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Characterization of Nanocrystalline Aluminum Alloy 5083 Powders Produced by Cryogenic Attrition

by Tiffany Ngo

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14. ABSTRACT
Cryomilling, the mechanical attrition of powders with milling balls at cryogenic environment and temperatures, was used to nanostructure AA5083 through grain size refinement. If the current military vehicle protective structures can be replaced with stronger cryomilled nanostructure AA5083, the vehicles will have significantly lowered weight and be highly fuel efficient. Cryomilling was completed for 8-, 16-, and 24-h periods to refine the grain size of AA5083 powder. Scanning electron microscopy revealed the morphology of the agglomerates. X-ray diffraction (XRD) was used to investigate the degree of grain size refinement and the phase purity. Gas fusion analysis, through the Leco system, was utilized in identifying how much oxygen, hydrogen, and nitrogen are in the cryomilled samples. There were no noticeable differences in agglomerate size and shape among the cryomilled powders with varying milling time. The XRD data showed that there was significant grain size refinement without detectable contamination.

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1. Introduction

1.1 Army's Desire for Success

US Army scientists are constantly improving the equipment and supplies used by Soldiers in the field to make their job easier. However, as the Army frequently makes newly updated equipment and weaponry, the armored workforce vehicle gets heavier, which also affects the fuel efficiency, how far the vehicle can go, and how many trips can be made, which influences the budget.

The goal of this cryomilling project is to replace the steel that the vehicles are made of with aluminum. The replacement aluminum material will make the vehicle lighter in weight and will also allow for an increase in the amount of supplies that can be transported in a single load. It can also improve the miles per gallon, which will improve the operating costs since less fuel is being used.

1.2 Literature Review

Aluminum was chosen as the metal to replace steel because aluminum has a lower density (2.7 g/cm^3) compared to steel (7.0–8.0 g/cm^3). To make this metal more functional to replace steel in the vehicle, it is processed through cryomilling, a technique in which the aluminum alloy powder size refinement occurs at cryogenic temperatures. Milling is a process of grinding solid matter under exposure of mechanical forces that trench the structure by overcoming interior bonding forces. The grinding will increase the surface area of the solid, allow for manufacturing a solid with the desired grain size, and enable pulping of resources.

Cryomilling the aluminum alloy 5083 powders is important because the grain size refinement of the powders can be used to make stronger parts, making it more durable to process it for vehicle applications.

1.3 Purpose

The purpose of this experiment is to refine the grain size of the aluminum alloy powder. The desired outcome is to reduce the size of the grains of aluminum alloy 5083 and strengthen parts to support the fabrication of the vehicle and the supply trips by Army personnel. This improvement can lead to cost improvements and gas mileage savings.

1.4 Objective

Cryomilling, the mechanical attrition of powders with milling balls at cryogenic environment and temperatures, was used to nanostructure AA5083 through grain size refinement. If the current military vehicle protective structures could be replaced with stronger cryomilled nanostructure AA5083, the vehicles would have significantly lower weight and be highly fuel efficient.

Cryomilling was completed for 8-, 16-, and 24-h periods to refine the grain size of AA5083 powder. Scanning electron microscopy (SEM) revealed the morphology of the agglomerates. X-ray diffraction (XRD) was used to investigate the degree of grain size refinement and the phase purity. Gas fusion analysis was tested using the Leco ONH 836 system (Leco), which identified how much oxygen, hydrogen, and nitrogen were in the cryomilled samples. There were no noticeable differences in agglomerate size and shape among the cryomilled powders with varying milling time. The XRD data showed that there was significant grain size refinement without detectable contamination.

2. Experiment

2.1 Process

The grain size refinement of the aluminum 5083 powders was completed through cryomilling prior to identifying the chemical impurities through gas fusion analysis with the Leco system. After the chemical analysis, SEM was used to identify the crystal construction of the refined grains through structural analysis, and XRD was used to investigate whether there had been contamination or phase changes in the powders.

By placing the aluminum alloy powders into the attritor machine, stainless steel balls crushed and eventually (through a method similar to fatigue) decreased the powder size (Figs. 1–3). However, when the AA5083 powder was added into the attritor machine, the powder did not immediately break down into smaller particles. The milling initially "pancaked" the powders because the stainless steel balls collided with each other in addition to the powder at 180 rpm. The process went through the continuous cycle of collisions and eventually dispersed smaller particles, resulting in the desired grain size refinement from the broken agglomerates.

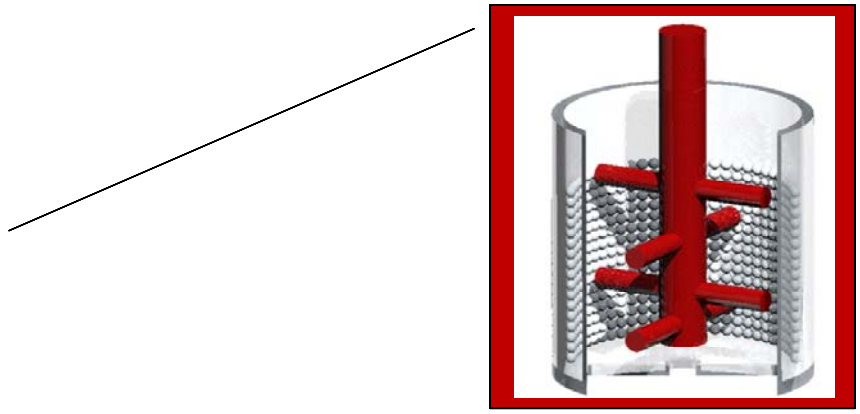

Fig. 1 Milling attritor machine

Fig. 2 Inside process of cryomilling

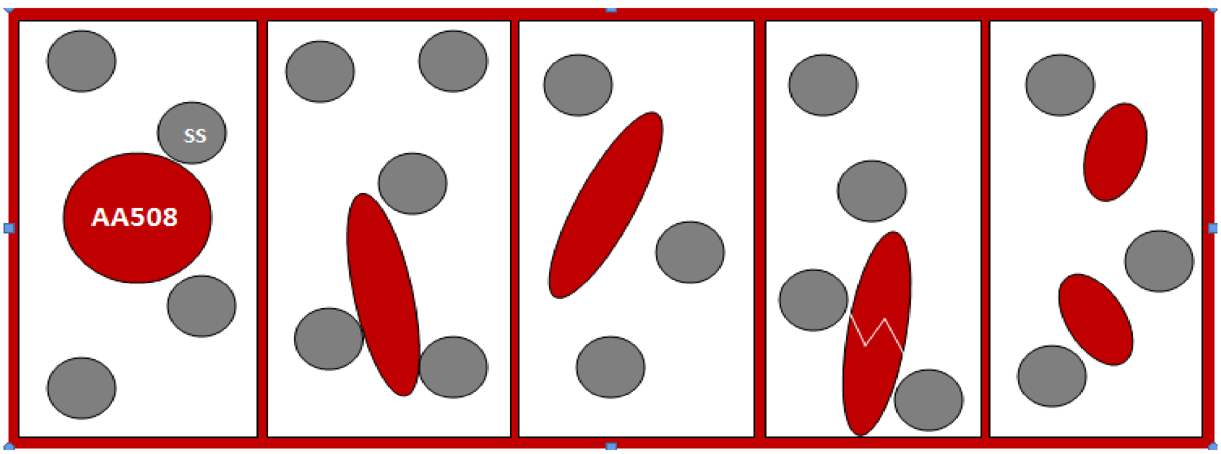

Fig. 3 Basic process of cryomilling

For the milled powders to reach cryogenic temperature, liquid nitrogen was used. The purpose of using liquid nitrogen was to form a protective layer over the cryomilling process and minimize the oxidation.

After the milling process, the powders were placed in the Leco system. In the system, a sample of the aluminum powder was placed into a graphite crucible. The sample in the crucible was then heated to a high enough temperature to melt the sample. As the sample melted, the hydrogen, oxygen, and nitrogen content were analyzed. Then, a powder sample was placed into the SEM. The SEM bombarded the sample with electrons, and an image was created based on how the electrons are "bounced off" or ejected from the sample. There were 2 types of electrons used to make the image: secondary electrons (SEs) and backscattered electrons (BSEs). The difference between the 2 was that SEs were related to surface topography and BSEs were related to chemical content. The SEM identified any visible morphological changes between the milled powders over different time periods.

XRD was used to assure that grain size refinement was successful. The X-rays were scattered by regularly spaced atoms of a crystal, which allowed information about the structure of the crystal to be obtained. In other words, the XRD was used to identify the grain size refinement and if there were phase changes or if there had been contamination of the powders.

2.2 Purpose

The aluminum alloy powders were cryomilled for grain size refinement because as the grains of the aluminum decreased to nanosize, it strengthened the parts.

Analyzing the nitrogen, oxygen, and hydrogen content using the Leco system was critical because a higher nitrogen content in the aluminum alloy powders resulted in increased strength. However, the more hydrogen content, the lower the strength, so it was important to minimize the amount of hydrogen. The oxygen content was also documented since it was unknown whether the influence on the powders would be positive or negative.

Analyzing the structure of the grains through XRD was important to assure that the phases from the sample were the expected ones. Analyzing the structure of the grains through SEM was important to determine if there was grain refinement.

3. Data and Results

3.1 Cryomilling Data

Table 1 shows the samples that were analyzed. Under the Sample ID category, the number in front of the title represents the number of hours the sample ran. The CM stands for cryomilling, and the number after the dash represents the batch number. Al5083 is the baseline sample. The 8-h sample is a combination of two 8-h batches.

Table 1 Cryomilling samples

Sample	Sample ID	Milling Time	Notes
Al 5083	Al 5083	Not Milled	As-astomized powder
CM1314-0	8CM	8 h	As-milled powder result of two 8-h mill runs blended together
CM17	16CM-1	16 h	As-milled powder
CM18	16CM-2	16 h	As-milled powder
CM15	24CM-1	24 h	As-milled powder
CM19	24CM-2	24 h	As-milled powder
CM16	24CM-3	24 h	As-milled powder

3.2 Leco Results

In the nitrogen graph (Fig. 4) it is noted that the nitrogen concentration increases with milling time. In the oxygen and hydrogen graphs, it is determined that the values level off and plateau, except for the 16-h cryomilled batch 2. However, it is believed that the point is an outlier because the attritor machine temporarily dried out during that cryomilling process, which could explain the high levels of hydrogen and oxygen.

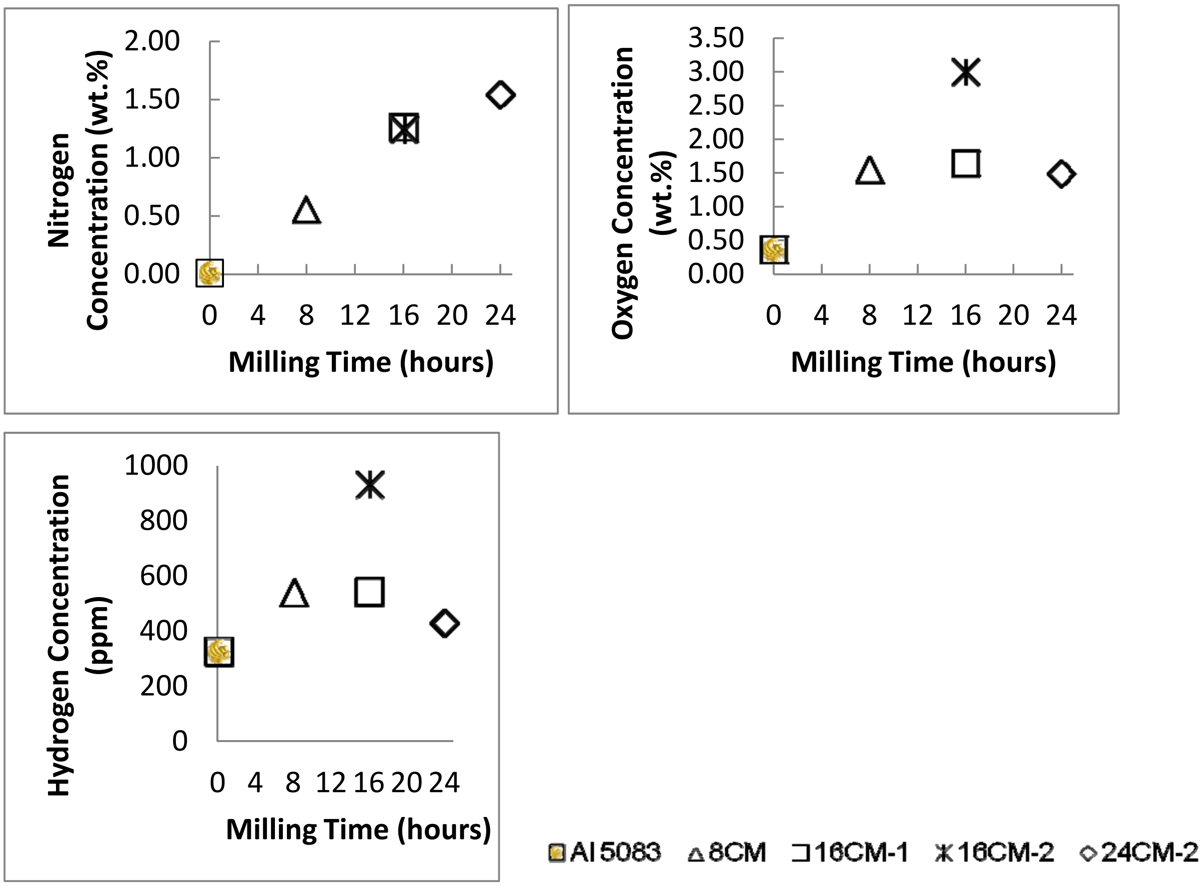

Fig. 4 Leco graph

3.3 SEM Data

The SEM photo in Fig. 5 is the as-received powders. These are 20–30 µm, very round particles that have not been processed in the cryomilling machine. After 8 h of cryomilling (Fig. 6), there is obvious morphological change compared to the as-received powders. It is visible that it is "pancaked," no longer round, and crushed.

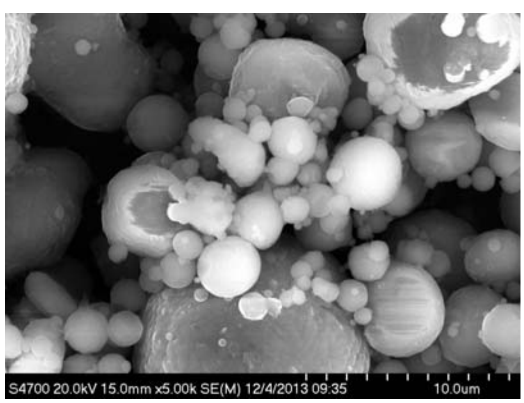

Fig. 5 As-received powders (AA5083)

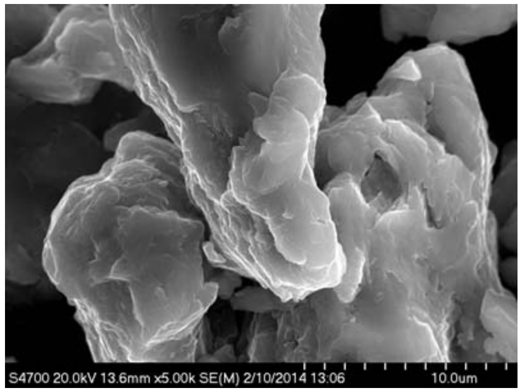

Fig. 6 An 8-h cryomilling run

Figure 7 shows the 16-h cryomilled powder and Fig. 8 is the 24-h cryomilled powder. There is no visible morphological change compared to the 8-h run on both of them. It is inferred that there is no visible grain refinement between the 8- and 24-h cryomilled run.

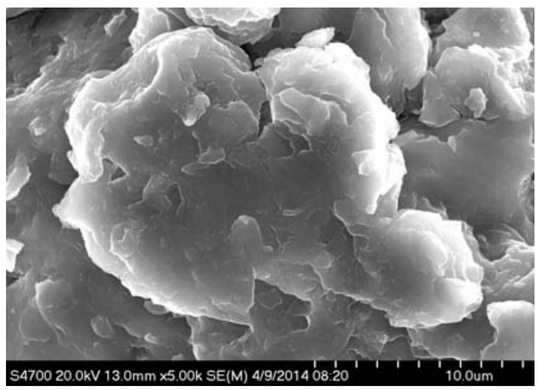

Fig. 7 A 16-h cryomilling run

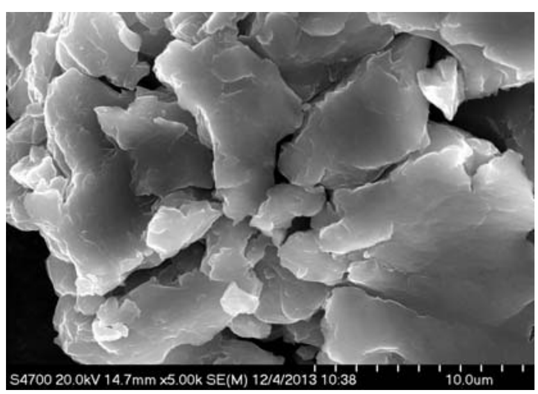

Fig. 8 A 24-h cryomilling run

3.4 XRD Results

From the as-received powders (bottom spectrum) of the XRD results (Fig. 9), the other spectrums visibly overlap above the peaks very nicely. From these results, it is found that there are no additional peaks, and, therefore, no phase changes or contamination of the powders during cryomilling.

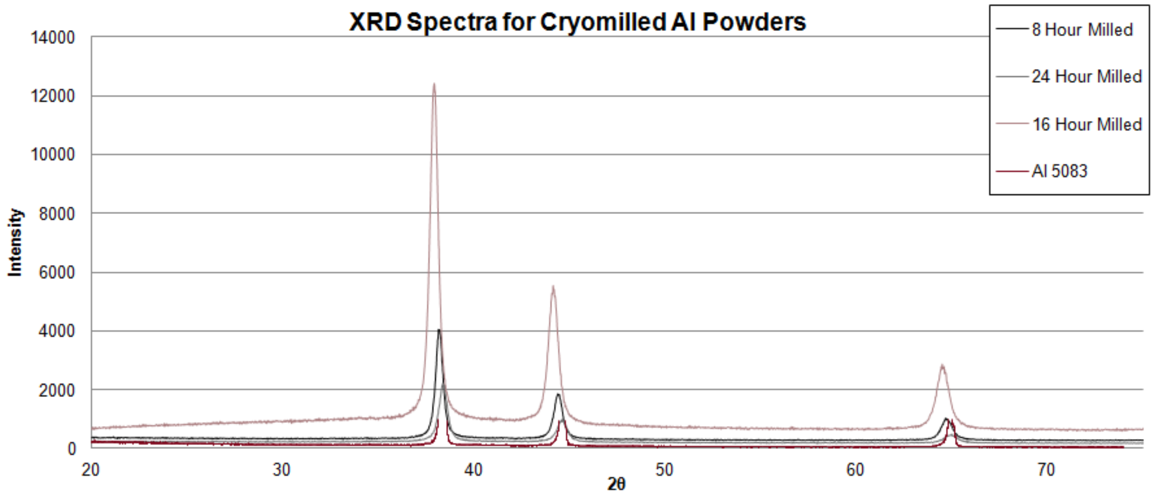

Fig. 9 Cryomilling graph

3.5 Grain Size via XRD

After 8 h of cryomilling, the crystallite size of the powders is nearly 21 nm (Table 2).

Table 2 Sample size under XRD

Sample ID	Size (nm)
8CM	21
16CM1	18
16CM-2	18
24CM-1	13
24CM-2	14
24CM-3	17

After 16 h of cryomilling, the size is nearly 18 nm. After 24 h, the size ranges between 13 and 17 nm. From this grain size identification, this data is significant different from the as-received powders crystallite size, which is over 100 nm (identifying the exact size is complicated because the machine that was used to identify the grain size is only capable of reaching 100 nm and the as-received powders exceeded that count).

4. Summary

The results from the Leco system revealed that additional cryomilling time introduced more oxygen, nitrogen, and hydrogen. Nitrogen was introduced during the cryomilling process using a bath of liquid nitrogen to increase the content with milling time. Oxygen was introduced but the metal that was used, aluminum, will always oxidize. Liquid nitrogen was included in the cryomilling process to form a protective layer over the process and minimize the amount of oxidation as much as possible. Hydrogen was introduced with the knowledge that the powders started off with a surrounding layer of hydrogen and oxygen. As the balls continuously collided during milling, they slowly broke off pieces of the hardened layer. However, during the 16-h cryomilled batch 2, the attritor machine temporarily dried out, which was attributed to the liquid nitrogen also drying out. Thus, exposing the powder to oxygen and hydrogen in the form of moisture led to an increase in the percentage of hydrogen and oxygen.

The results from the SEM revealed that there had been initial morphological changes. However, there were no further visible morphological changes during the 8–24 h cryomilled run. Although there had been no visible grain size refinement in the SEM, it was verified that there had been refinement through the XRD. The results from the XRD revealed that there were also no phase changes or contamination of the powders.

5. Conclusions

This project was initiated to exploit the many new technology advances for equipment and supplies to support Army personnel. The effects on armored vehicle weight, fuel efficiency, vehicle travel distance, number of trips, and budget were considered.

From the Leco data, it was identified that nitrogen content increased with milling time, while oxygen and hydrogen content did not. From the SEM identification of milled powders, it was determined that the agglomerate size and shape were largely unchanged with milling time at 8, 16, and 24 h. From the SEM observations, there was not a lot of discrepancy between the batches at 8-, 16-, and 24-h cryomilled runs. While it was verified through XRD that there had been grain size refinement, the morphological changes were not visible in SEM. From XRD, it was also verified that there were no phase changes and no contamination to the powders.

From this data it had been determined that aluminum alloy 5083 powders could undergo grain refinement and develop enough strength to make parts for replacement of the metal in the vehicle. Since aluminum is lighter than steel and has a lower density compared to steel, this would lower the weight and improve fuel efficiency.

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