PHYS309 Project

Crystall Grain Size and Strain Analysis of Mechanically–Milled MgB_2 by P-XRD

Ibraheem F. Al-Yousef, Moayad M. Ekhwan, Umar T. Al-Huwaymel

Department of Physics, KFUPM

April 18, 2022

Abstract

XRD experiments provide invaluable diffraction spectra from which crystallographic information may be obtained. Powder-XRD is used to study the effect of ball-milling on the crystalline grain size and strain of MgB_2. The grinded sample is ball–milled for 2 hrs at 40 rpm in 3 min intervals that are spaced by 1 min cooldown and compared to the non–milled sample.

1 Introduction

X–Ray Diffraction (XRD) experiments are often done to obtain crystallographic information. In this project, Powder–XRD or PXRD is used to study the effect of mechanical grinding, specifically ball–milling, on the crystalline grain size and strain of MgB_2, a superconductive crystalline material. Smaller grain size results in broader peaks. Moreover, the broadening effect has a contribution from the strain experienced by the crystalline grains. The total broadening β_T is defined as the sum of both contributions. It is the measured in the Full–Width at Half Maximum (FWHM) corresponding to the broadening of the peak, in radians.

$$\beta_T = \beta_D + \beta_\epsilon \tag{1}$$

where β_D is the broadening due to crystalline size, D, and β_ϵ is the broadening due to strain, ϵ. Scherer equation implies:

$$D = \frac{K\lambda}{\beta_D \cos\theta} \leftrightarrow \beta_D = \frac{K\lambda}{D\cos\theta} \tag{2}$$

where λ the XRD source wavelength and $K = 0.9$ is the shape factor. Meanwhile the strain broadening is given by:

$$\beta_\epsilon = 4\epsilon \tan\theta \tag{3}$$

combining both equations:

$$\beta_T = \frac{K\lambda}{D\cos\theta} + 4\epsilon\tan\theta \tag{4}$$

multiplying by $\cos\theta$:

$$\beta_T \cos\theta = \epsilon(4\sin\theta) + \frac{K\lambda}{D} \leftrightarrow y = mx + b \tag{5}$$

Least square fitting of this form can be used to determine $m = \epsilon$ and $b = K\lambda/D \leftrightarrow D = K\lambda/b$. Those can be used to determine the three broadening factors as well, using equations (1), (2), and (3), which can be compared to the average from measured data.

2 Experimental

The grinding procedure was to ball–mill for 2 hrs at 40 rpm in 3 min intervals that are spaced by 1 min cooldown. The PXRD operated with $\lambda = 0.154184$ nm, with starting angle of 5.00093 degrees, scanning 6926 up to 40.0085562 over around 25 minutes. For the PXRD data analysis, the python module `xrdfit` was mainly used to apply Gaussian–Lorentzian fitting to the peaks, from which the FWHM and accurate peak positions are determined. All the code implemented is attached as supporting material, `'PHYS309_XRDProject.ipynb'`.

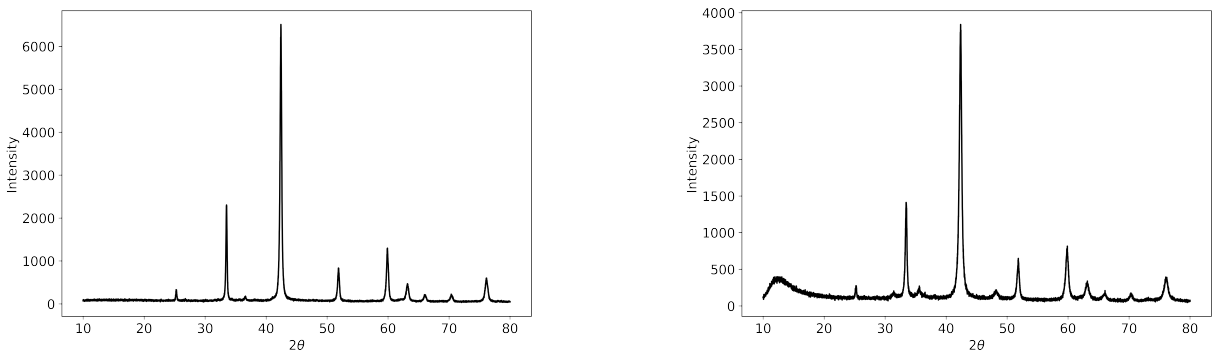

Figure 1: PXRD spectra, ungrinded sample (left), grinded sample (right).

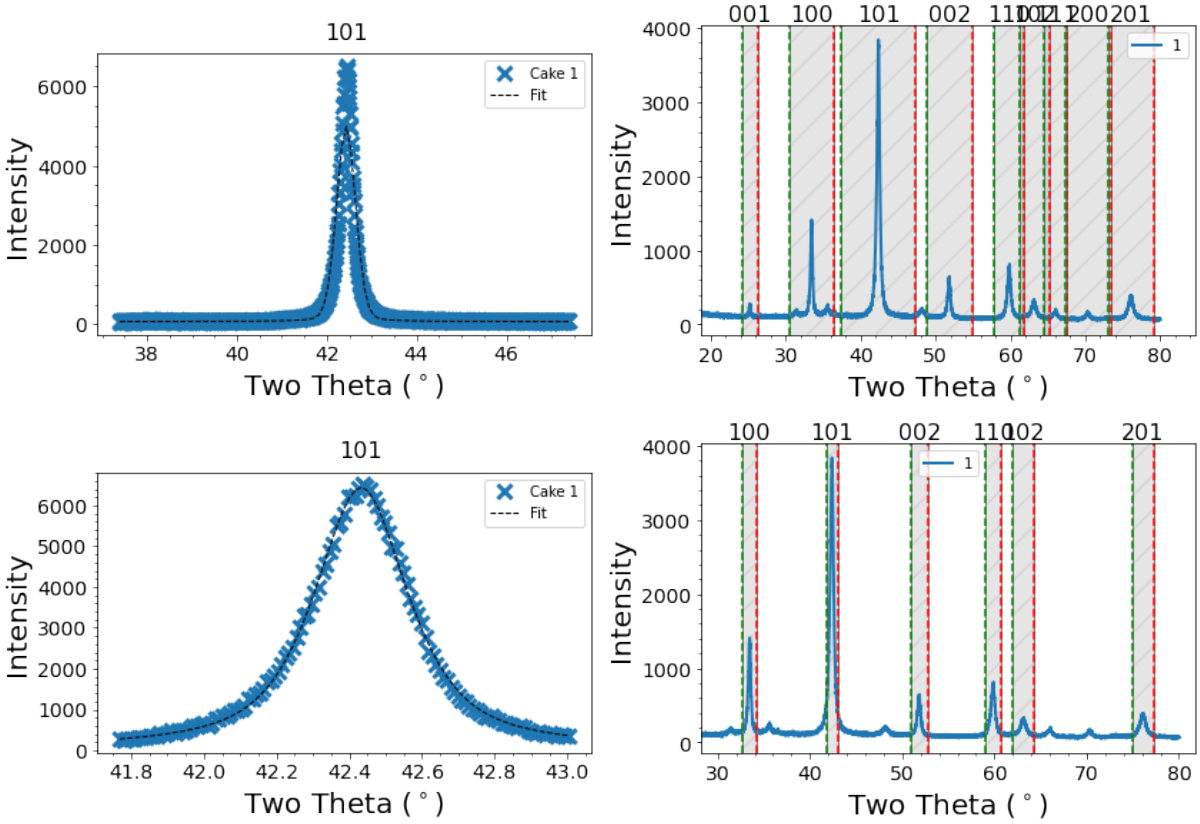

Figure 2: Plots showing the peak fitting and the intervals, old (above), revised (below).

3 Results & Discussion

First, the broadening is observed, qualitatively, by overlaying the two spectra as can be seen in **Figure 3**. The Williamson–Hall plot for both samples was generated and linearly fitted in **Figures 4,5**, the data and calculations are found in **Table 2**. A revision from the previous analysis found that the peak fitting intervals were larger than needed, which gave bad fit FWHM. This is seen in **Figure 2**. Peaks corresponding the 001, 111, and 200 planes were found to have very low signal–to–noise ratio and so were disregarded to reduce the error. The linear fit approximation result are

summarized in **Table 1**.

Sample	D (nm)	β_D (rad)	ϵ	β_ϵ (rad)	`diff.`
0hrs	187	.000846	.002706	.005684	1e-06
2hrs	86	.001831	.003455	.007256	0.0

Table 1: Obtained results by W–H linear fit method, `diff.` denotes $|\text{FWHM}_{avg} - \beta_{T(fit)}|$.

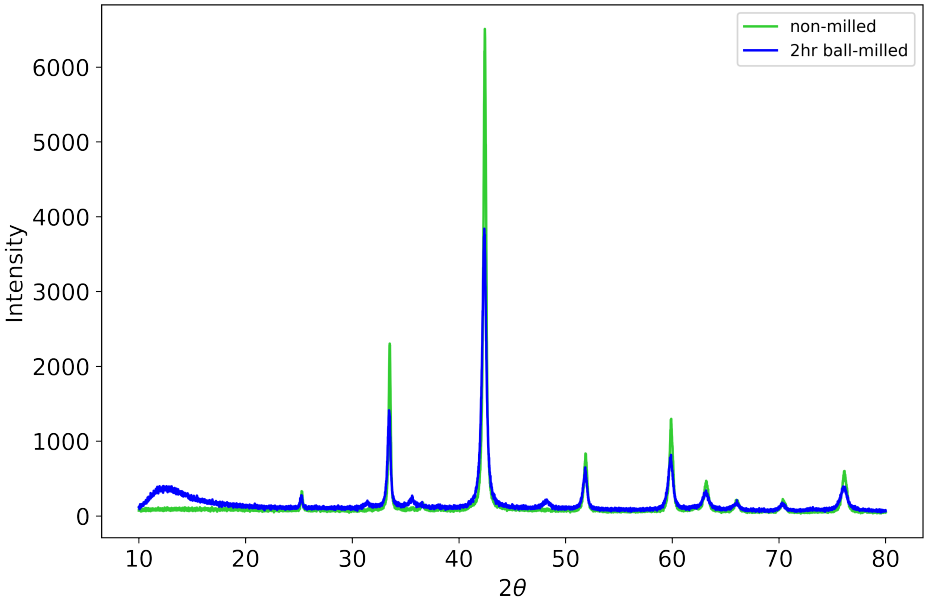

Figure 3: PXRD spectra of both samples.

.

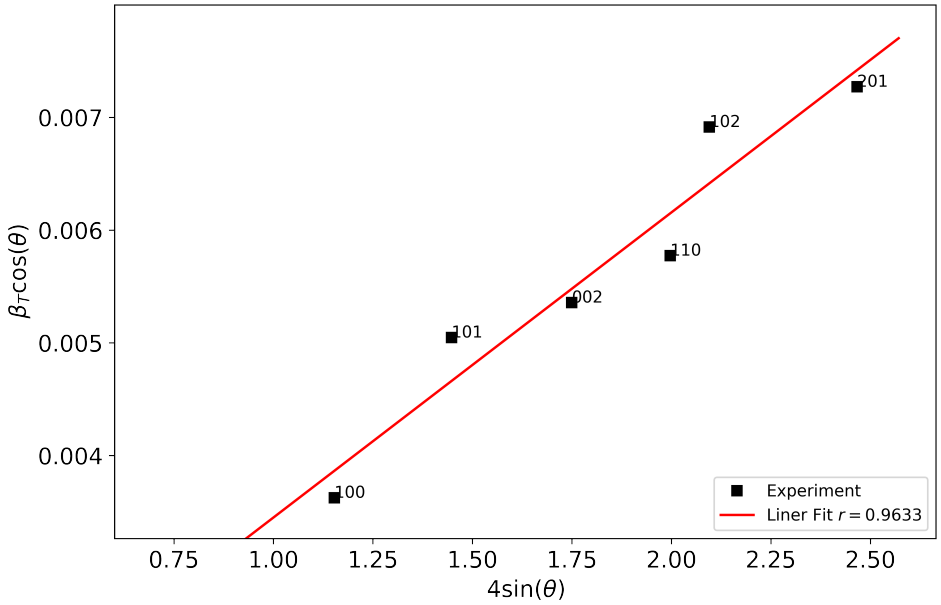

Figure 4: W–H plot of the ungrinded MgB_2 sample.

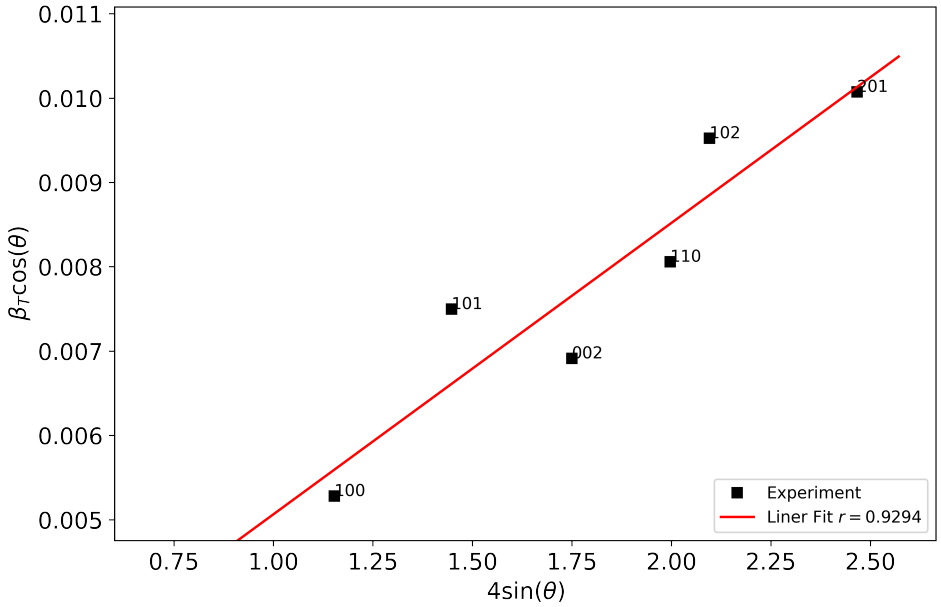

Figure 5: W–H plot of the grinded MgB_2 sample.

hkl	2θ	FWHM	$\beta_T \cos\theta$	$4\sin\theta$	FWHM	$\beta_T \cos\theta$	$4\sin\theta$
100	33.515651	.003786	.003626	1.153308	.005514	.005280	1.153209
101	42.432582	.005413	.005046	1.447559	.008043	.007498	1.447508
002	51.881419	.005957	.005357	1.749763	.007687	.006912	1.749790
110	59.903347	.006663	.005773	1.997077	.009301	.008059	1.996931
102	63.183799	.008117	.006914	2.095462	.011180	.009523	2.095644
201	76.144659	.009236	.007271	2.466623	.012797	.010074	2.466486

Table 2: PXRD data and calculations of ungrinded sample (left), grinded sample (right).

4 Conclusion

The PXRD analysis of the crystalline grain size and strain was achieved using the Williamson–Hall method for a ball–milled and ungrinded MgB_2 samples. Both linear fits had an r value greater that 0.9 after excluding low–intensity high signal–to–noise peaks. The effect of the mechanical grinding is expected to reduce the crystalline grain size and strain. Indeed, the W–H average size was is halved from 187 nm to 86 nm. The opposite effect was observed for the strain as it increased from $2.706 - 3$ to $3.455e - 3$. A TEM experiment might be helpful in validating the W–H method results.