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DEPARTMENT OF MINES AND TECHNICAL SURVEYS

OTTAWA

MINES BRANCH INVESTIGATION REPORT IR 65-56

THE EFFECT OF GRAIN SIZE ON DIFFRACTED X-RAY INTENSITIES OF QUARTZ

by

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COPY NO. 5

JULY 13, 1965

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SUMMARY OF RESULTS

Grain size has an important effect on the intensity of the X-ray beam diffracted from the $10\bar{1}1$ plane of quartz. The optimum grain size, both in terms of peak intensity and reproducibility of results, was found to be in the range from 5 to 40 μ.

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INTRODUCTION

In order to determine the effect of sized and unsized ground products on the intensity of the diffracted X-ray beam and to establish optimum analytical conditions for quantitative analysis, it is necessary to know the effect of varying grain size. The relationship between the grain size of sized samples of quartz and the intensity of a diffracted X-ray beam has been investigated by a number of workers, including Brindley (1, 2) and Weiskirchner (3). In routine diffractometer analysis, however, this relationship is not of direct value because, in practice, an unsized product is analyzed, and this normally consists of material with a wide range of sizes. This investigation was undertaken to determine the effects of both sized and unsized products on the intensity of the diffracted X-ray beam and, from this, to establish the optimum degree of comminution.

APPARATUS

The apparatus used was a Philips basic X-ray unit with a wide-angle goniometer, scaling circuit, scintillation counter, pulse-height analyzer and a strip chart recorder.

The unit was operated at 40kV and 20mA. The scaling factor was set at 128 with a time constant of 4. The pulse-height analyzer base line was set at 6V, the window at 3V, and the amplifier gain at 60. The divergence slit was 1°, the receiving slit .003" and the scatter slit 1°. The scanning rate was at 1°,2θ per minute and the chart speed was 1 inch per minute.

Nickel-filtered copper radiation was used throughout the entire series of tests.

EXPERIMENTAL PROCEDURE

A sample of Brazilian quartz was crushed, pulverized and sampled by means of a laboratory sample splitter.

One half was then screened in a power driven Ro-tap giving -48+65, -65+100, -100+150, -200+325, -325+400 and minus 400 mesh fractions. The -400 mesh fraction was sized by means of a Roller air sizer which produced -40+20, -20+10, -10+5 and -5μ fractions. The -5μ fraction was then ground with alcohol in a mechanical mortar for 48 hours and sized further to -5+2, -2+0.2 and -0.2μ fractions using a sedimentation column technique described by Brydon (4).

Ten individual rectangular aluminum diffractometer mounts were prepared from each sized fraction. Each mount was scanned on the X-ray

diffractometer at 2θ values from 20 to 27° in order to cross the $10\bar{1}1$ reflection which has a d-value of 3.34A and diffracts at a 2θ value of 26.60° with copper radiation. All sized fractions larger than ~5μ were then ground for 1 hour in a mechanical mortar and re-scanned.

The other half of the sample was screened through individual screens one mesh size at a time, in order to obtain fractions that would contain all sizes below that particular screen size. The resulting samples were scanned on the X-ray diffractometer, after which each was then ground for one hour and re-scanned.

Intensities of the $10\bar{1}1$ quartz peak were determined simply by measuring the peak height on the diffractometer chart.

RESULTS OF INVESTIGATION

As shown in Figure 1, the maximum diffracted intensity of the $10\bar{1}1$ quartz peak is obtained with material having a grain size between about 5 and 40 microns. The intensity is diminished at both coarser and finer grain sizes. The declining intensity for extremely fine-grained material has been attributed to an amorphous or imperfectly crystalline layer surrounding the more perfectly crystalline material (Armstrong (5); Nagelschmidt et al. (6); Alexander et al. (7)). However, after etching the -5 and +2 micron fraction with concentrated hydrofluoric acid for 15 minutes, which should have been sufficient to remove any amorphous layer, there was no improvement in the diffraction intensities.

The effect of grinding the sized fractions in a mechanical mortar is shown in Figure 2. The diffracted intensities of the fractions from 5 to 40 microns in grain size remained essentially unchanged, whereas the intensities of the coarser fractions showed a substantial increase.

Figure 3 shows the results obtained with material consisting of a range of grain sizes. The results are much more erratic than those obtained with sized samples. After grinding, however, the peak heights are much more consistent (Figure 4), and not strongly dependent on the grain size of the samples before grinding.

CONCLUSIONS

The optimum grain size for quantitative X-ray diffractometry of quartz, both in terms of maximum peak height and reproducibility, is between 5 and 40 microns. This is in general agreement with the results of other investigators, such as Brindley (1, 2) and Weiskirchner (3). It follows, therefore, that samples containing quartz should be ground to sizes falling predominantly within this range. In practice this can be achieved by grinding the samples in a mechanical mortar for a suitable period of time. Over-grinding, however, must be avoided because of the decrease in diffracted intensity at sizes below 5 microns.

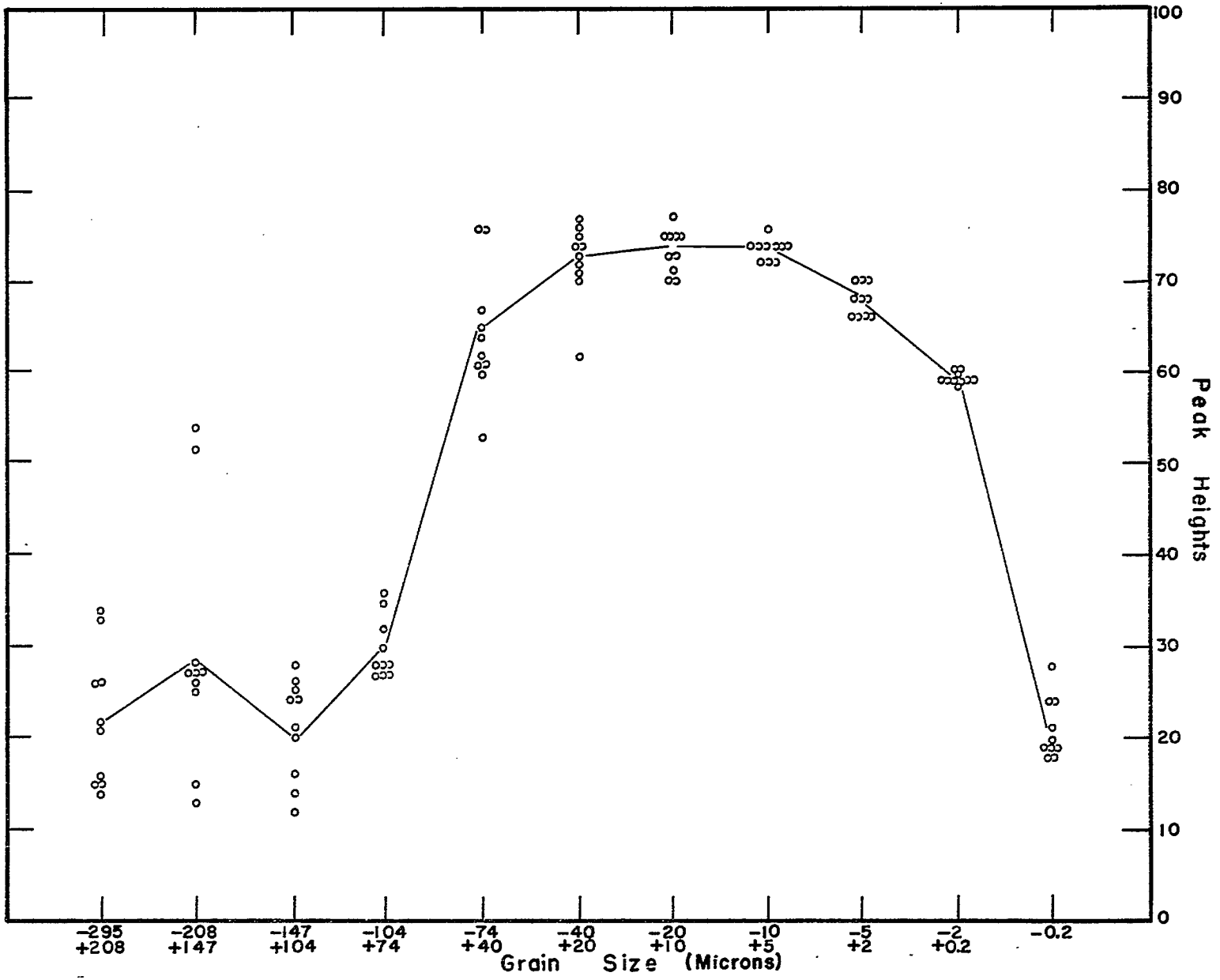

Figure 1 - Peak heights of the quartz $(10\bar{1}1)$ reflection obtained with screened fractions of quartz. Each circle in this and subsequent figures represents one determination.

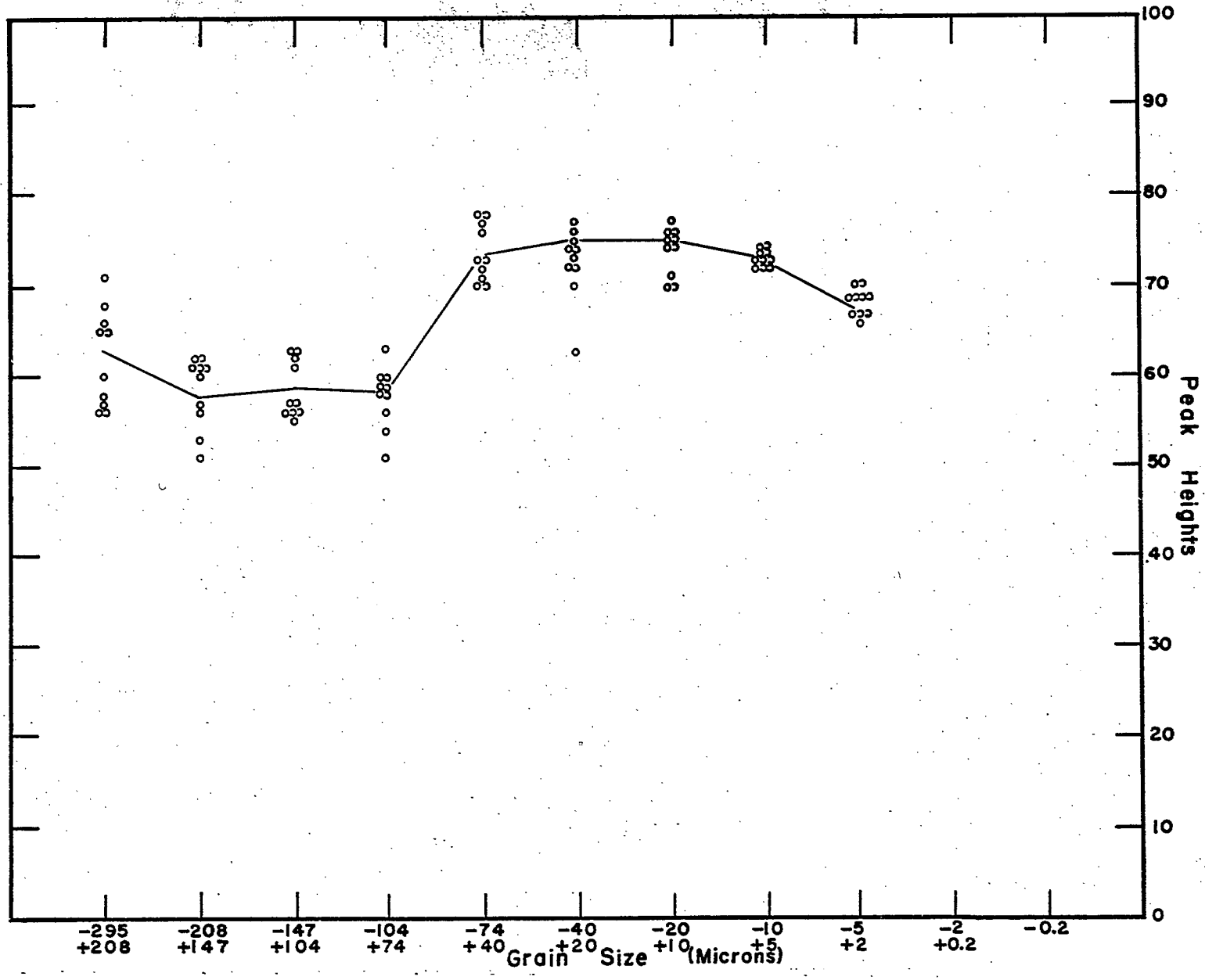

Figure 2 - Peak heights of the quartz ($10\bar{1}1$) reflection of the quartz samples shown in Figure 1, followed by one hour grinding in an agate mortar.

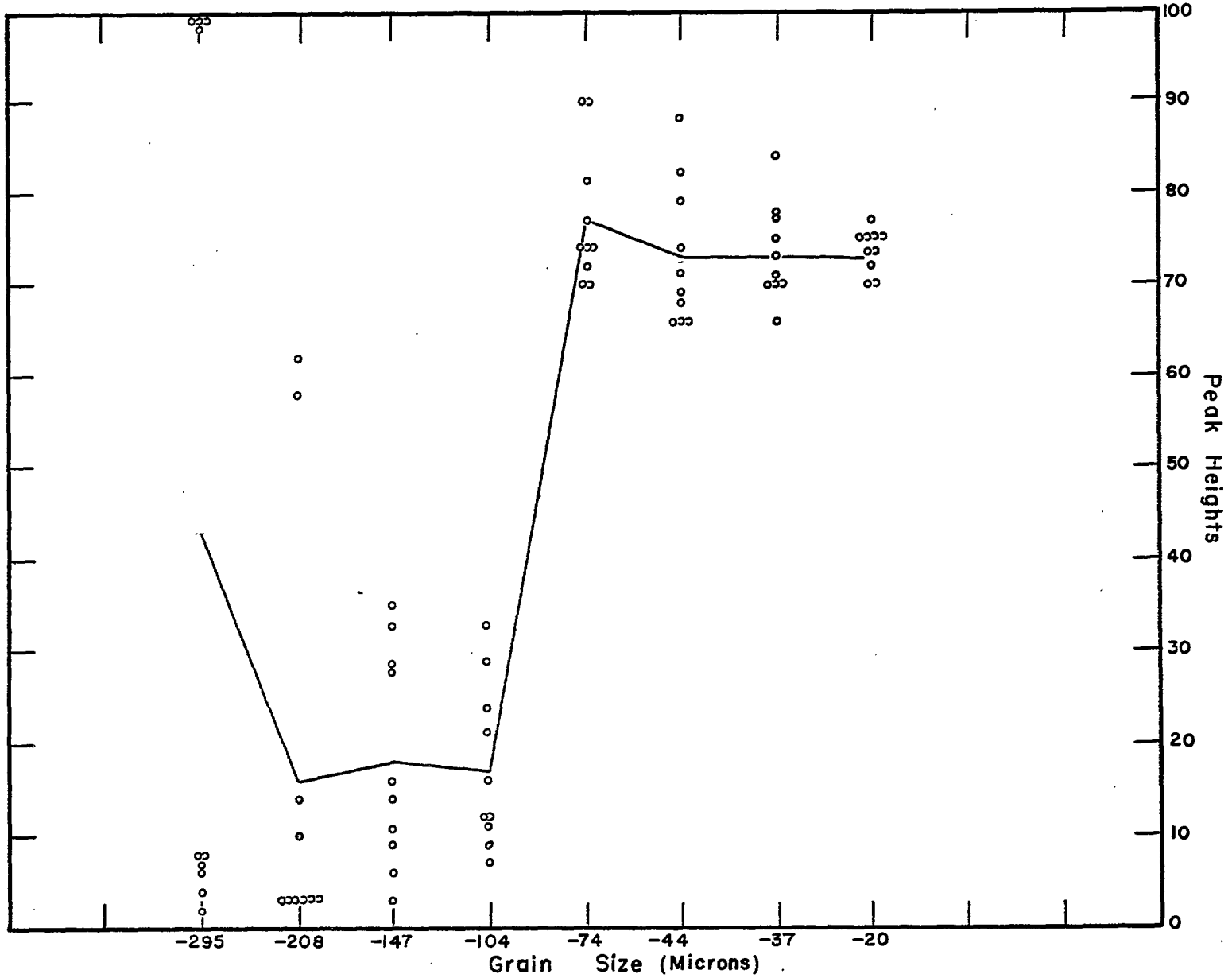

Figure 3 - Peak heights of the quartz ($10\bar{1}1$) reflection obtained with unsized samples of quartz, the maximum grain size of each fraction being shown on the abscissa.

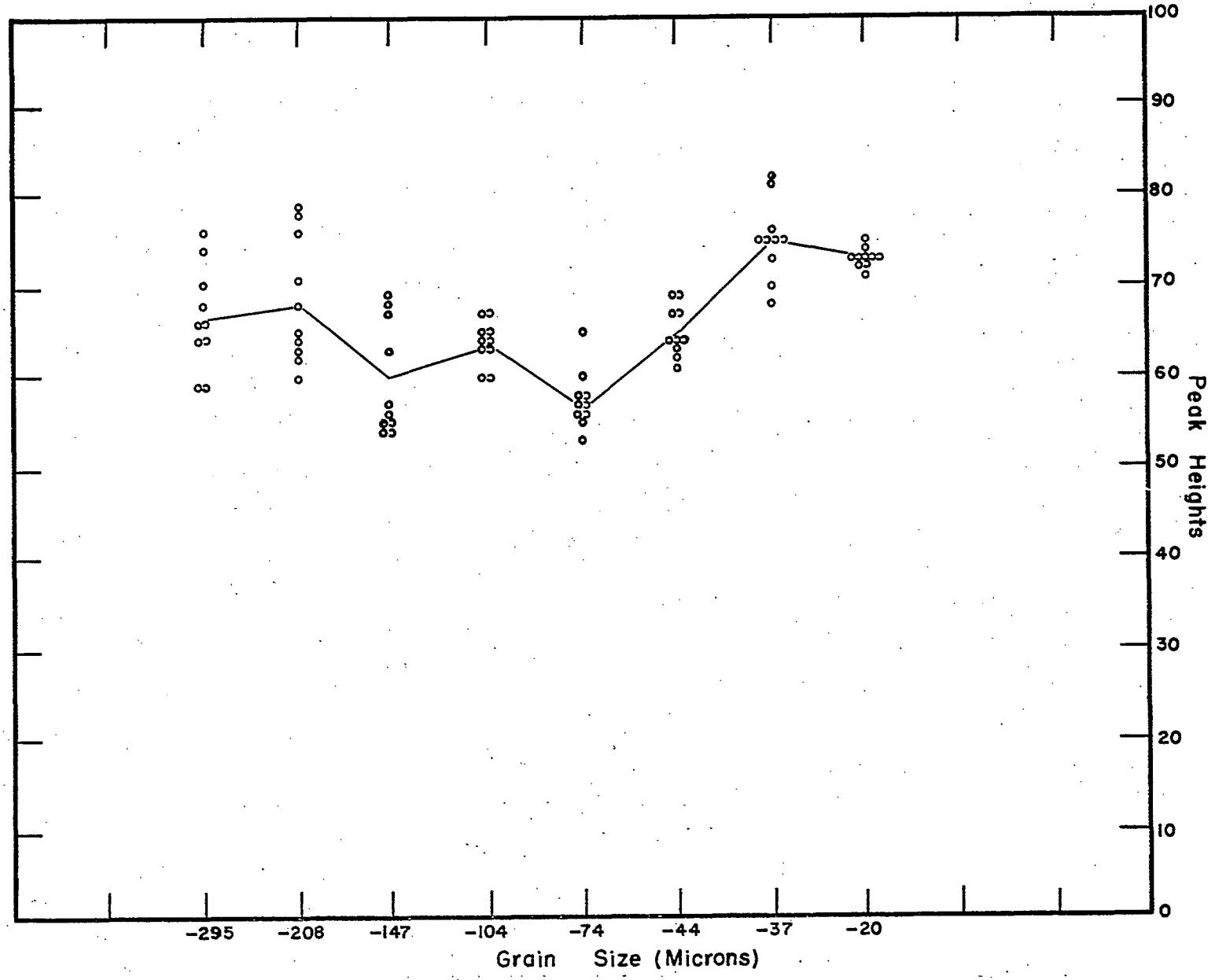

Figure 4 - Peak heights of the quartz ($10\bar{1}1$) reflection obtained with the quartz samples shown on Figure 3, followed by one hour grinding in an agate mortar.

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