COMMUNICATIONS

DE LA FACULTÉ DES SCIENCES
DE L'UNIVERSITÉ D'ANKARA

Série A: Mathématiques, Physique et Astronomie

TOME 22 A ANNÉE 1973

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Ankara, Turquie

Communications de la Faculté des Sciences de l'Université d'Ankara

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Orientation Preference In The Growth Of Alkali Metal Single Crystals By Using Bridgeman Technique

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ABSTRACT

The single crystals of alkali metals (Na, K, Li, Rb and Cs) have been obtained in varying sizes of glass capillaries by using Bridgeman technique without employing a seed. The cubic crystals grown this way are theoretically [3] expected to have random orientations. We checked the theory by determining the distribution of orientations for each metal and found that there is no obvious preferred orientation about any crystallographic axis.

χ^2 analysis and Binomial distribution have been used as mathematical tools in experimental varification of the theory of random orientations and our data have been found to support it to a considerable extent.

I. INTRODUCTION

The growth of alkali metal crystals (Na and K) normally requires the use of Bridgeman technique [1]. It is a method of the preparation of single crystals by the controlled solidification [2] of molten metals. In this method a boat is used as the mold and a seed is located at one end of the boat in such a way that the known crystallographic axis of the seed is along the boat axis. When the molten metal is cooled down slowly from the seed end along the axis of the boat a single crystal is produced and the crystal grows along the crystallographic axis of the seed.

There is very little work done on growing the crystals of the remaining alkali metals which are Li, Rb, and Cs, and the

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present work concerns into growing the crystals of these metals as well as those of Na and K by using Bridgeman sealed tube technique without using a seed and studying their orientations by X–ray Laue method.

The single crystals obtained by using Bridgeman technique without employing a seed are normally expected to have random orientations [3]. However, Watanabe [4] has shown that Zn, Bi and Sn crystals were grown with a preference along the directions perpendicular to the (001), (111) and (001) planes, respectively, which is possible due to the differences in the crystallographic axes in these noncubic crystals. On the other hand, the crystals of alkali metals are cubic, such that [100], [010] and [001] crystallographic axes, and similarly [110], [101] and [011] axes, are equivalent to each other. Therefore, for isotropic cubic crystals one should not expect a preferred orientation along any direction. However, in the field of crystal growth there is a lack of balance between experimental results and theory, and the crystal growth from the melt incolves many problems. This might require getting some experimentally obtained information about crystal orientations.

In the present work we have studied the orientation behavior of the alkali crystals Na, K, Li, Rb and Cs obtained by the Bridgeman sealed tube technique to see experimentally if the above prediction about preferred orientation is valid. One factor on which the crystal growth might depend, if confined to grow in a capillary tube, is the dimension of the capillary and it has been discussed here in connection with crystal orientation.

The alkali metals were melted in a cruicible containing liquid paraffin to avoid reaction with air and were sucked into the glass capillary tubes of internal diameter $0.1-0.6$ mm of quartz and other various types of glass compositions. It is conventional to achieve the progressive freezing in the Bridgeman technique either by lowering the sample through a furnace or by the movement of the furnace itself [1]. Due to the experimental difficulties, however, we placed the tubes into the molten metal in an oil bath which was allowed to cool for several hours. In

this way the progressive freezing was in the radial direction along the perpendicular to the tube axis.

We were able to grow single crystals of alkali metals in almost 70 % of the cases; this percentage was quite lower for Rb and Cs. Only single crystals were found suitable to study the orientation by using stereographic projection of the Laue patterns, obtained with the help of Cu-Kα radiation and cylindrical camera.

Since an obvious preferred orientation was not apparent from the experiments, we made the hypothesis that the distribution of orientations of single crystals of alkali metals was random. We used the methods of statistical analysis in order to test whether our hypothesis was valid or not.

II. DETERMINATION OF ORIENTATION

When certain geometrical conditions are satisfied a beam of X-rays is diffracted from a crystal which acts as a three dimensional grating. One can consider diffracted rays as being reflected from planes of lattice points. In a crystal there are a very large number of planes of lattice points. Each of the spots on the film is due to the reflection of X-rays by one set of equally spaced parallel planes determined by Miller indices (hkl).

In order to get reflection from the lattice planes, the Bragg's law must be satisfied. Among the various ways of satisfying this law we used the Laue method with cylindrical cameras of diameters 50 mm and 57.3 mm., where a single crystal is held stationary in a beam of white radiation by the use of a goniometer head.

In the study of crystals, it is necessary to represent on paper the three dimensional relations of the crystal. One such representation is the stereographic projection, which is commonly used in discussing crystal orientation. Since the study of crystals is closely related with the angular and zonal relations of crystal planes, the determination of the orientation of single crystals is much facilitated and data from X-ray photographs are conveniently analyzed by the use of the stereographic projection.

1. *Interpretation of Patterns.* The Laue pattern of a single crystal consists of a set of diffraction spots which depend upon the orientation of the crystal. Identification of these spots is difficult since the wavelength of the incident beam is not known for a particular spot.

There is a geometrical relation between any diffraction spot and the stereographic projection of the pole belonging to the planes giving rise to the diffraction spot. Therefore, a chart can be prepared to convert positions of the spots on the film to posions on the stereographic projection. The polar coordinates ρ and Φ of each spot on the film can be read from the appropriate chart [5] as the pair of polar coordinates of the corresponding pole on the surface of the reference sphere.

As an illustration, let us consider a tracing of the film of a sodium single crystal numbered as Na-49, shown in Figure 1. The ρ and Φ values of these spots are read and tabulated in Table 1. We next plot these spots on the stereographic projection by the aid of a Wulff-net and find their Miller indices to disclose the orientation of the crystal.

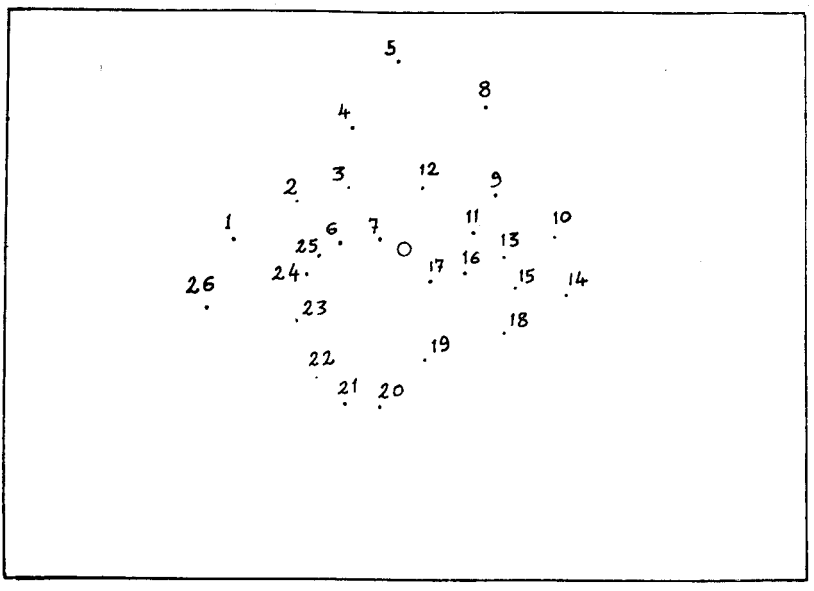

Figure 1. The tracing of the Laue photograph Na-49.

It may happen that a Laue spot may be associated with a diffused spot, produced by reflection of a characteristic radiation Cu-Kα of which the angle θ can be measured by using the chart mentioned above. The angle θ is the Bragg angle given by the Bragg equation

$$n\lambda_{K\alpha} = 2\ d\ \sin\ \theta$$

where $d = \frac{a}{\sqrt{h^2+k^2+l^2}}$ and a is the edge of the unit cell of the given lattice. Finding the Miller indices of the Laue spots associated with the diffused spots give us a very good start to find the indices of all other remaining spots.

TABLE I

Indexed Laue Spots in Figure 1.

Number of Spots	$\varrho°$	$\varnothing°$	Position	K$\alpha - \theta°$	hkl
1	87	36	Upper-left	38	$\bar{1}\ \bar{1}\ 1$
2	67	27	"	26	$\bar{2}\ \bar{1}\ 1$
3	45	29	"		$\bar{3}\ \bar{1}\ 0$
4	32	47	"	21	$\bar{1}\ 0\ 0$
5	28	85	"		$\bar{3}\ 1\ 0$
6	86	14	"		$\bar{2}\ \bar{3}\ 1$
7	—	—	"	15	$\bar{1}\ \bar{1}\ 0$
8	38	48	Upper-right		$\bar{1}\ 1\ 0$
9	60	25	"		$\bar{1}\ 3\ 0$
10	85	32	"	34	$0\ 3\ 1$
11	76	16	"	21	$0\ 1\ 0$
12	20	40	"		$2\ \bar{1}\ 1$
13	86	21	Lower-right		$1\ 3\ 1$
14	75	36	"		$1\ 3\ 2$
15	72	25	"	26	$1\ 2\ 1$
16	70	14	"		$2\ 3\ 1$
17	40	12	"		$2\ 1\ 1$
18	53	32	"		$1\ 1\ 1$
19	21	65	"	15	$1\ 0\ 1$
20	27	70	Lower-left		$2\ \bar{1}\ 3$
21	34	53	"	26	$1\ \bar{1}\ 2$
22	43	43	"		$1\ \bar{2}\ 3$
23	60	31	"		$0\ \bar{1}\ 1$
24	77	23	"		$\bar{1}\ \bar{3}\ 2$
25	87	19	"	26	$\bar{1}\ \bar{2}\ 1$
26	74	45	"		$\bar{1}\ \bar{1}\ 2$

2. *Determination of the Orientation.* It should be noted that the stereographic projection which is shown in Figure 2 gives a complete description of the orientation of the given crystal. The specimen is a single sodium wire (sealed in a capillary tube) and its axis is along the north - south direction of the projection.

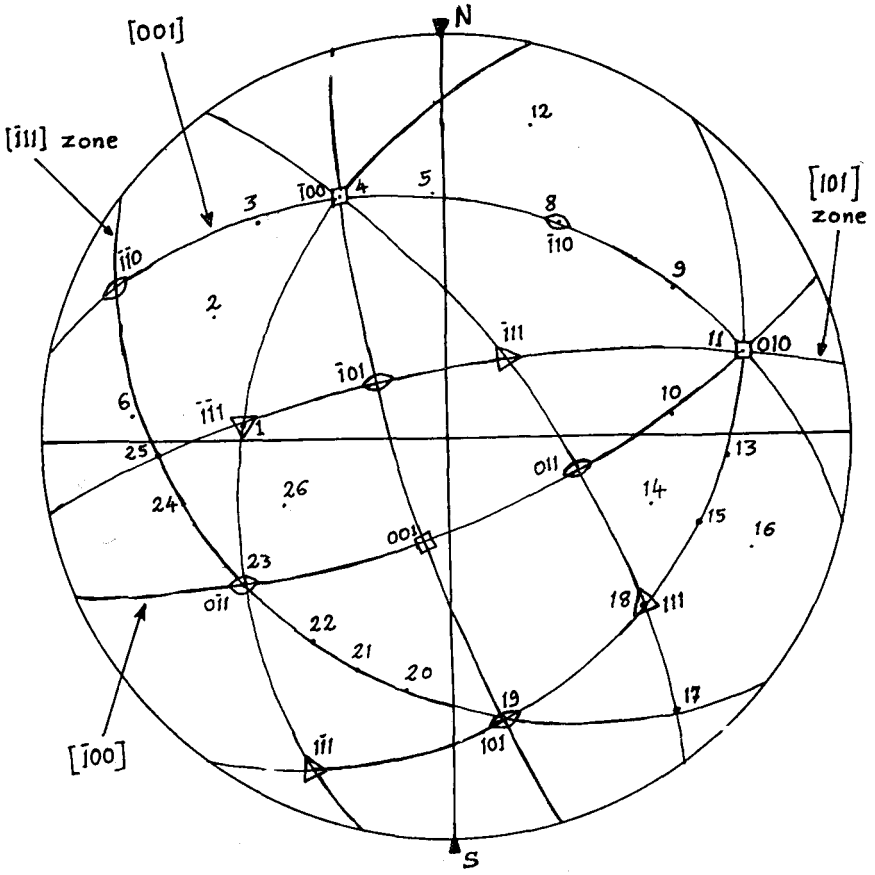

Figure 2. The stereographic projection showing the orientation of the specimen Na–49.

Since the reference sphere is covered by 48 spherical triangles which are all similar and equivalent to each other, and each one of them has <100>, <110> and <111> as its vertices [6], it is sufficient to consider only one of them and locate the orientations of all the specimens in one triangle which enables us to

study the kind of distribution for the orientation of the single crystals of each alkali metal.

III. EXPERIMENTAL PROCEDURE AND RESULTS

We first prepared a batch of single crystals of sodium consisting of specimens with approximately the same diameters (average diameter being $r^- = 0.10 \pm 0.01$ mm. and called Group I sodium crystals). We determined the orientations of all of them by the Laue method and placed them together in the same unit stereographic triangle. Next we prepared Group II ($r^- = 0.19 \pm 0.02$ mm), Group III ($r^- = 0.27 \pm 0.02$ mm.), Group IV ($r^- = 0.34 \pm 0.02$ mm), Group V ($r^- = 0.44 \pm 0.03$ mm), Group VI ($r^- = 0.49 \pm 0.03$ mm), and Group VII ($r^- = 0.60 \pm 0.04$ mm) sodium crystals, determined their orientations and found the distribution of orientations of each group of crystals. We finally plotted the orientations of all the sodium crystals in the same triangle shown in Figure 3.

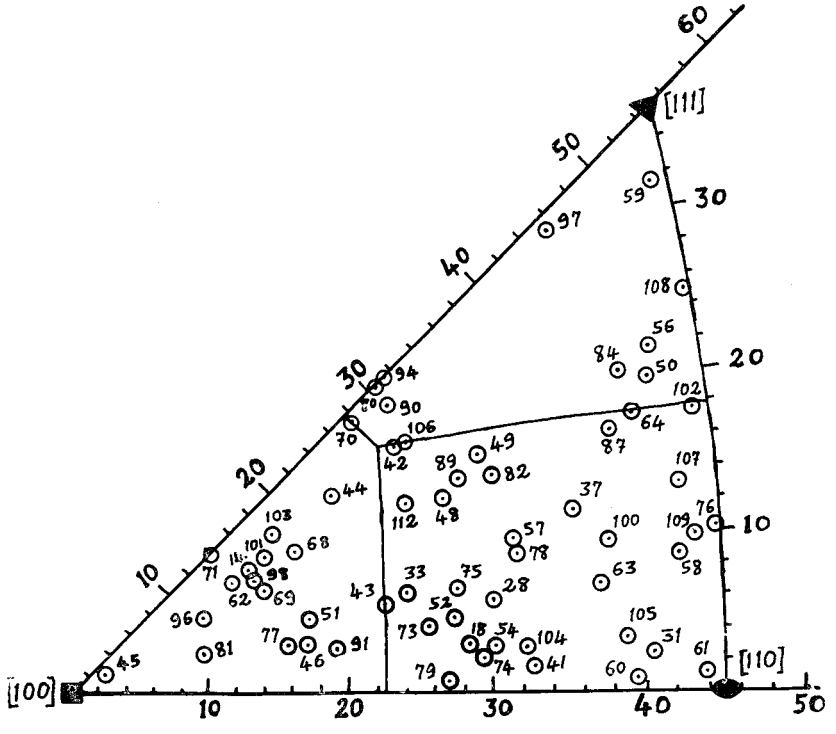

Figure 3. The distribution of orientations of Na grouped samples.

For lithium, Potassium, Rubidium and Caesium we produced two groups of crystals for each metal with two different diameters [7], approximately about 0.3 mm and 0.7 mm., both with wall thickness of nearly 0.06 mm. We determined first the orientation of each crystal and found the distribution of orientations for each group of crystals. Finally we plotted the orientations of all the crystals belonging to the same metal in the same triangle for Li, K, Rb and Cs as shown in Figure 4.

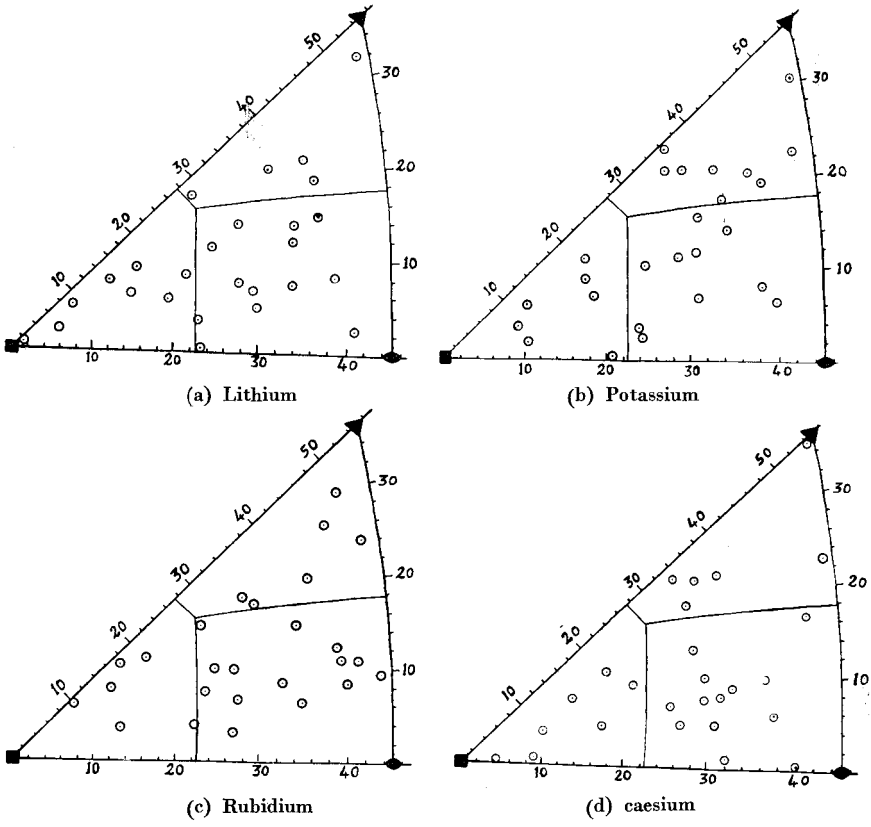

Figure 4. The distribution of orientations of Li, K, Rb and Cs grouped samples.

When we roughly examine the distribution of orientation of each group of crystals for every metal and finally that of the total number of crystals for each metal, we do not see an obvious in-

fluence of specimen size. It appears that the orientations are scattered randomly and we do not observe a marked concentration near any of the three crystallographic axes [100], [110], and [111], i. e., a preferred orientation does not appear from the experiments as was originally anticipated on account of the theory of crystal growth. In order to study the distribution of orientations mathematically, under the consideration of the forgoing rough observation, we made a hypothesis about random orientations.

In the literature [8], the statistical methods have been used for the treatment of the data about the orientations of single crystals. In a somewhat similar treatment we made use of χ^2 statistics, and Binomial distributions to test our hypothesis.

Let us consider again the [100], [110], and [111] triangles which can be divided into three different areas (or categories) such that when any orientation falls into one category it is clossest to one of the theree principal poles which is associated with this category. The areas of the categories are different.

On the surface of the reference sphere there are 6 of [100], 12 of [110], and 8 of [111] poles. Therefore in the triangle the relative area of the category which is associated with the pole [100] is 6/26 and those of the second and third categories are 12/26 and 8/26. It means, out of N orientations, $F_1 = 6/26\ N$ is expected to fall into the first category, and $F_2 = 12/26\ N$ and $F_3 = 8/26\ N$ are expected to fall into the second and third categories, respectively. F_1, F_2, and F_3 are called theoretical frequencies. Let f_1, f_2 and f_3 be the observed frequencies. Then the statistics that we shall use for testing the hypothesis is

$$\chi^2 = \sum_{i=1}^{k} \frac{(f_i - F_i)^2}{F_i}$$

where k is the number of categories.

Suppose we are given a distribution of $f_1 = 2$, $f_2 = 4$ and $f_3 = 2$ for which χ^2 is going to be 0.135. By using χ^2 tables [9], we find that the chance of χ^2 being less than 0.211 is 10 percent, being less than 4.61 is 90 percent and being less than 5.99 is 95 percent. Since χ^2 is less than 0.211 for our sample considered

above, we say we have sufficient reason to accept the hypothesis at the 90 percent level of significance. It is accepted that the hypothesis can be rejected when the level of significance (α) is 5 percent or below.

For some samples, some orientations lie very close to the border lines between categories. By considering various possibilities we obtained the listed values of χ^2 in Table II as maximum and minimum values for each sample.

We see from the χ^2 table that for only two cases our hypothesis seems to be rejectable at the high significant level. For the other cases the hypothesis of random eorientations is true at 10 percent level of significance. For some cases, the level of significance is much higher than 10 percent reaching 50 percent and even 90 percent.

Since, in some of the cases, there might be some doubt about accepting the random hypothesis, it is desirable to use some other method of statistical analysis, e.g., the Binomial distribution for which we can use the graphs given by Dixon and Massey [9]

In order to use the binomial distribution for testing our hpothesis, we must have two categories which will be referred here to as proportions of individuals. Let P denote one proportion of the individuals in the universe of orientations. Suppose we have a random sample of N orientations. The proportion P can be treated as a special case of mean value of the sample which is denoted by $\overline{X}$ and is equal to X/N.

As an example, let us take the rubidium sample Number I of size 13. Let us denote the value of the observed frequency associated with the direction [110] by X_{110} and it is equal to 7 in our sample.

Let P_{110} be the theoretical value of P which is equal to $12/26 = 0.46$. By making use of the graph with the confidence coefficient 0.90 and knowing the computed value of X_{110} to be equal to $7/13 = 0.54$, we can find the values of confidence limits from the vertical scale of the forgoing graph corresponding to the

TABLE II

The calculated values of χ^2 and α for various samples

Alkali Metal	Number of Groups	$\bar{r}$ (mm.)	N	Observed Frequencies f_1	f_2	f_3	χ^2.10 = 0.211 .50 = 1.386 .50 = 1.386 .95=5.991	α
Na	I	0.10 ∓ 0.01	11	2	8	1	3.53	0.10
				2	6	3	1.33	0.50
	II	0.19 ∓ 0.02	8	2	4	2	0.14	0.90
	III	0.27 ∓ 0.02	10	5	5	0	6.29	0.025
				4	4	2	1.72	0.10
	IV	0.34 ∓ 0.02	6	1	5	0	3.78	0.10
	V	0.44 ∓ 0.03	7	2	4	1	0.92	0.50
				2	3	2	0.12	0.90
	VI	0.49 ∓ 0.03	8	3	2	3	1.61	0.50
	VII	0.60 ∓ 0.04	11	2	7	2	1.44	0.10
	VIII		61	18	34	9	7.57	0.025
				17	30	14	2.04	0.10
Li	I	0.26 ∓ 0.02	13	4	6	3	0.583	0.50
				5	5	3	1.083	0.50
	II	0.72 ∓ 0.04	13	4	7	2	1.500	0.10
	III		26	8	13	5	1.875	0.10
				9	12	5	2.625	0.10
K	I	0.32 ∓ 0.02	13	3	5	5	0.417	0.50
				3	4	6	1.666	0.10
	II	0.65 ∓ 0.04	13	4	5	4	0.500	0.50
	III		26	7	10	9	0.625	0.50
				7	11	8	0.250	0.50
Rb	I	0.29 ∓ 0.02	13	2	7	4	0.500	0.50
	II	0.73 ∓ 0.04	13	4	7	2	1.500	0.10
				3	8	2	1.666	0.10
	III		26	6	14	6	0.833	0.50
				5	15	6	1.417	0.10
Cs	I	0.22 ∓ 0.02	13	4	6	3	0.583	0.50
	II	0.69 ∓ 0.04	13	3	7	3	0.417	0.50
	III		26	7	13	6	0.750	0.50

value of X_{110}/N. As shown in Figure 5, the confidence limits are calculated as 0.28 and 0.78. Since $P_{110} = 0.46$ falls into the interval of these confidence limits, we have no reason to reject the hypothesis of random orientation at 10 percent level of significance. In the same way we find that $P_{100} = 0.23$ and $P_{111} = 0.31$ fall into their corresponding intervals of confidence limits. We conclude from this result that our random hypothesis is acceptable at the 10 percent level of significance for this particular sample. The hypothesis has been tested in the same way for each sample plus the grouped samples and the result was shown in Table III.

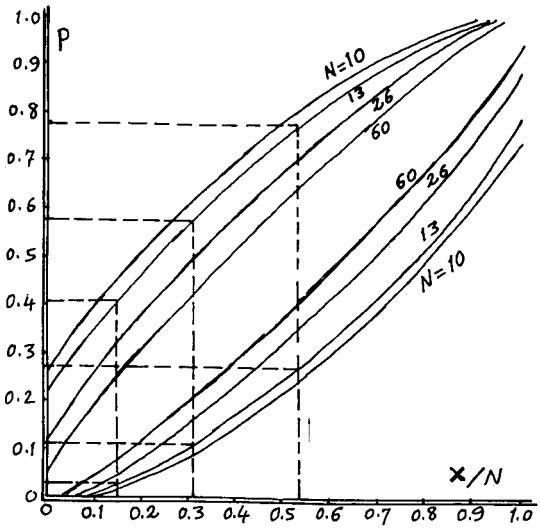

Figure 5. Confidence belts for proportions.

We observe from the above table that in a few cases the hypothesis is rejectable while in all other cases it is acceptable at the 10 percent level of significance and in most cases, at even higher than 10 percent.

IV. CONCLUSION

The study of distributions of orientations of alkali crystals grown in glass capillaries by using the Bridgeman technique

TABLE III Calculated Confidence Limits

Alkali Metal	Number of Gurop	N	X			$\overline{X} = X/N$			Calculated Confidence Limits for the 10 percent level of significance					
			X_{100}	X_{110}	X_{111}	$\frac{X_{100}}{N}$	$\frac{X_{110}}{N}$	$\frac{X_{111}}{N}$	$P_{100}=6/26$ $=0.23$		$P_{110}=12/26$ $=0.46$		$P_{111}=8/26$ $=0.31$	
Na	I	11	2	8	1	0.18	0.73	0.09	0.02	0.48	0.42	0.92	0.01	0.39
			2	6	3	0.18	0.55	0.27	0.02	0.48	0.26	0.82	0.07	0.58
	II	8	2	4	2	0.25	0.50	0.25	0.06	0.58	0.20	0.79	0.06	0.58
	III	10	5	5	0	0.50	0.50	0.00	0.20	0.79	0.20	0.79	0.00	0.28*
			4	4	2	0.40	0.40	0.20	0.15	0.70	0.15	0.70	0.03	0.51
	IV	6	1	5	0	0.17	0.83	0.00	0.01	0.60	0.40	0.98	0.00	0.42
	V	7	2	4	1	0.29	0.57	0.14	0.05	0.66	0.20	0.87	0.01	0.55
			2	3	2	0.29	0.42	0.29	0.05	0.66	0.12	0.77	0.05	0.66
	VI	8	3	2	3	0.37	0.26	0.37	0.10	0.70	0.07	0.06	0.10	0.70
	VII	11	2	7	2	0.18	0.64	0.13	0.02	0.48	0.36	0.87	0.02	0.48
	VIII	61	18	34	9	0.29	0.56	0.15	0.19	0.42	0.44	0.65	0.08	0.25*
			17	30	14	0.28	0.49	0.23	0.18	0.41	0.38	0.61	0.01	0.35
Li	I	13	4	6	3	0.31	0.46	0.33	0.11	0.58	0.21	0.72	0.07	0.50
			5	5	3	0.38	0.39	0.23	0.16	0.64	0.16	0.64	0.07	0.50
	II	13	4	7	2	0.31	0.54	0.15	0.11	0.58	0.28	0.78	0.02	0.41
	III	26	8	13	5	0.31	0.50	0.19	0.16	0.49	0.31	0.68	0.08	0.38
			9	12	5	0.35	0.46	0.19	0.19	0.54	0.28	0.64	0.08	0.38
K	I	13	3	5	5	0.23	0.38	0.39	0.07	0.05	0.16	0.64	0.16	0.64
			3	4	6	0.23	0.31	0.46	0.07	0.50	0.11	0.58	0.21	0.72
	II	13	4	5	4	0.31	0.38	0.31	0.11	0.58	0.16	0.64	0.11	0.58
	III	26	7	10	9	0.27	0.38	0.35	0.13	0.46	0.22	0.57	0.19	0.54
			7	11	8	0.27	0.42	0.31	0.13	0.46	0.25	0.61	0.16	0.49
Rb	I	13	2	7	4	0.15	0.54	0.31	0.02	0.41	0.28	0.78	0.11	0.58
	II	13	4	7	2	0.31	0.54	0.15	0.11	0.58	0.28	0.78	0.02	0.41
			3	8	2	0.23	0.62	0.15	0.07	0.50	0.36	0.85	0.02	0.41
	III	26	6	14	6	0.23	0.54	0.23	0.11	0.42	0.35	0.71	0.11	0.42
			5	15	6	0.19	0.58	0.23	0.08	0.38	0.39	0.01	1 70	5.42
Cs	I	13	4	6	3	0.31	0.46	0.23	0.11	0.58	0.21	0.72	0.07	0.50
	II	13	3	7	3	0.23	0.54	0.23	0.07	0.50	0.28	0.78	0.07	0.50
	III	26	7	13	6	0.27	0.50	0.23	0.13	0.46	0.32	0.68	0.11	0.42

without using a seed has shown that there is no obvious influence of crystal size on the orientations.

The idea of preferred orientation has been discussed. Because of the geometry and physical properties of the crystals, including the fact that in cubic crystals the crystallographic axes [100], [010] and [001], and similarly [110], [101] and [011] axes, are equivalent to each other, we might not expect preferred orientations. Our experimental work and statistical test support this theoretical view to a considerable extent.

ACKNOWLEDGMENT

The author wishes to thank Prof. Weymouth (University of Nebraska) and Prof. Perlitz for valuable suggestions and the Physics Department of the Science Faculty of Ankara University for giving the permission to do most of the experimental work in their X-ray diffraction laboratory.

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ÖZET

Bridgeman tekniği kullanılmak suretiyle, yarıçapları değişik ince kılcal cam borular içinde alkali metallerin tek kristalleri elde edilmiştir. Nüve kristal (tohum) kullanmaksızın, uçları kapalı tüpler içinde elde edilen tek kristallerin X-ışını Laue metodu yardımıyla yönelmeleri tayin edilmiştir. Bu şekilde elde edilen tek kristallerin yönelmelerinin teorik olarak gelişigüzel bir dağılım göstermesi beklenmektedir. Kristal büyümesi ile ilgili denel çalışmaların bazan bu sahadaki teorik çalışmalarla pek uyuşmadığı gerçeği göz önünde tutularak küp şeklindeki alkali metal kristallerinin bir tercihli yönelmeye sahip olup olmadıkları denel olarak araştırılmıştır. χ^2 analizi ve Binom dağılımı gibi statistik metotlar kullanarak alkali metallerin tek kristallerinin dağılımlarının teoride ön görüldüğü şekilde gelişigüzel olduğu ve denel sonuçların bunu büyük ölçüde desteklediği görülmüştür. Ayrıca, kristallerin yarıçaplarının yönelme üzerinde bir etkisi görülmemiştir.

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Prix de ce numéro : 5 TL (pour la vente en Turquie).

Prière de s'adresser pour l'abonnement à : Fen Fakültesi Dekanlığı Ankara, Turquie.

Ankara Üniversitesi Basımevi–1973