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A LINE PROFILE BROADENING ANALYSIS OF COLD WORKED AND ANNEALED α-70-30 BRASS BY FOURIER TRANSFORM METHOD

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Abstract

The Fourier transform method has been utilized to analysis line profile broadening and root mean square strain of cold worked and annealed 70-30 α-brass. The crystallite size obtained from cold worked fillings are found small as compared to that of annealed specimen. Where as the root-mean square strain obtained from cold worked specimen are found little higher than annealed specimen.

1. Introduction

A detailed knowledge of crystallite size, shape and strain in a finely divided powder often helps to correlate many physical properties of a system undergoing transformation and solid state reactions [1]. The analysis of broadened x-ray diffraction lines is one of the most powerful method of investigating of dislocations in heavily deformed materials. Further x-ray line broadening analysis provides a method of finding bulk average size of coherently diffracting, domains and r.m.s. strain. Various methods are available for detailed study of line profiles and strain analysis [2,3]. A Fourier transform method has been indicated by stokes for obtaining the corrected profile of the line. Fourier transforms are made from experimental curve and from the curve for the well crystallized powder. The quotient of these two functions is the Fourier transform of true line profile of the powder studies. Foruier transform method is the most powerful technique for deducing the pure diffraction profile of a line free from the effects introduced by the measuring apparatus [4]. A detailed description of this method is given in the article below. The present investigation aims to find the variation of crystallite size and strain in cold worked and annealed 70-30 α-brass and hence to show if these have any influence on the stacking faults.

2. Theory / Literature Review

2.1 Broadening Due to Crystallite Size

A pure maximum diffraction produced by a crystalline powder has a natural profile which is determined largely by the crystallite size distribution, the nature and magnitude of lattice distributions [5]. For a polycrystalline specimen consisting of sufficiently large and strain free crystallites, line broadening will be exceedingly sharp.

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In actual experiments line profiles of such sharpness are never observed because of the continued effects of number of instrumental and physical factors g (ϵ) that broaden the pure diffraction line profile f (ϵ). Of more fundamental importance to the present discussion is the fact that the shape and breadth of the pure profile of f (ϵ) are determined both by the mean crystallite size and by the particular imperfections prevailing in the crystalline lattice, (vi) Therefore, there are two main tasks to analysis the line profile. (i) extracting the pure diffraction profile f (ϵ) from the experimental profile h (f) and (ii) extracting the size and imperfection information from the profile f (ϵ) of suitable lines of the pattern. Operation (i) is feasible because of the convolution relationship among:

h (ϵ), g (ϵ) and F (ϵ) i.e.

$$h(\epsilon) = -\alpha\int^{+\alpha} g(n), f(\epsilon - n)\, dn \quad \ldots\ldots (1)$$

this convolution may be expressed as

$$h(\epsilon) = g(\epsilon), f(\epsilon) \quad \ldots\ldots (2)$$

Where g express the sum total of the apparatus effects upon the line profile being measured. The variable is the angular deviation of any point from the theoretical scattering angle 2θ. Thus f (ϵ) can be obtained from h (ϵ) by the process of deconvolution (unfolding). Operation (ii) involves two cases (a) only size or imperfection broadening present and (b) both causes of broadening present simultaneously.

2.2 Fourier Transform Method of Line Profiles

With reference to equation (1), let the Foruier transforms of h (ϵ), g (ϵ) and f (ϵ) be H (γ) G (γ) and F (γ) respectively. By applying Fourier integral theorem [7], it can be shown that

$$F(\gamma) = H(\gamma) / G(\gamma) \quad \ldots\ldots (3)$$

and further more

$$F'(\gamma) = \sum_{\gamma} H(\gamma) \exp / G(\gamma) \cdot -2\pi i\epsilon(\gamma) / \epsilon m) \quad \ldots\ldots (4)$$

Where,

$$G(\gamma) = Gr(\gamma) + iGi(\gamma) \quad \ldots\ldots (5)$$

Similarly

$$H(\gamma) = Gr(\gamma) + iHi(\gamma) \quad \ldots\ldots (6)$$

Substituting values of G (γ) and H (γ) from equation (5) and (6) in equation (3), we can find the value of F (γ) as:

$$F(\gamma) = H(\gamma) / G(\gamma)$$

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$$= \quad Hr + iHi \,/\, Gr + iGi \quad \ldots\ldots (7)$$

Further more by multiply and dividing the above equation by

(Gr - iGr), we get, F (γ) as

$$F(\gamma) = \frac{(Hr + iHi)(Gr - iGi)}{Gr^2 + Gr_i^2} \quad \ldots\ldots (8)$$

which gives, real and imaginary components of F as:

$$Fr = \frac{Gr\,Hr + Hi\,Gi}{Gr^2 + Gi^2} \quad \ldots\ldots (9)$$

$$Fi = \frac{Hi\,Gr + Hr\,Gi}{Gr^2 + Gi^2} \quad \ldots\ldots (10)$$

Now, the desired pure diffraction line profile f (ϵ) can be computed as follows.

$$f(\epsilon) = \sum_{\gamma} Fr(\gamma) \cdot \frac{\cos 2\pi\,\epsilon(\gamma)}{\epsilon m} + \Sigma\, Fi(\gamma) \sin \frac{2\pi\,\epsilon\,\gamma}{\epsilon m} \quad iF\,\gamma(\gamma)\ \text{Sin}$$

$$\frac{2\pi\,\epsilon(\gamma)}{\epsilon m}_{\gamma} + i\Sigma\, Fi(\gamma) \cos \frac{2\pi\,\epsilon\gamma}{\epsilon m} \quad \ldots\ldots (11)$$

Since the summations extend overall +ve, and -ve values of f (ϵ) therefore the last two terms on the right hand side of above equation reduce to zero and thus f (ϵ) can simply be calculated from:

$$F(\gamma) = \Sigma\, Fr(\gamma) \cos \frac{2\pi\epsilon\gamma}{\epsilon m} + \Sigma\, F(\gamma) \sin \frac{2\pi\epsilon\gamma}{\epsilon m} \quad \ldots\ldots (12)$$

To, measure experimental profile, angular range ϵm is divided into 60 parts, then select an artificial unit of angle so small that m = 60 units. Then, for evaluating pure diffraction profile

f (ϵ), eq. (12) will take a form of:

$$F(\gamma) = 1/60\, \Sigma r\, Fr(\gamma) \cos \frac{2\pi\epsilon(\gamma)}{60} + 1/60 \sum_{\gamma} F \sin \frac{2\pi\epsilon(\gamma)}{60} \quad \ldots\ldots (13)$$

For better inference, it is suggested to divide the measurement range ± ½ ϵm into a sufficiently large number of intervals. Despite of its limitations, the Fourier method is the most powerful techniques for deducing the pure diffraction profile of a line free from the effects introduced by the measuring apparatus.

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3. Experimental Work

3.1 Sample Preparation

For present work, sample was prepared by hand-pressing the 70-30 α- brass filling into a suitable holder using Canada balsam in Xylol as a binder. The grain size taken was 0.01 mm. Surface damage was removed by etching. Three sample were used, one was cold worked and other two were used as standard samples both of them were annealed at 300°C in Nitrogen atmosphere. One of which was annealed for three days and other for one week.

3.2 Experimental Procedure

A Philips automatic power diffractometer with a copper (Cu) target, Nicle filter, 1° divergence silt, l° scatter slit and 0.2 mm receiving slit was used to study the broadening of the line profiles of the specimens prepared. The intensities for reflections from 70-30 α- brass fillings were measured in the continuous mode with scan ratio 0.01° 2θ per minute.

Printouts of these experiments for three sample for all reflections form (111) upto (222) are given in the figure 1.2 and 3. Correction for instrumental broadening was carried out with Stock's method, the back ground was assumed to be linear. Fourier co-efficient for the samples used for cold worked and annealed peaks were calculated with a programmable scientific calculator. Then these co-efficient were used to find the co-efficient of Fourier series which were corrected for instrumental broadening.

4. Experimental Results

Fourier co-efficient by one week annealed and three days annealed samples are given in Table 1 and 2. These co-efficient were calculated from the experimental printouts (figure 1,2,3) and logarithmic plots of the distortion co-efficient A^D_n (1) versus order of reflections l = n (h+k+l) and loralrithmic plot of particle size A^S_n versus $ho^2 = (h^2 + k^2 + l^2)$ for 70-30 α- brass as shown in figure (4) and (5). These co-efficientes are free from instrumental broadening, the real part of these co-efficient was normalized to $A_0 = 1$ and therefore used for further calculation to evaluate domain size and average strain, see tables 1,2,3,4 and figures 4 & 5. Domain size obtained was 179°A and root mean square strain is 0.0059 (5.9×10^3) for cold worked specimen by using three days annealed standards, where as domain size for the same specimen, by using one week annealed standard was found 155°A and root mean square strain was 0.00654 (6.5×10^{-3}). The second derivative of the particle size co-efficient gives very small particle size about 65°A, and 25°A for one week annealed and three days annealed samples, see figures (6 and 7).

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5. Conclusions

The particle size values obtained from cold worked 70-30 α-brass fillings are often surprisingly small, of the order of 25°A and 179°A. Obviously the material has not been fragmented into separate small particles of this size. Instead of this cold work has produced some sort of a domain structure within the filling, such that the different domains diffract essentially incoherently with respect to one another, as in our case. In some materials stacking faults are responsible for a large part of the particle sizes roadening, but there are some other materials which show very small particle size and for which there is no-evidence of stacking faults. For these materials there must be other types of effective domain boundaries. One type of boundary which may be important in a cold worked material is the small angle boundary.

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TABLE : 1

INTERCEPTS AND SLOPES FROM ONE WEEK ANEALED SAMPLE

n	L (A^O) Crystal Size	Size Coef. LnA_L^S ($x10^{-2}$)	Fourier Cos. Coeff. A_L ($x10^{-2}$)	Distoration Coeff. LnA^D_n (*l*)
1	22	-10.0	90.048	-1.6×10^{-2}
2	44	-27	76.338	-2.5×10^{-2}
3	66	-46.5	62.8	-1.33×10^{-2}
4	88	-62.6	53.79	-5×10^{-3}
5	110	-72.0	48.92	-2×10^{-3}
6	132	-77.75	45.95	-5×10^{-3}
7	154	-81.75	44.15	-5×10^{-3}
8	176	-85.0	85.05	-3.33×10^{-3}
9	198	-86.5	42.105	3.3×10^{-3}

TABLE : 2

INTERCEPTS AND SLOPES FROM 3 DAYS ANEALED SAMPLE

n	L (A^O)	LnA_L^S ($x10^{-2}$)	A_L ($x10^{-2}$)	LnA^D_n (*l*) $x\ 10^{-2}$
1	22	-9.0	91.39	1.75
2	44	-23.5	79.06	2.84
3	66	-40.0	67.0	2.16
4	88	-52.5	59.15	1.33
5	100	-59.0	55.43	1.42
6	132	-61.0	54.0	2.16
7	155	-63.5	52.99	2.25
8	176	-64.0	72.23	2.58
9	198	-64.0	57.72	2.75

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TABLE : 3

FOURIER COEFFICIENTS BY ONE WEEK ANNEALED SAMPLE

n	L(A)°	(111) Reflection			(222) Reflection		
		Al	Norma-lized Al	LnAl	Al	Norma-lized Al	LnAl
0	0	0.9845	1	0	0.5002	1	0
1	22	0.8465	0.860	0.151	0.3694	0.759	-0.303
2	44	0.6940	0.705	0.350	0.2849	0.570	-0.3565
3	66	0.5915	0.601	0.509	0.2640	0.528	-0.640
4	88	0.5220	0.530	0.634	0.2558	0.511	-0.671
5	110	0.4763	0.844	0.726	0.2412	0.482	-0.729
6	132	0.4464	0.453	0.791	0.2195	0.439	-0.824
7	155	0.4277	0.434	0.834	0.2092	0.418	-0.872
8	176	0.4161	0.423	0.861	0.2037	0.409	-0.893
9	198	0.4090	0.415	0.878	0.2021	0.402	-0.94

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TABLE : 4

FOURIER COEFFICIENTS BY THREE DAYS ANNEALED SAMPLE

n	L(A)^{o}	(111) Reflection			(222) Reflection		
		Al	Norma-lized Al	Lnal	Al	Norma-lized Al	Lnal
0	0	1.5110	1	0	0.6012	1	0
1	22	1.3086	0.866	-0.144	0.4407	0.733	-0.311
2	44	1.0903	0.720	-0.326	0.3366	0.560	-0.580
3	66	1.0903	0.627	-0.467	0.3310	0.517	-0.659
4	88	0.8562	0.567	-0.567	0.3002	0.499	-0.694
5	110	0.8005	0.509	-0.635	0.2800	0.466	-0.764
6	132	0.7665	0.507	-0.679	0.2534	0.421	-0.864
7	155	0.7479	0.495	-0.703	0.2412	0.401	-0.913
8	176	0.7393	0.489	-0.714	0.2346	0.390	-0.941
9	198	0.7361	0.487	0.719	0.2313	0.385	-0.955

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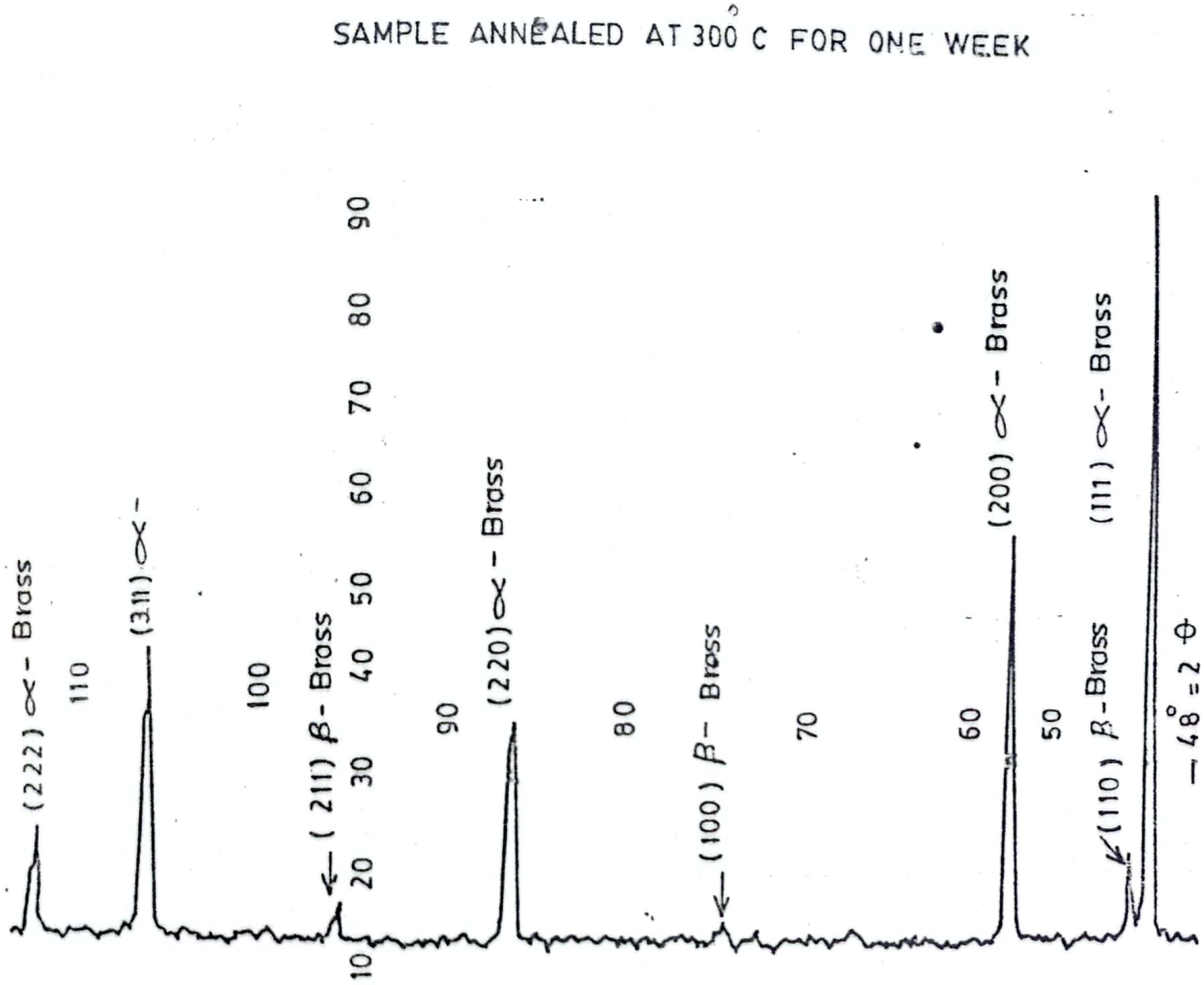


FIGURE 1. RECORDING OF ONE-WEEK ANNEALED SAMPLE.

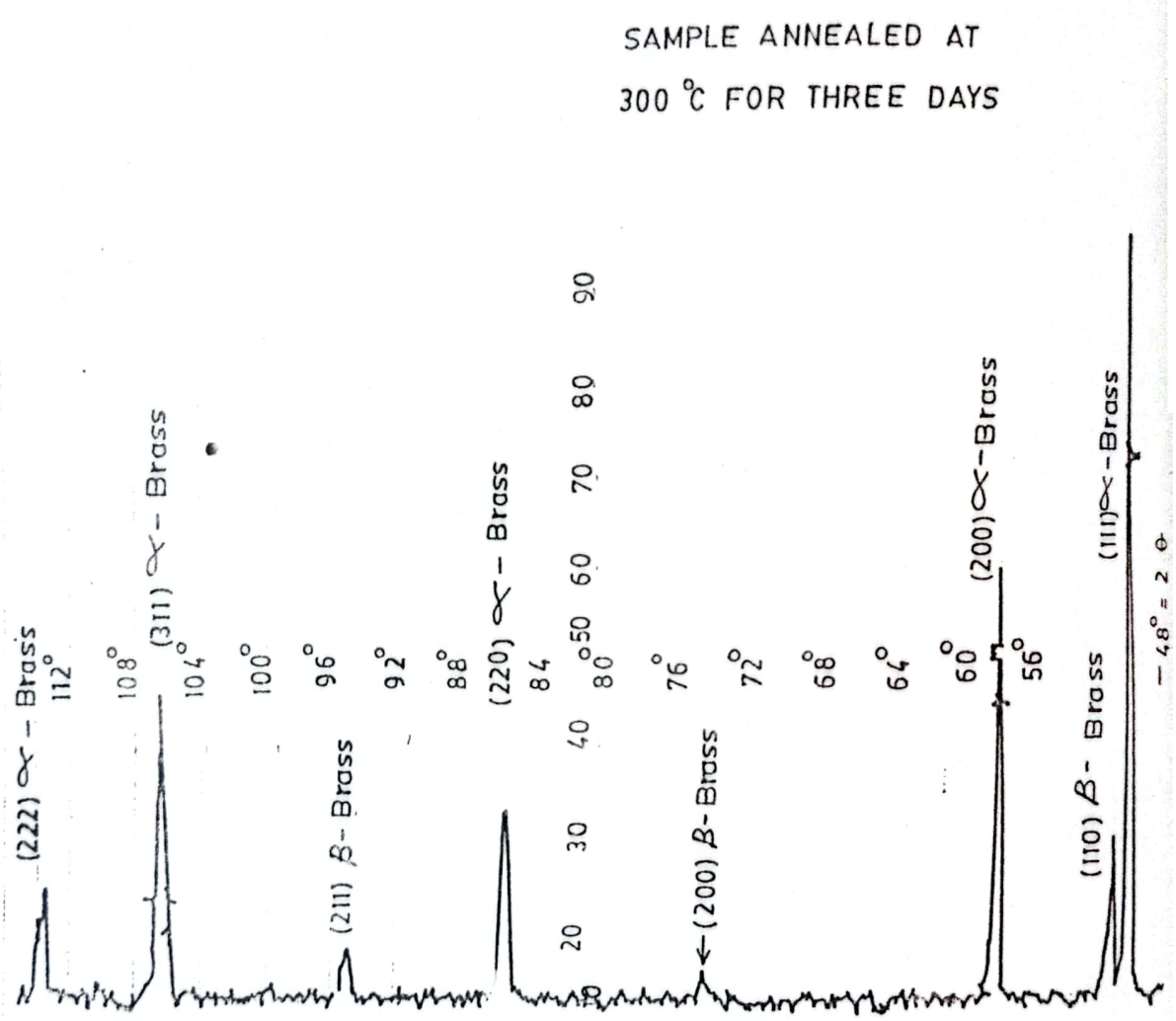


FIGURE 2. RECORDING OF THREE-DAY ANNEALED SAMPLE.

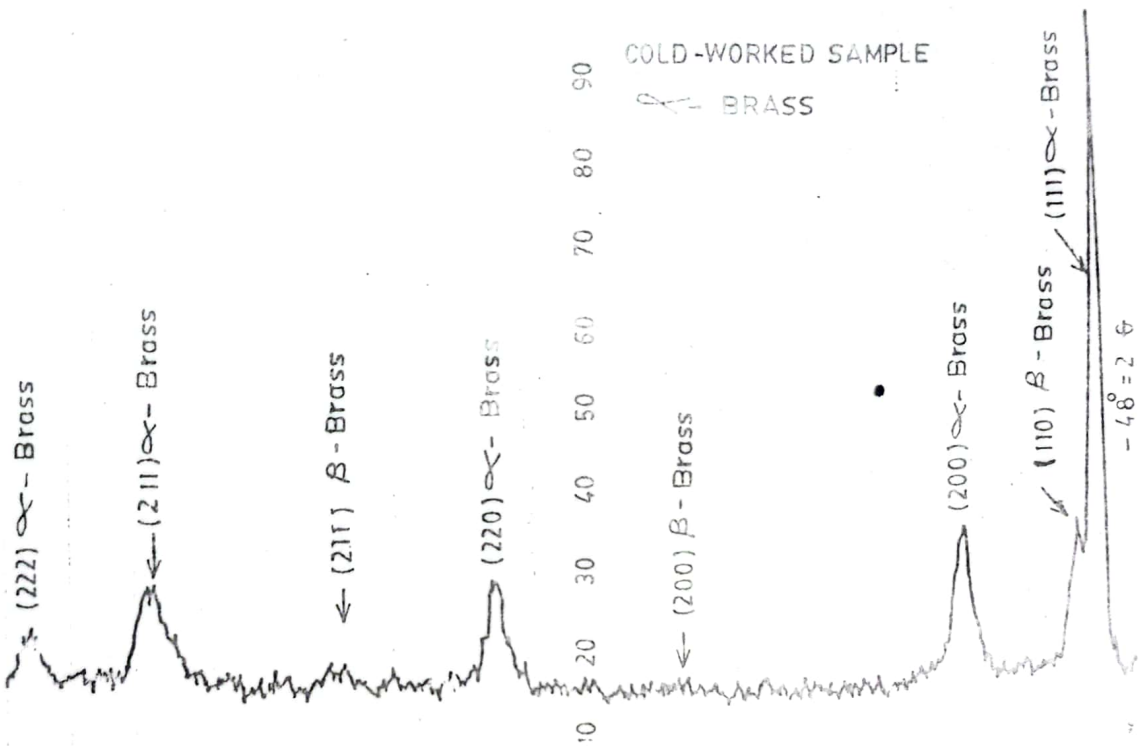


FIGURE 3. RECORDING OF COLD-WORKED SAMPLE.

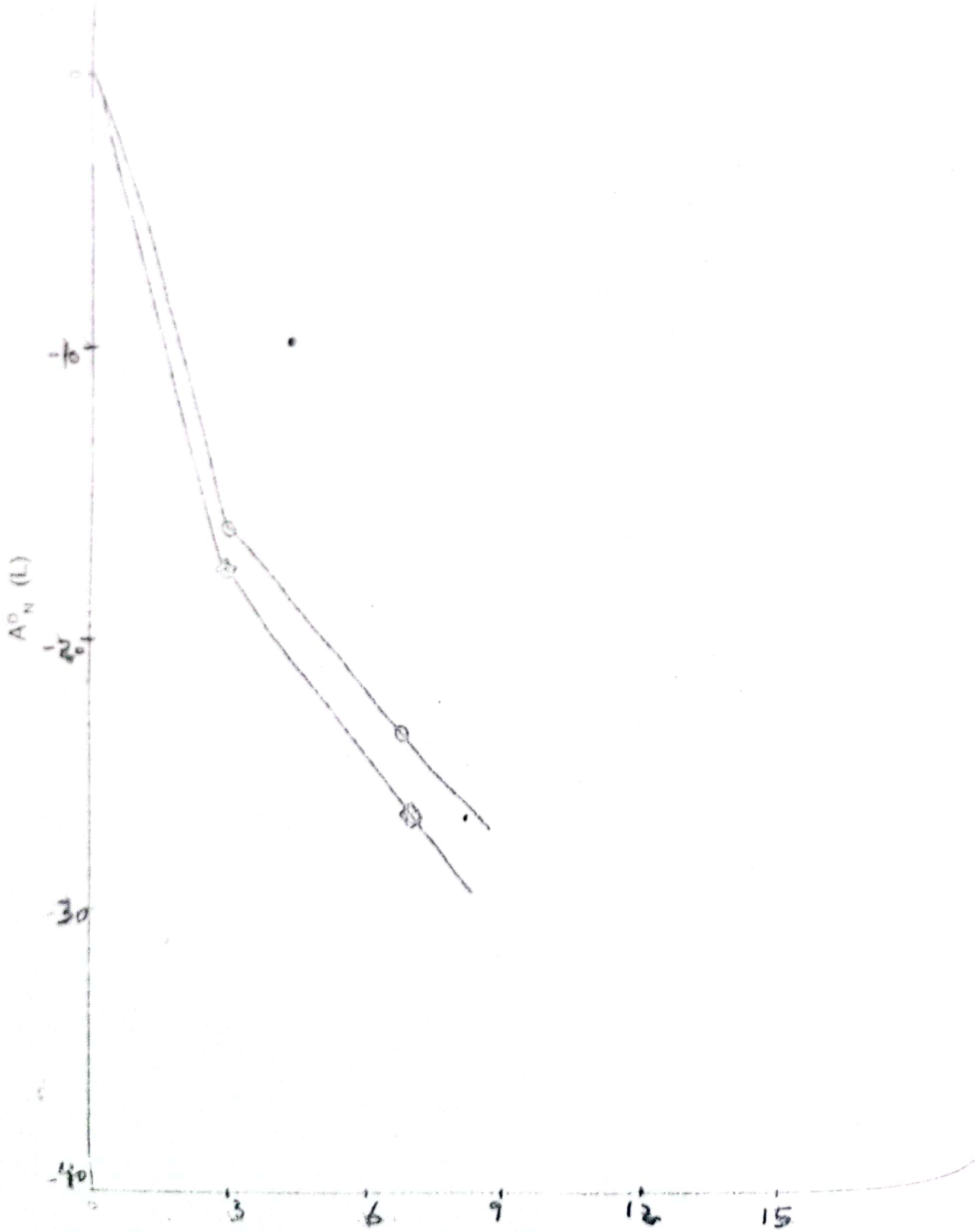


figure 4: Plot of Distortion Co-efficient Versus L = n (h + k + l)
O for one week annealed sample
□ for 3 days annealed sample

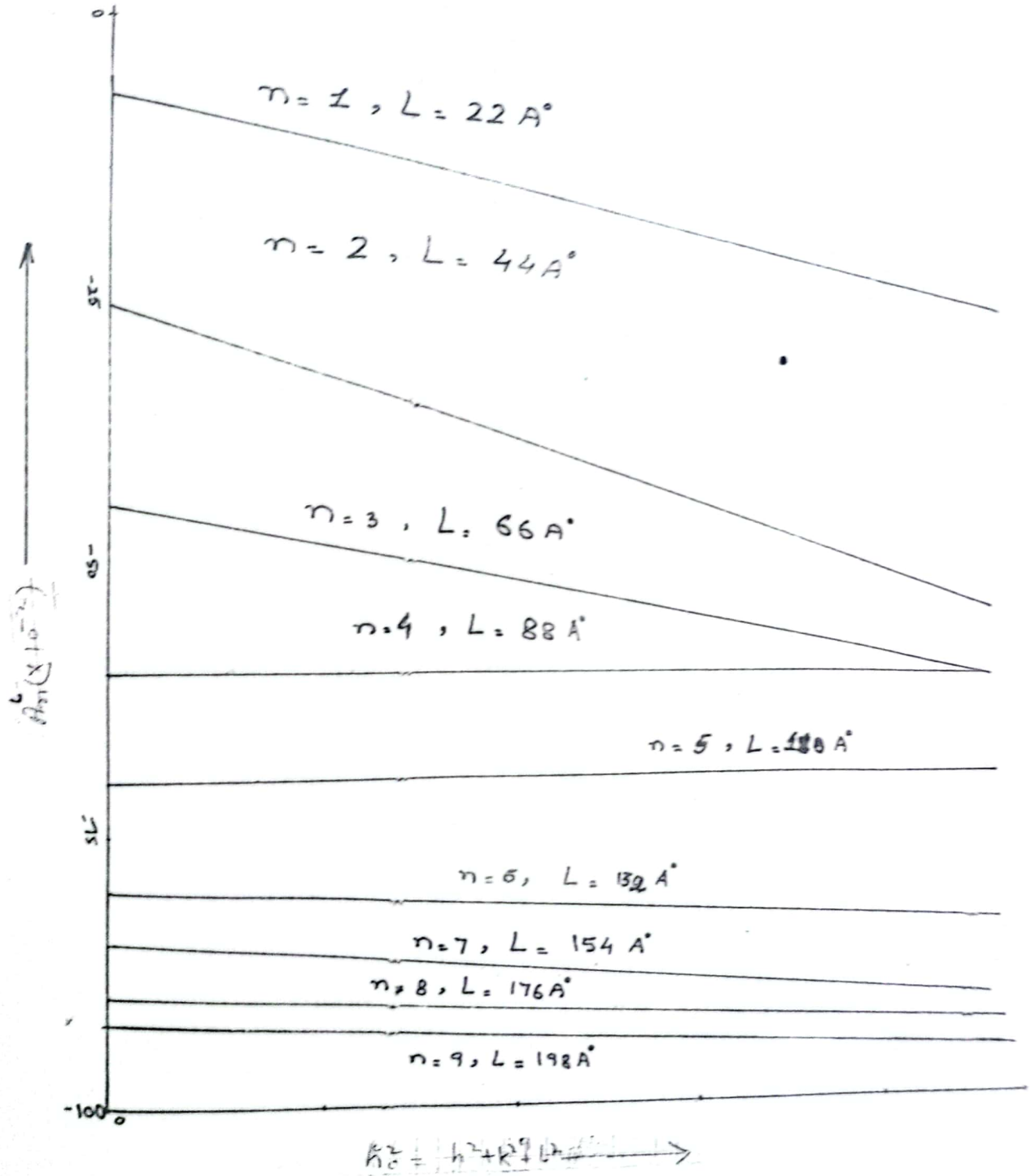


FIGURE 5 : PLOT OF $l n A_n^2$ VERSUS ho^2
(one week annealed sample)