displacement
correction. Positive values correspond to shrinkage while negative values correspond to expansion. The corrected curve is found by subtracting the values measured for the graphite from the measured values. It is seen that the graphite actually shrinks a little bit in the beginning. This is consistent with the literature [7].

![Graph showing displacement correction](image)

**Figure 2.2** This graph shows the correction to take into account the expansion of the graphite. The dashed line is the measured displacement, the dotted line is the measured displacement for a run, and the solid line is the corrected displacement. The corrected displacement is found by subtracting the graphite curve from the measured curve.

### 2.4 Density Measurements

After the samples were prepared, we polished away all the extra graphite foil to take a density measurement. The samples were examined to make sure that they were free of defects. We wanted to make sure that we were finding the density of the sample and not that of a sample and graphite mixture.

After ensuring that the samples were free of defects, they were placed in a supersonic washer with nanopure water. They were cleaned for five minutes per sample.
2.5 Hardness Testing

The samples were then air dried to make sure there was not water on them. Density measurements were taken using the Archimedes principle. The samples were measured dry, then wet for a total of six times per sample to be able to find a statistical distribution. After each wet measurement, the samples were air dried to remove all water in preparation for the next measurement. Two samples were alternated when they were measured. It was desirable not to collect all of the measurements for one sample at once to increase the randomness of the measurements.

2.5 Hardness Testing

In preparation for hardness testing, the samples were cut in half using a Buehler IsoMet 1000 Precision Saw, and then this half was cut in half again. One of these quarters of the sample was tested for hardness. The stage of a Leco PR-4X hot press mount was cleaned with acetone. The stage was then covered with release agent on the top and bottom. We placed a quarter of one of the samples with the middle face down on the stage so that this part would be showing after the sample was mounted. Figure 2.3 shows how the sample was cut and how it looks after it was mounted. The part that is face up in the mounted sample is the part that gets put facedown on the stage. Next, we lowered the stage a little bit so that the sample was below the bottom plate. We measured approximately 22 mL of red Bakelite powder to mount the sample. With the sample below the plate, we carefully added Bakelite powder with a spatula around the sample so that it would not tip over. We continued adding powder and lowering the stage until we had emptied all the powder. Next we lowered the stage all the way and turned the upper part into position to mount the sample. Then we mounted the sample at a temperature of 300°F, a pressure of 4200 PSI, and a hold time for 7 minutes.
Figure 2.3 This figure shows how the samples are cut and mounted. The sample is cut in half and then cut in quarters as shown by the dashed lines. One quarter is then mounted in red Bakelite powder. The mounted sample is drawn cut in half.

After the sample was mounted, the sample was polished with silicon carbide paper to an 800 grit finish using a Leco Spectrum System 1000 polisher. We started with lower grit paper and moved to higher grits, making sure that the lines from the previous paper were removed each time. Each time we switched to a different paper with a higher grit, we changed the direction we were holding the sample, that is, we had a vertical positioning of the sample for one, then moved to a horizontal positioning. The sample needed to be the same thickness all the way across for the hardness testing. We used a digital caliper to measure the thickness after each paper to make sure we had uniform thickness.

The hardness was found using a Leco LM247 AT Microhardness Tester. This system pushed a pyramidal indenter at 300 grams force for 13 seconds. The computer then uses a microscope to measure the diagonals. The hardness is then found using the equation:

\[ HV = 1854.4 \times \frac{P}{d^2} \]  

where \( HV \) is the Vickers hardness, \( P \) is the downward force in grams force, and \( d \) is the diagonal length of the indentation in \( \mu \text{m} \). Care was taken to ensure that the automatic measurements were correct. The indentations were examined to see that
the right lengths were measured. Figure 2.4 shows an example of the indentation.

Figure 2.4 Indentation for hardness testing. The box around the indentation defines the diagonal lengths.
Chapter 3

Results and Analysis

3.1 Density

Several samples of FCAV were sintered to different maximum temperatures to compare the densities. All of the densities reported are normalized to a fraction of the theoretical density to get a relative density. Figure 3.1 shows the results. As expected, the density increases with increasing maximum temperature. The uncertainty in the measurements decreases with temperature. At lower temperatures, the samples allow more water to enter during the testing. This makes it more difficult to get accurate measurements of the mass in water.
Chapter 3 Results and Analysis

Figure 3.1 Percent theoretical density of samples of FCAY sintered to different maximum temperatures. As expected the density increases with increasing maximum temperature. Error bars are shown for all data points, they are just smaller than the dot size for two of the points.

In order to explore the effects of heating rate on density, several samples of FCA were sintered at different heating rates. The results are shown in figure 3.2. Heating rate does not appear to affect the density. All of the samples had a relative density around 97.5%.

Figure 3.2 Percent theoretical density of samples of FCA sintered at different heating rates. Heating rate does not affect the density. Error bars are shown for all data points, they are just close to the dot size.

The effects of milling time and composition were also explored. Samples of FCA,
3.2 Hardness

FCAY, and FCAYT were prepared. The samples were prepared from powders that had been milled for different amounts of time. Each of the samples was sintered at a 150°C/min. rate. Figure 3.3 shows the results. From this we can see that neither mill time nor composition affects the density.

![Figure 3.3 Percent theoretical density of samples of different compositions milled for different times. Neither mill time nor composition affects the density. Error bars are shown for all data points, they are just within the dot size.](image)

The densities are similar to the data reported by Allahar et al [6] from an experiment very similar to the present work. They found that the relative density did not exceed 97.5%. They suggested that a higher pressure could lead to increased densification.

3.2 Hardness

The effects of heating rate on hardness were explored by testing the same samples sintered at different heating rates that were used for density testing. The results are shown in figure 3.4. The values for copper and stainless steel are included for reference. The hardness values were approximately 200 HV for each of the samples.
The highest uncertainty was 6 HV for these data. Heating rate does not have an effect on the hardness of the sample.

![Diagram showing Vickers hardness for several samples of FCA sintered at different heating rates. Lines for the hardness of copper and stainless steel shown for reference. Error bars are shown for all data points, they are just within the dot size for some points.](image)

**Figure 3.4** Vickers hardness for several samples of FCA sintered at different heating rates. Lines for the hardness of copper and stainless steel shown for reference. Error bars are shown for all data points, they are just within the dot size for some points.

Composition and milling effects were also studied similar to the density section. The results are shown in figure 3.5. Milled samples are harder than samples that are not milled. The milled samples of the same composition have about the same hardness, regardless of the amount of time they were milled. There must be some minimum mill time required to increase the hardness. This work has insufficient data to comment on the minimum time required to increase the hardness. The composition has a large effect on the hardness. The samples containing yttrium oxide ($Y_2O_3$) only and also $Y_2O_3$ and Ti are much harder than the samples without when they are milled. Without milling, all of the samples are similar in hardness. The samples containing $Y_2O_3$ or $Y_2O_3$ and Ti that have been milled contain nanoclusters which increase the hardness as described by Allahar et al [6].
3.3 Activation Energy

The Young and Cutler [4] method was used to find the activation energy. The linear part of the graph was found and a line was fit to this portion. The slope corresponds to the activation energy as described previously. Figure 3.6 shows an example of the graph for finding the grain boundary diffusion activation energy for a sample of FCA which was not milled. The figure is plotted against inverse temperature, so the temperature increases from right to left. The linear section farthest to the right corresponds to the machine heating up. No sintering takes place at these temperatures, so the line is not fit to this portion. The results reported by Young and Cutler show a similar leveling off at higher temperatures. The sample may be in the final stages of sintering at this point. As all of the graphs are very similar, this is the only one that will be shown and only the activation energies will be reported for other samples.
Figure 3.6 Graph for finding the grain boundary diffusion activation energy. The Young and Cutler [4] method is used to find the activation energy. Since the x-axis is inverse temperature, the temperature increases from right to left. The first linear section is when the powder is warming, not sintering, so the linear section at the higher temperature is chosen to find the activation energy. The slope of the graph gives the activation energy.

Samples of FCA were created at different heating rates. The grain boundary diffusion activation energies are shown in the figure 3.7. The volume diffusion activation energies are shown in figure 3.8. The error in these values is very large. This is probably due to the error in the displacement measurements. Since there are only three data points, we cannot comment on the effects of heating rate in this paper.
3.3 Activation Energy

Figure 3.7 Grain boundary diffusion activation energies for FCA at three different heating rates. All of the samples were created from powders that were not milled.

Figure 3.8 Volume diffusion activation energies for FCA at three different heating rates. All of the samples were created from powders that were not milled. The large error probably comes from the error in the displacement measurements.
The effects of milling and composition on the activation energy were also explored. Figure 3.9 shows the grain boundary diffusion activation energy. Figure 3.10 shows the volume diffusion activation energy. All three compositions are plotted on the same graph.

![Graph showing grain boundary diffusion activation energies of samples milled different amounts of time and having different composition.](image)

**Figure 3.9** Grain boundary diffusion activation energies of samples milled different amounts of time and having different composition.
3.3 Activation Energy

The grain boundary diffusion activation energy appears to go down with mill time for FCA and FCAY. The uncertainty is largest for the FCA samples. This could be from a difference in the composition. $Q_B$ does not depend on mill time for FCAYT. $Q_B$ is largest for unmilled powders. Milling has little effect on $Q_V$.

Figure 3.10 Volume diffusion activation energies of samples milled different amounts of time and having different composition.
Chapter 4

Conclusion

4.1 Closing Remarks

ODS steels were studied and activation energies were found. Density and hardness was also explored. The relative density did not exceed about 98%. The hardness was shown to increase with milling time, but was unaffected by heating rate. These results were similar to those found in the literature [6].

Although activation energies were found, the uncertainties in these values were very high. This was due to the imprecision of the measuring technique. The activation energies appear to be independent of heating rate. Milling time affected the activation energies in the FCA and FCAY samples but did not affect the FCAYT samples.

It is the authors hope that this information leads to a greater understanding of SPS. When more is understood about a process, the process can be improved. This research was an attempt to add more to the current understanding of SPS and ODS steels.
4.2 Future Research

The relative densities found for these samples did not reach 100%. This could be due to a low pressure on the powder during sintering [6]. In the future, sintering could be done at higher and lower pressures to compare the effects that pressure has on SPS. All of the samples from this work were sintered at the same pressure. Variations in pressure could lead to interesting results.

Another future research project could be investigating the effects of hold time on the sample. All of the samples in this work were brought to the maximum temperature and then cooled down. Future researchers could hold the sample at maximum temperature for different amounts of time to see if this has any effect on density and hardness.

Future researchers could also redo the experiments to see if the same energies are found. The measurements in the SPS seem to lead to large uncertainties. Sintering in a dilatometer could overcome this issue. A dilatometer is much more sensitive to shrinkage measurements than the SPS. Since the dilatometer sinters differently than SPS, the values could be different, but this would give values with which to compare.
Bibliography


