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Synchrotron X-rays Monitoring Nano-Aluminum Grain Growth of a Metal Matrix Composite under Thermo-mechanical Conditions

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The experimental *in situ* synchrotron study presented here examines the micro structural features of tri-modal Aluminum Metal Matrix Composites (MMCs) that influence their mechanical properties. The thermo-mechanical environment under which these MMCs, consisting of coarse-grained and nanocrystalline aluminum as well as boron carbide reinforcement particles, are manufactured is captured through *in situ* high-energy X-ray characterization. Results of peak identification indicate the presence of dispersoids, Al_4C_3, $FeAl_6$, and AlN, formed after cryomilling and analysis of the peaks provide quantitative volume fractions of these compounds in the range of 2.6%, 1.9% and 1.2% respectively. The full width half maximum (FWHM) values of the X-ray diffraction data, collected over time from the sample, were analyzed to establish the change in nano-Aluminum grain size as a function of temperature and applied compressive load. Results monitor the rate of growth in nano-aluminum grain size with an overall increase of 101nm under a compressive stress of 50 MPa with a temperature ramp of 5°C/min for 25-315°C. The results will enable mechanical properties of these MMCs to be correlated with quantitative values of dispersoids to maximize their high strength potential. In addition, the findings on dispersoid content and rate of grain growth provide significant information on processing parameters towards optimizing the manufacturing of these materials.

Nomenclature

K	Constant of 0.9 for spherical particles
λ	Wavelength (nanometers)
θ, θ_0	Diffraction Angle (radians)
L	Grain Size (nanometers)
$FWHM$	Full Width Half Maximum
$\delta 2\theta$	True FWHM (radians)
B_{ins}	Instrumental FWHM
B_{exp}	Measured FWHM
β	True FWHM
r	Radius (mm)
z	Sample-to-detector distance (mm)

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I. Introduction

TRI-MODAL Aluminum Metal Matrix Composites (MMCs) have exhibited exceptional properties such as high strength, low density and good corrosion resistance. These properties make MMCs attractive candidates for replacement and addition to conventional alloys used in the aerospace industry with applications in aeropropulsion, missiles, and electronic packaging in aircraft[1,2]. Current applications of aluminum-based MMCs with SiC particulates include the manufacturing of the ventral fin of an F-16 fighter plane as well as the rotor blade sleeves on the Eurocoptor EC120 and N4 helicopters[2]. The combination of nanocrystalline aluminum and coarse grain aluminum particles with ceramic reinforcement particles, investigated in this work, contributes to the high strength and ductility of this trimodal material[3–7]. The typical manufacturing process for these MMCs, shown schematically in Fig. 1, involves cryomilling and extrusion, forging, and rolling during which varying thermo-mechanical loads are applied to the material.

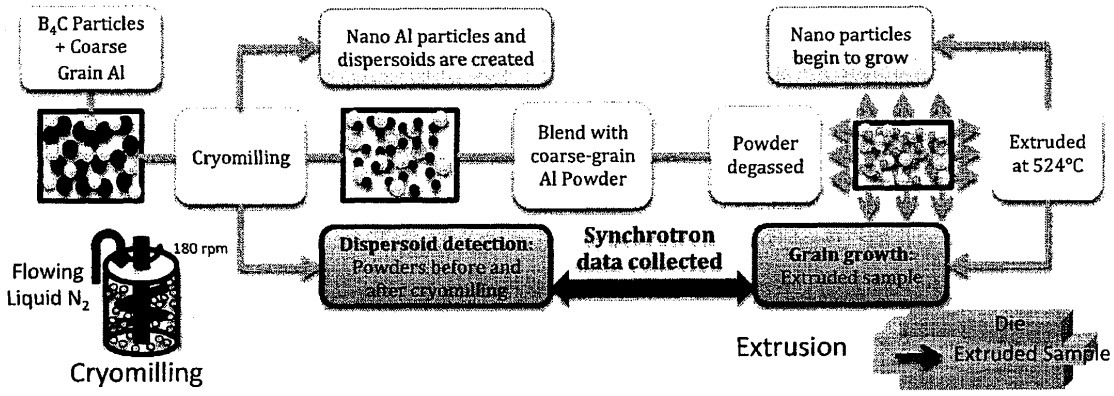

Figure 1. Manufacturing Process. Schematic of the manufacturing process used and the factors influencing the mechanical properties, dispersoid formation and grain growth within this process studied using synchrotron X-ray diffraction.

The initial step in the MMC manufacturing process, as shown in Fig. 1, involves cryomilling, where powders are milled in a cryogenic media, in this case liquid nitrogen[8]. The cryomilling of coarse grain Al with B_4C particles results in nanocrystalline Al and B_4C agglomerates as well as various dispersoids[6], such as Al_4C_3, $FeAl_6$, and AlN. The AlN, formed from the nitrogen medium used for the cryomilling process, is of particular interest and believed to influence the strength of the material through reinforcement, grain pinning, and thermal stability[4,6]. The volume fraction of these second-phase particulates is said to be the most influential parameter related to their positive influence on the properties of the material, as the concentration of the dispersoids within the grain boundaries contributes to the particle reinforcement and grain growth inhibition, helping maintain the materials high strength[7,9]. However, the strength of the material has been seen to decrease as the volume fraction of the dispersoids exceeded 1.5%[7]. This suggests the volume fraction of the additional compounds should be monitored when processing in order to avoid a critical concentration that would reduce the strength of the composite material[10,11]. Microscopy is generally used for determining the volume fraction and presence of the dispersoids in question, with some promising results. However, due to the low volume content, the technique proves to be difficult, time consuming, and is often believed to under-estimate the actual amount[4,6,7,9]. In many cases, the presence of dispersoids is difficult to determine and thus considered negligible to the focus of the research. Other studies are only able to determine the correlation of dispersoid concentration and strength from ex-situ data taken from samples of different cryomilling times based on the assessment that dispersoid concentration will increase with a longer cryomilling time. Synchrotron X-ray diffraction provides, through peak identification and relative intensity analysis, a more accurate material detection and volume fraction estimation to help better monitor the amount of dispersoid formation, enabling their correlation to bulk material strength in future.

In addition to the effects of dispersoid formation, the thermo-mechanical applications that accompany the manufacturing of bulk MMCs, albeit necessary, are believed to contribute to the increasing grain size of the nanocrystalline particles, thus limiting the potential strength of the bulk material[3,4,6]. Studies to ascertain the grain growth typically utilize standard microscopy techniques and low energy X-ray diffractometers for ex-situ experiments to determine

grain size and growth. Lavernia et. al. has shown that XRD analysis has the advantage of being able to provide grain size readings over a large volume, eliminating the need for thin films that risk not representing the true microstructure [12–14]. Synchrotron X-ray diffraction has proven to be a promising technique for measuring the grain growth of nanocrystalline particles under various stresses [6,14–21]. High-energy XRD offers a much higher angular resolution, allowing for easier detection of grain growth via *in situ* experiments, faster data collection, and ability to use bulk samples for testing [12,15,22–24]. The Williamson-Hall method, commonly used for the determination of small grain sizes, allows for accurate results that incorporate the effects of instrumental broadening, and strain broadening of the peaks along with the grain size [25].

In the synchrotron studies presented here, the microstructural features including dispersoid formation and grain growth that influence the mechanical properties of a tri-modal MMC were examined within the manufacturing process as highlighted in Fig 1. Through phase identification from synchrotron measurements of powders prior to and after cryomilling, the volume fractions of the various dispersoids and impurities resulting from the cryomilling process were determined quantitatively. The analysis of the synchrotron peak widths collected, allowed for the *in situ* grain growth behavior of the nanocrystalline aluminum to be observed as a function of temperature and external mechanical loading, representing the effects of the extrusion process.

II. Experimental Procedure

As part of the manufacturing process which involves cryomilling, boron carbide (B_4C) particles were introduced into an Aluminum 5083 matrix. This synthesis method results in the existence of nano and coarse grain Al 5083 and B_4C particles within the samples. After manufacturing the bulk material, the sample was cut into a rectangular specimen of approximately 5 mm X 5 mm X 10 mm. The X-ray diffraction experiments performed were conducted at the Advanced Photon Source, Sector 1-ID at the Argonne National Laboratory. The experimental method for thermo-mechanical testing, including the parameters for the data collection, was established based procedures and methods developed in conducting similar *in situ* synchrotron thermo-mechanical characterization experiments in previous work [26]. The beam dimensions used were 200 μm X 200 μm with a beam energy of 70 keV which corresponds to a wavelength of 0.1771 $\AA$. The experimental setup is illustrated in the schematic of Fig. 2. The X-ray beam was operated in transmission mode and collected by a GE detector as diffraction rings. The MTS electromechanical testing system that was mounted on an XYZ stage for mobility provided the mechanical loading and the coupled thermal loading was provided with an infra-red furnace around the sample.

A. Experimental study of dispersoid formation

As reference and dispersoid-containing samples, two powders were tested including an aluminum alloy 5083 powder to represent the material before the cryomilling process and Al 5083 with B_4C after a 24 hour cryomilling process respectively. The powders were analyzed by placing a small amount of the material in the the line of the transmitted X-ray beam. The X-ray diffraction data was collected and compared for the two samples to ascertain the formation of dispersoids and their volume fraction in this ex-situ approach.

B. *In situ* experimental study of grain growth

The MMC specimen shown in Fig. 2a was tested under the thermo-mechanical condition with an external load corresponding to a uniaxial compressive stress of 50 MPa and a ramp temperature from 25 - 315°C as shown in Fig. 2d. Once a specimen was placed on the MTS electromechanical testing system and leveled to be perpendicular to the incident beam, reference measurements were taken for both conditions: unloaded at room temperature and loaded at room temperature. While mechanically loaded, the furnace was programmed for a temperature ramp of 5°C/min and measurements of the thermo-mechanical conditions were recorded. The specimen setup is shown in Fig. 2c. Data was collected over time via a scan of 7 points across the face of the specimen with each point the size of the X-ray beam as illustrated in Fig. 2b. The collected data provides the *in situ* monitoring of grain growth under thermo-mechanical conditions.

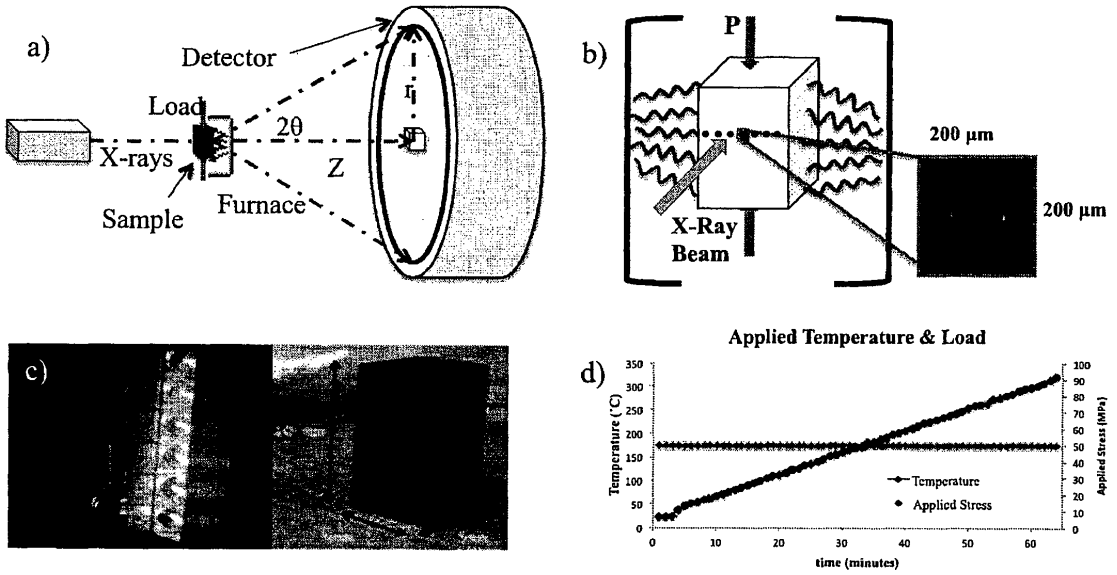

Figure 2. Experimental setup and schematic of sample to detector geometry for grain size experiment. a) sample to detector geometry used later for instrumental broadening corrections, b) bulk sample under thermo-mechanical loading while X-ray data is taken across the face, c) sample set in MTS machine and dimensions, d) thermo-mechanical loading, constant 50 MPa compressive load with increasing temperature load.

III. Experimental Results

A. Detection of Dispersoids

The X-ray diffraction data taken was analyzed to present results for assessment of the dispersoids including X-ray diffraction plots for phase identification. The plots presented below represent the peaks of the present materials in terms of intensity and d-spacing. These plots were used for phase identification. Phase identification of the samples was essential to identify the constituents and determine the volume percentage of the dispersoids that might alter the material properties of the MMCs.

A qualitative comparison of the peaks are present in Fig. 3. The intensities of the peaks for a specific material can provide an estimated volume percentage of the compounds present within the material.. The volume fractions were calculated relative to the peak intensities as shown in Table 1.

Table 1. Peak Identification and Volume Fraction

Material	Peak	Volume Fraction %
Al	(111)	90.315
B_4C	(104)	4.014
Al_4C_3	(107)	2.637
$FeAl_6$	(222)	1.867
AlN	(100)	1.167

B. Nano-Aluminum Grain Growth Trend under Simulated Manufacturing Conditions

In general it is expected that the size of the nano aluminum grains will grow as a function of thermo-mechanical loading. The grain size is related to the 2D diffraction patterns, which can show the trend of the grain size through the Full Width Half Maximum (FWHM) and intensity of the aluminum peaks. As the grains grow, the FWHM of the peaks is expected to decrease, creating an appearance of a thinner and taller peak. This is illustrated in the results obtained as shown in Fig. 4, where the diffraction patterns vary over an increase in temperature and time.

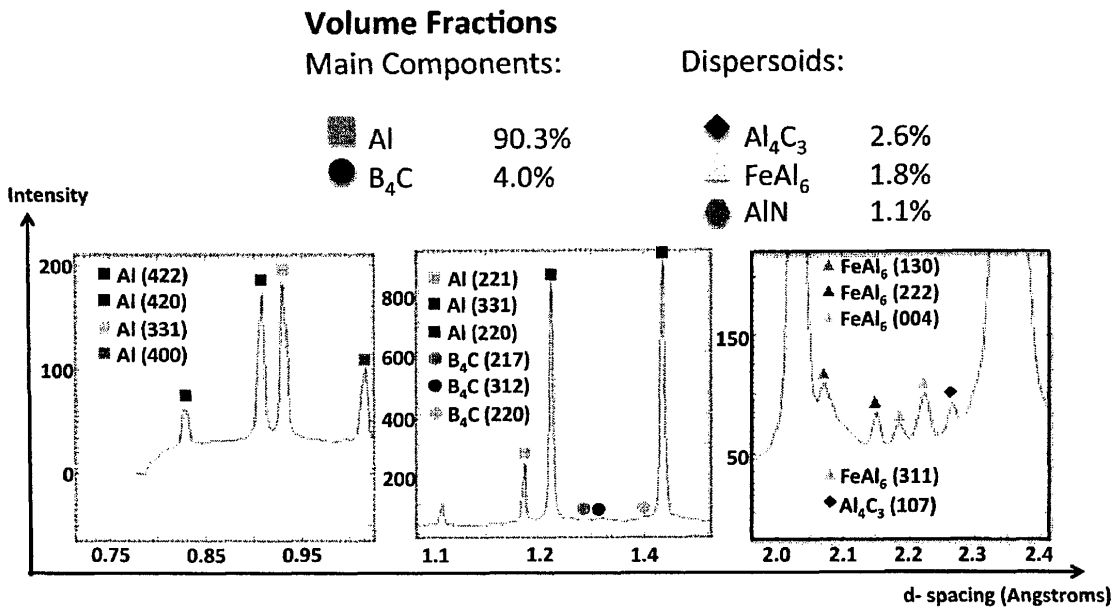

Figure 3. Peak identification of major components and dispersoids. Dispersoids were identified from these results and quantitative volume fraction values determined from relative intensities.

The X-ray diffraction data provides a relationship between the FWHM of the peaks and the grain size using a form of the Scherrer equation. The Williamson-Hall method will allow us to obtain an accurate estimate of the grain size as well as accommodate for the effect of strain on the nano-Al particles[17]. As shown in Eq. (1), there is an indirect relationship between the FWHM and the grain size[3,18,19,27–29].

$$\frac{(\delta 2\theta)^2}{tan^2\theta_0} = \frac{K\lambda}{L}\left(\frac{\delta 2\theta}{tan\theta_0 sin\theta_0}\right) + 16e^2 \tag{1}$$

The true FWHM in radians, $\delta 2\theta$, and the peak maximum, θ_0, are used in relation to the specified wavelength, λ, the Scherrer constant, K, and the grain size, L, in nanometers. However, in order to use Eq. (1), the instrumental FWHM broadening was removed from the experimental FWHM broadening using Eq. (2) and the results from the 24-hour cryomill powder. The FWHM after the instrumental correction can then be related to the strain and grain growth effects on the FWHM broadening.

$$\beta = B_{\text{exp}} - \frac{B^2{}_{ins}}{B_{\text{exp}}} \tag{2}$$

The FWHM value, initially in pixels, was converted to radians using the diffraction angle (2θ), the radius (r), and the sample-to-detector distance (z) as shown in Fig. 2. The Eqs. (3-5) below illustrate the calculation process using trigonometry,

$$\tan(2\theta) = \frac{r}{z} \tag{3}$$

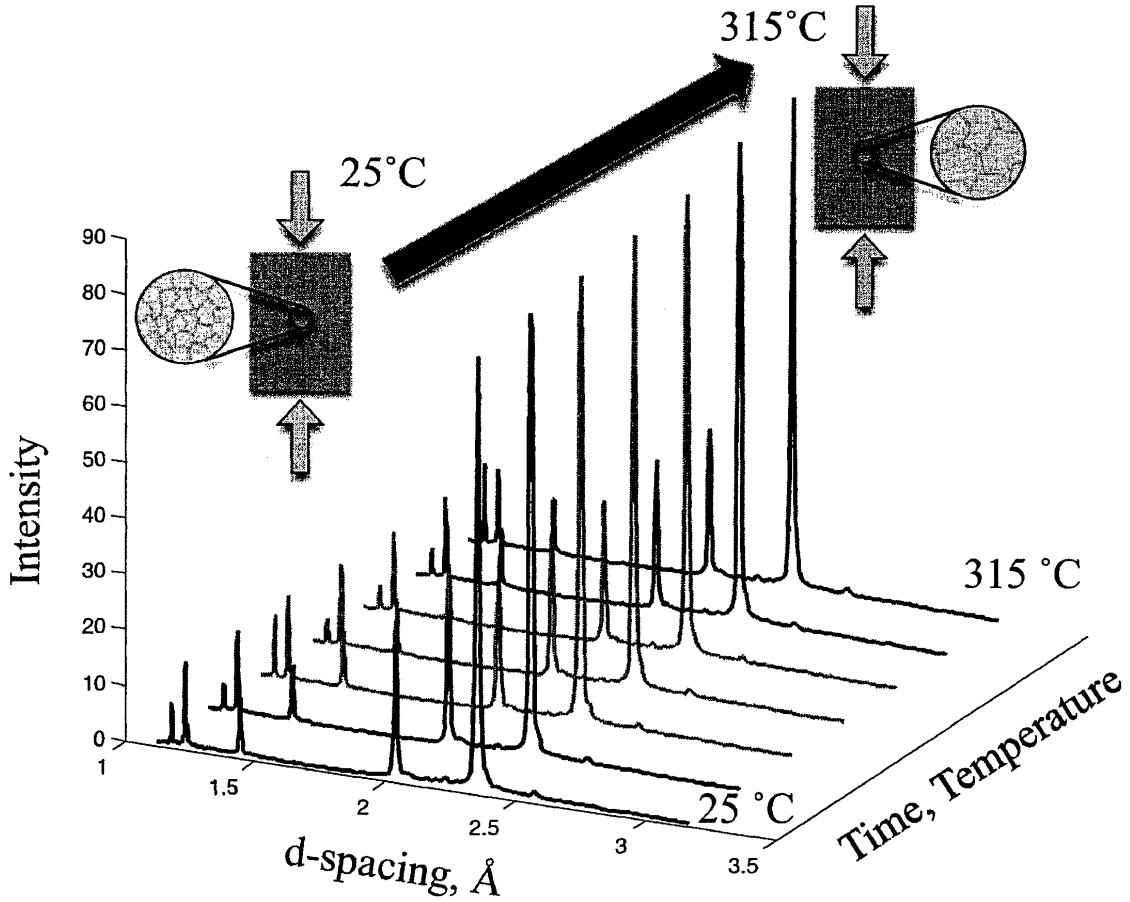

Figure 4. X-ray diffraction plots over temperature and time. Plot indicates an increasing trend in grain size as the sample is thermo-mechanically loaded with decreasing FWHM accompanied by increasing peak intensity.

Taking the derivative,

$$\sec^2(2\theta)d(2\theta) = \frac{dr}{z} \tag{4}$$

Eq. (5) was solved for $d(2\theta)$,

$$d(2\theta) = \frac{dr}{z}\cos^2(2\theta) \tag{5}$$

This value of $d(2\theta)$, which equals the FWHM (radians), was substituted into Eq. (1) to determine the grain size L in nanometers at given time intervals.

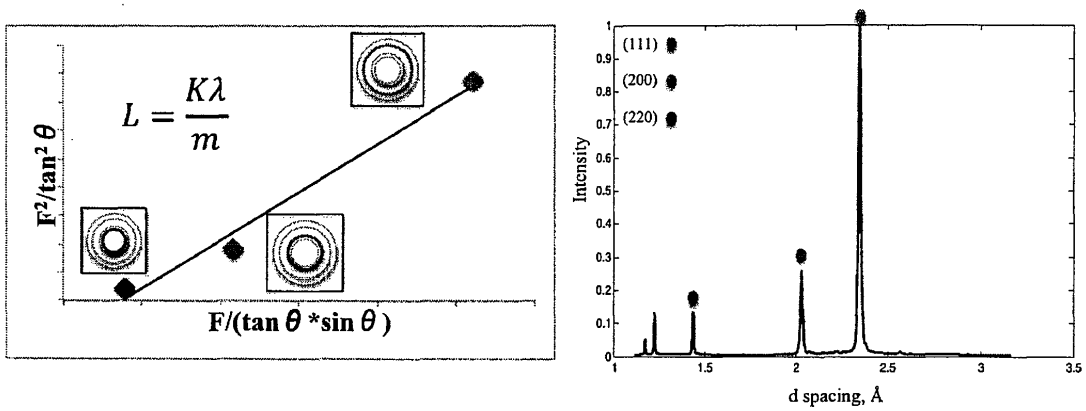

Figure 5. Relation between Aluminum peaks and slope calculation from X and Y axis values.

The synchrotron x-ray diffraction data was analyzed by incorporating the above theory in algorithms developed for the analysis of 2D diffraction data in previous work[26]. The instrumental broadening was determined for each diffraction angle of interest using the 24-hour cryomill powder as the baseline. This instrumental effect was removed from the corresponding data for each diffraction angle analyzed and the nano Aluminum grain growth as a function of temperature and strain for the sample was determined. The Williamson-Hall method was used by calculating the slope of the linear trend plotted using the three main Aluminum peaks of crystallographic planes (111), (200) and (220). The crystallographic planes were chosen based on their prominent nature, promising the most accurate measurements for the FWHM. This relationship to the diffraction rings and 2D diffraction pattern can be seen in Fig. 5. With this analysis, the initial nano Aluminum grain size was determined to be approximately 93 nm and as the thermo-mechanical test (50 MPa with a temperature ramp of 5°C/min for 25-400°C) concluded, the grain size was determined to be approximately 194 nm as shown in Fig. 6. These results follow the expected relationship where FWHM decreases (thus grain size increases) with loading. Similar studies can provide such results for various combinations of thermal and mechanical conditions to establish optimized manufacturing conditions for enhanced mechanical properties.

IV. Discussion

Through the use of high-energy synchrotron radiation, high-resolution *in situ* nano-Al grain size and dispersoid volume fraction quantification was performed on a metal matrix composite undergoing a coupled thermal and compressive mechanical load.

It is believed that the formation of certain dispersoids, particularly AlN, during the cryomilling process helps limit grain growth and contribute to the strength of the material. The volume fraction of these dispersoids is also known to

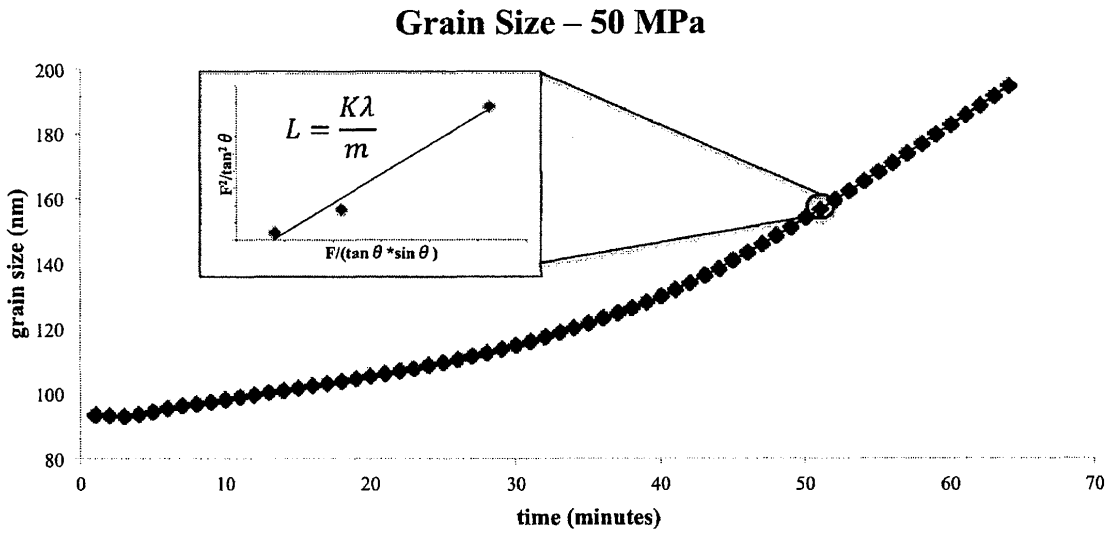

Figure 6. Grain growth trend. Plot of grain size with respect to time under corresponding mechanical and thermal load.

be of importance, however is not frequently calculated using accurate techniques. Despite several studies claiming the importance of dispersoid concentrations, experimental calculations of these parameters have proven difficult. A study by Schoenung et. al. further investigated the nitride concentration within trimodal MMCs, and found that the material had concentrations of about 0.84 wt.% of nitrogen after a 24 hour cryomilling[30]. One study used high resolution transmission electron microscopy (HRTEM) and secondary ion mass spectrometry (SIMS) to detect the presence of specifically nitrogen containing dispersoids. Although these methods were able to detect relative nitrogen content, for the low volume fraction of dispersoids to be detected in this material, the methods proved to be time consuming and difficult[6,9,31]. Although quantitative determination of the volume fraction is not easily obtained, this information is critical to the material and mechanical properties. The development of magnesium and copper nanocomposites was analyzed to determine their effect on material properties and it was demonstrated that an increase in volume fraction of reinforcement nanoparticles improved the strength and thermal stability of the material[32–34]. However, in aluminum MMC research, it has been suggested that the volume fraction of nano-reinforcement addition is of importance, and that if this volume fraction exceeds 1.5% in value, the strength and ductility of the material decreased[7].

Several studies have used X-ray diffraction to analyze MMCs, however these studies are normally focused on the grain size of the nano-Al particles, not the dispersoid concentrations. With the use of high-energy X-ray diffraction as shown in this work, the presence and volume fraction of various dispersoids and contaminants can be accurately determined to help observe the effects dispersoids have on the mechanical properties of metal matrix composites. The presence and volume fraction of dispersoids such as Al_4C3, $FeAl_6$, and AlN were found and compared to previous studies. The concentration levels for the three compounds are seen to be in the range of results obtained through other methods[6,9,31]. In this case, the combined volume fractions of dispersoids were seen to be approximately 5-6%. With the improved methods presented here for volume fraction determination of these dispersoids, a complete study can now be performed to quantitatively establish their effects on the strength.

The research done by Waly et. al. on the microstructural evolution of a magnesium alloy under thermomechanical processes showed the presence of grain growth after varying extrusion loads and the correlation of these loads with the material strength. This shows the negative effect grain growth has on the material properties[30]. Similar research on other materials as well as MMCs has shown a decrease in strength with an increase in nanocrystalline particles due to thermo-mechanical applications[6,16,18,35,36]. Several studies focusing on nano and multiscale materials have showed the increased benefit of grain growth data acquired via high-energy synchrotron radiation[24,33,37].

By utilizing high-energy synchrotron radiation and an electromechanical testing system on a stage coupled with a furnace, high resolution XRD data, real time grain growth from thermo-mechanical loading was obtained in this study. The grain growth of the nanocrystalline Al particles was calculated using a form of the Williamson-Hall method, based

on the inverse relationship between the FWHM of the Al peaks and the grain size of the particles [13,38]. The Williamson-Hall equation is able to relate the grain size and the microstrain to the FWHM of X-ray diffraction peaks, this ability to detect the individual effects of the microstructure on the FWHM can later be used to observe the microstrain as well as the grain size behavior of nanoscale particles under thermomechanical loads [17,39]. The initial grain size was calculated as 93 nm and grew to 194 nm. Two things should be noted from these results: i) Since the sample was subjected to a manufacturing process prior to testing, it already has experienced grain growth from the extrusion to form the sample, which is verified by the initial grain size of 93 nm whereas the grain size before extrusion is approximately 50 nm. In addition, other research has calculated the grain size after manufacturing to be about 100 nm through microscopy measurements, verifying our initial results [6]; ii) As the sample undergoes thermo-mechanical loading, the grains steadily grow, doubling in size at a temperature a little more than half that of the extrusion temperature. The determination of the rate of grain growth was achieivable through the *in situ* capability developed in these tests. These results have verified recent studies while also showing the continued growth of the nanocrystalline particles of post-manufacturing samples [9].

V. Conclusion

The goal of this synchrotron study was to examine the microstructural features that influence the mechanical properties of tri-modal Aluminum Metal Matrix Composites. This study introduced the experiments performed at the Argonne National Laboratory, described the correction for instrumental broadening, and presented the initial analysis of the presence of dispersoids and their concentration and the nano Aluminum grain size as a function of temperature and strain for a sample undergoing a constant mechanical load and a temperature ramp. Material identification conveyed the formation of dispersoids during the cryomilling portion of the manufacturing process. The presence of such dispersoids helps improve the strength of the material as well as maintain nanocrystalline size particles, reducing grain growth from thermomechanical loads. At the same time, exceeding a critical amount could be detrimental to the mechanical properties. The results presented here show the capability to relate quantitative values to mechanical strength.

Peak broadening results indicate a decrease in FWHM, thus a growth in nano Aluminum grain size, as a function of thermo-mechanical load. Therefore, it is seen that the effects of mechanical loading on the FWHM at temperature have a greater impact than initially suspected. Future work will be directed toward obtaining trends for samples under different thermo-mechanical conditions and determining appropriate loading parameters to meet the grain size requirements that will ultimately effect the strength of the bulk material.

By utilizing high-energy synchrotron radiation *in situ* measurements provide high resolution results for dispersoid concentration determination. In addition, the grain growth behavior of nano Al particles can be monitored to better determine the effects various manufacturing processes can have on the material properties of tri-modal Aluminum Metal Matrix Composites.

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