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AKTIEBOLAGET ATOMENERGI

STOCKHOLM · SWEDEN · 1960

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<u>Summary:</u>

Metallographic structure, microhardness and texture have been studied on various types of beryllium metal including hot pressed powder, a rolled strip and an extruded tube.

It was found that beryllium exhibits its highest hardness in directions perpendicular to the basal plane. Good ideas of the prevailing textures were obtained with an ordinary X-ray diffractometer.

Completion of manuscript in January 1960
Printed and distributed in May 1960

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1. Introduction

Facilities for the metallographic preparation of beryllium have recently been obtained at the Atomic Energy Company (1). As a starting application for this equipment some preliminary studies on microstructure, microhardness and texture have been made on various types of beryllium metal.

2. Experimental

The following test materials were used:

A. Metal from the beryllium reflector in the materials testing reactor R2, probably hot pressed powder.

B. Strip of hot rolled beryllium with cross section 1 x 10 mm, obtained from Pechiney.

C. Hot extruded tube, 9.5 mm bore and 1 mm wall thickness, obtained from Tube Investment Co. The reduction ratio is stated to be 12.4:1.

D. Electrolytic flake from Pechiney.

The drybox used for metallographic preparation was divided into two compartments, for grinding and for mechanical polishing respectively. In the compartment for grinding, specimens were also cut from the bulk material with a hack saw. The specimens were mounted in an epoxy resin and the sawed surface was wet ground on successively finer silicon carbide papers with paper no 600 as the final step. The ground surface was polished on Alumina-2 and Alumina-3. In some cases attack polishing was employed using a suspension of Alumina-3 in 3 % oxalic acid. As this procedure often caused excessive pitting, it was sometimes followed by normal polishing on Alumina-3. Usually no etching was required to bring out the grains which because of the hexagonal close packed lattice of beryllium could be clearly seen in polarized light. In some cases etching in a solution of 5 % HF in ethyl alcohol was tried with no advantage since this etch often resulted in severe pitting.

The size and shape of the grains were examined under the microscope using polarized light whereas the occurrence of oxide inclusions was studied in unpolarized light. It proved difficult, however, to distinguish between inclusions and etch pits due to attack from water during the specimen preparation.

Microhardness was measured with a Bergsman microhardness tester using a 10 g or 50 g weight (2).

Texture was studied by recording the intensities of diffracted X-rays (CuKα radiation) from crystallographic planes parallell to the specimen surface using a Philips X-ray diffractometer. By comparing the results for specimen surfaces differently oriented relative to a suitable axis in the original beryllium piece, it was possible to get a rather accurate concept of the prevailing texture. In order to account for the effect of different sizes of the exposed specimen surfaces the recorded intensities were normalized so that the sum of the intensities of all reflections was the same for different specimens. The intensities were also compared with the theoretically calculated intensities of a textureless sample (3).

While examining the specimen under polarized light the angle between the nicols was always kept the same. When turning the specimen, the intensity of the light reflected from a given grain varied so that it appeared dark in a certain position and then continuously shifted to bright appearance. Some grains, however, remained more or less grey during the specimen revolution. The surface of such a grain was probably closely parallell to the crystallographic basal plane (0001) which should exhibit optic isotropy. With the specimen in a given position several hardness impressions were made in dark, bright and grey grains. The latter in general proved to be harder than the bright and dark ones. Since some grains may appear grey owing to an angular position intermediate to those corresponding to dark and bright reflection it could also be explained why not all grey grains exhibited higher hardness values. Hence, it was concluded that the high hardness values were characteristic of grains having (0001) parallell to the specimen surface. This conclusion was verified by the subsequent X-ray study that provided information on the prevalence of grains having this orientation.

3. Results

A. Beryllium from the R2 reflector

The R2 beryllium reflector was found to consist of equiaxed grains with an average diameter of 15 to 20 microns. This is illustrated in fig. 1. Small twins were quite frequent, probably as a result of deformation during hot pressing. In some cases growth of twins was evident. Sections having different orientations exhibited similar structures.

Figs. 2 and 3 show the same area in polarized and normal light respectively. As could be seen there is no obious concentration of inclusions at grain boundaries. The additional black dots that appear in fig. 2 are probably due to pits that appear more readily under polarized light.

The results of hardness measurements are shown in table 1. The higher hardness level of the grey grains is obvious.

Fig. 4 b shows the relative intensities of reflections from lattice planes parallell to the specimen surface obtained in X-ray diffraction. The relative intensities deviate slightly from those theoretically calculated for a textureless sample as shown in fig. 4 a. Thus the reflection 002 is more intense than for a textureless sample indicating a slight overrepresentation of grains having (0001) parallell to the selected specimen surface.

B. Beryllium strip from Pechiney

The average grain diameter for the beryllium strip was 80 to 100 microns. Figs. 5 and 6 show the grain structure in a longitudinal section perpendicular to the rolling plane and in a transverse section. It proved difficult to avoid scratches when preparing this material and the specimen preparation is not satisfactory. In particular this concerns the transverse section which, according to hardness measurements, was found to be softer than sections with other orientations. As far as could be observed twins were essentially absent. This possibly indicates a high working temperature. The specimen preparation did not permit a study of inclusions.

Hardness measurements for longitudinal sections perpendicular and parallell to the rolling plane as well as for the transverse section are reported in Table I. Grey surfaces generally display higher values than bright or dark ones, except for the transverse section. This indicates the absence of grains having (0001) parallell to the transverse section.

Fig. 7 b, c, and d show the corresponding X-ray results. The 002 reflection is missing for the transverse section (d), very strong for the flat surface (c) of the strip, and rather strong for the longitudinal section normal to this surface (b). Furthermore the intensity of the 100 reflection is considerably stronger for the transverse section than for the other sections. This implies that the crystals are oriented with $<10\bar{1}0>$ parallell to the longitudinal direction, i.e. the basal plane (0001) is parallell and the prism plane $\{1010\}$ is perpendicular to this direction. The [0001] directions of the crystals tend to be normal to the flat surface although a spread of these directions in the transverse plane is obvious. The texture thus inferred is schematically indicated in fig. 8.

C. Hot extruded tube

The hot extruded tube proved to be very brittle. Thus when specimens were cut off with a hack saw the tube fractured longitudinally. However, the fracture was observed to be preceeded by slight plastic deformation.

Fig. 9 shows the fibrous structure of an axial section through the tube. The grains are slightly elongated as seen in fig. 10 and the average grain diameter is 15 to 20 microns. The structure of a tangential section had similar appearance. The transverse section displayed no directional structure and the grains seemed equiaxed with an average grain diameter of 10 microns. Twins were seldom observed. Also for this material it was difficult to distinguish between possible inclusions and etch pits.

Hardness values for axial, tangential and transverse sections are given in Table I. In the axial and tangential sections some grey grains displayed high hardness values suggesting that their surfaces were closely parallell to (0001). In the transverse sections no such high hardness values were encountered.

The X-ray results for the three mutually perpendicular sections are shown in figs. 11 b, c and d. For the transverse section reflections from the basal plane 002 and 004 are missing whereas 100 and 200 reflections from $\{10\bar{1}0\}$ prism planes are quite strong. For the axial and tangential sections the opposite is true. Hence the typical texture after hot extrusion with $<10\bar{1}0>$ parallell to the extrusion direction is verified. Also for the axial section the low intensities of reflections of type (h 01) from pyramidal surfaces $(h\ 0\ \bar{h}\ 1)$ are in agreement with a $<10\bar{1}0>$ texture. A close analysis of the X-ray intensities results in the schematic representation of the texture shown in fig. 12. The results are in agreement with published data (3) on hot extruded tubes.

D. Electrolytic flake

The flakes had an average diameter of 5 and a thickness of about 0.1 mm. Microscopic investigation of metallographically prepared surfaces revealed that the flake in general was a single crystal since the intensity of polarized light was the same over its whole section. Microhardness measurements also yielded very similar values over the section.

4. Conclusions

The preliminary investigations reported here have been conducted with the main purpose of testing the drybox equipment for metallographic preparation of beryllium. The preparation technique permits the observation of grains in polarized light but at present it is not satisfactory for the study of inclusions. The technique could probably be developed considerably. Microhardness measurements have clearly demonstrated the anisotropy of beryllium and it has been established that the basal plane (0001) displays higher hardness values than other crystallographic planes. By means of an ordinary X-ray diffractometer good concepts of the textures of a strip and a tube of beryllium have been obtained. In both cases a strong fiber texture with $<10\bar{1}0>$ in the working direction was found in agreement with the literature.

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Table 1

Micro hardness values

Material	Section	Grain	Microhardness Vickers			Number of impressions
			Max	Min	Average	
A	random	DB	254	171	204	9
		G	332	274	300	4
B	longitudinal, parallell to rolling plane	DB	222	147	175	17
		G	406	222	312	9
	longitudinal, normal to rolling plane	DB	371	176	237	21
		G	349	236	284	9
	transverse	DB	188	157	166	10
		G	188	145	164	5
C	axial	DB	285	190	236	12
		G	361	252	322	3
	tangential	DB	271	187	229	5
		G	342	225	300	5
	transverse	DB	256	170	226	7
		G	252	182	214	4

A = R 2 reflector, hot pressed powder

B = rolled strip

C = extruded tube

DB = dark and bright grains

G = grey grains

Fig. 1
R 2 reflector.
Etched in 5 % HF,
polarized light.

150 x

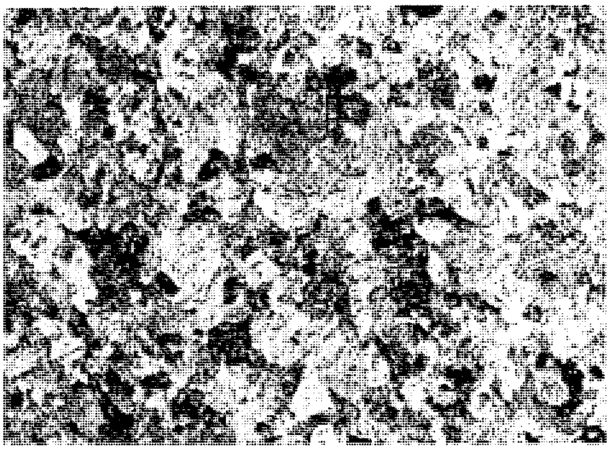


Fig. 2
R 2 reflector.
Unetched,
polarized light

800 x

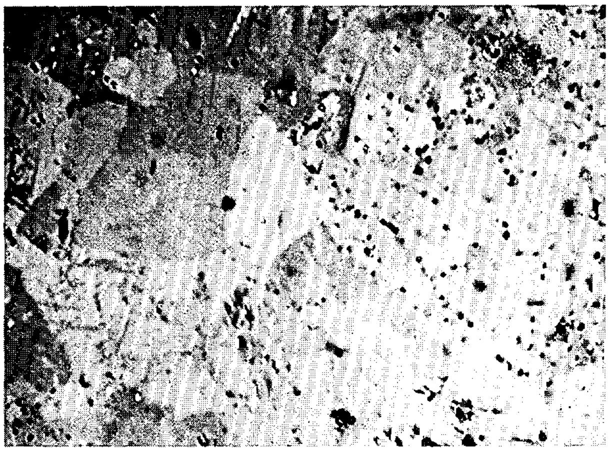
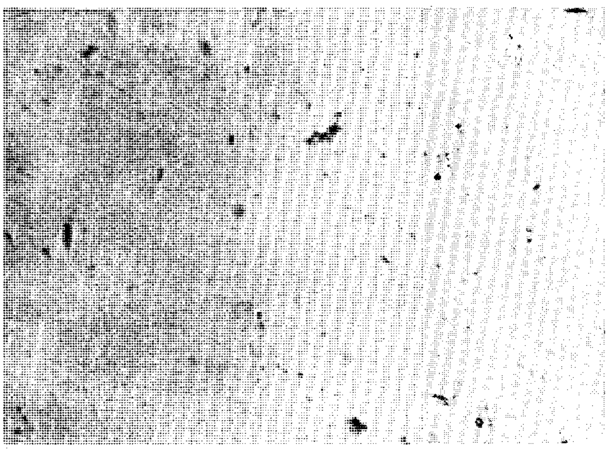


Fig. 3
R 2 reflector.
Unetched,
normal light.

800 x



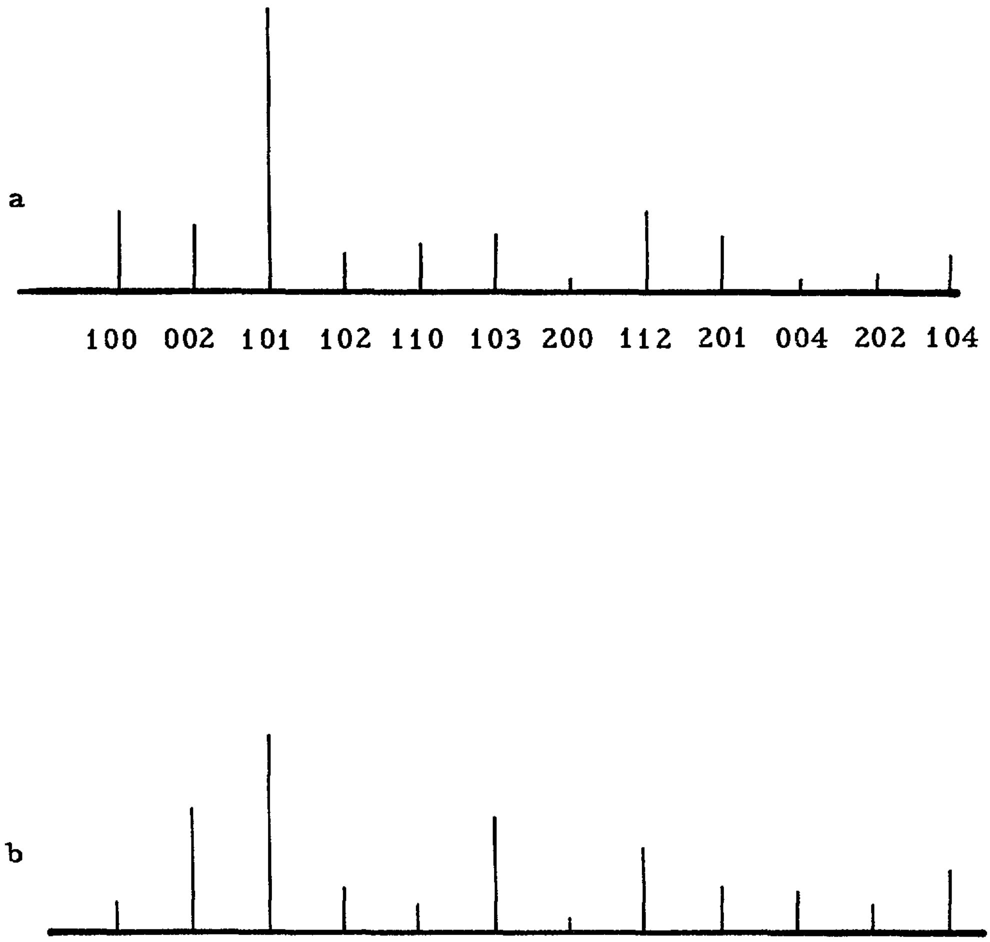


Fig. 4 R2 Reflector
Relative X-ray intensities for lattice planes parallell to specimen surface

a. Calculated for textureless sample
b. Random section through R2 reflector

Fig. 5

Beryllium strip.
Section in rolling direction normal to rolling plane.
Unetched,
polarized light.

150 x

Fig. 6

Beryllium strip.
Transverse section.
Unetched,
polarized light.

150 x

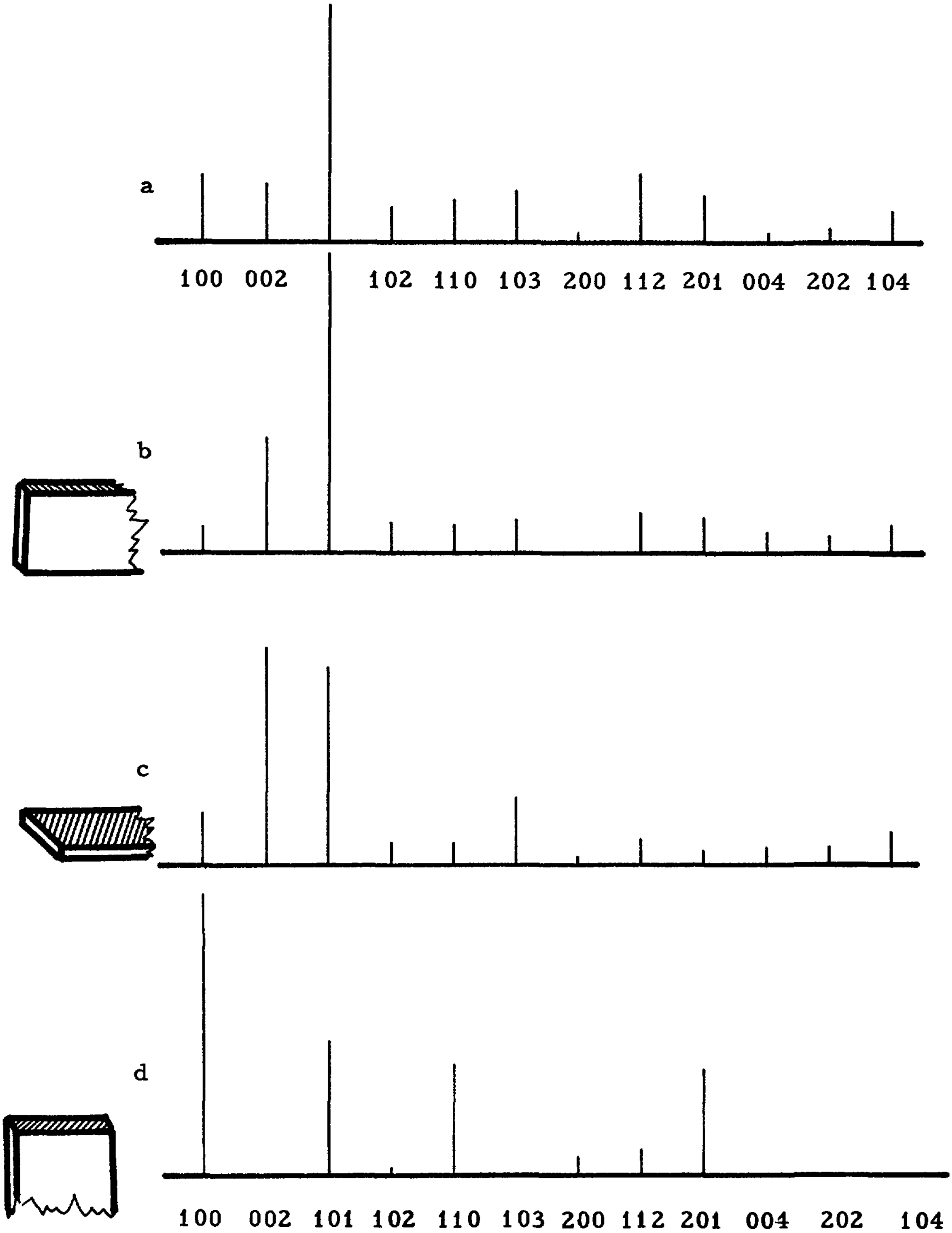


Fig. 7 Beryllium strip
Relative X-ray intensities for lattice planes parallell to specimen surface

a. Calculated for textureless sample
b. Beryllium strip. Longitudinal section normal to rolling plane
c. " " " " parallell " " "
d. " " Transverse section.

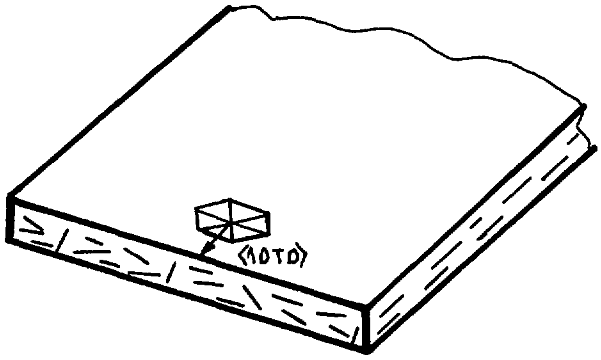


Fig. 8 Basal plane projections in beryllium strip

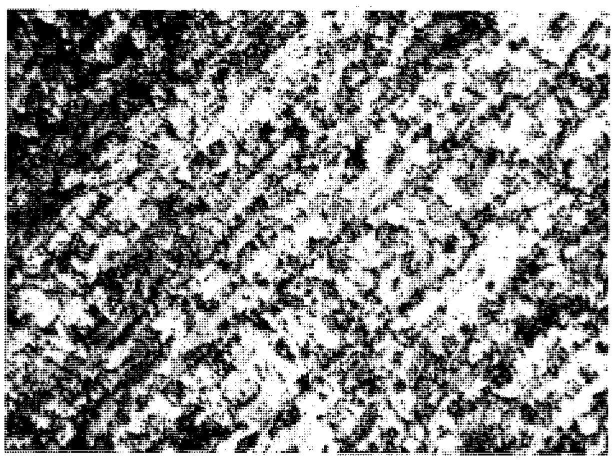


Fig. 9
Extruded tube,
axial section.
Attack polished,
polarized light.

150 x

Fig. 10
Extruded tube,
axial section.
Attack polished,
polarized light.

500 x

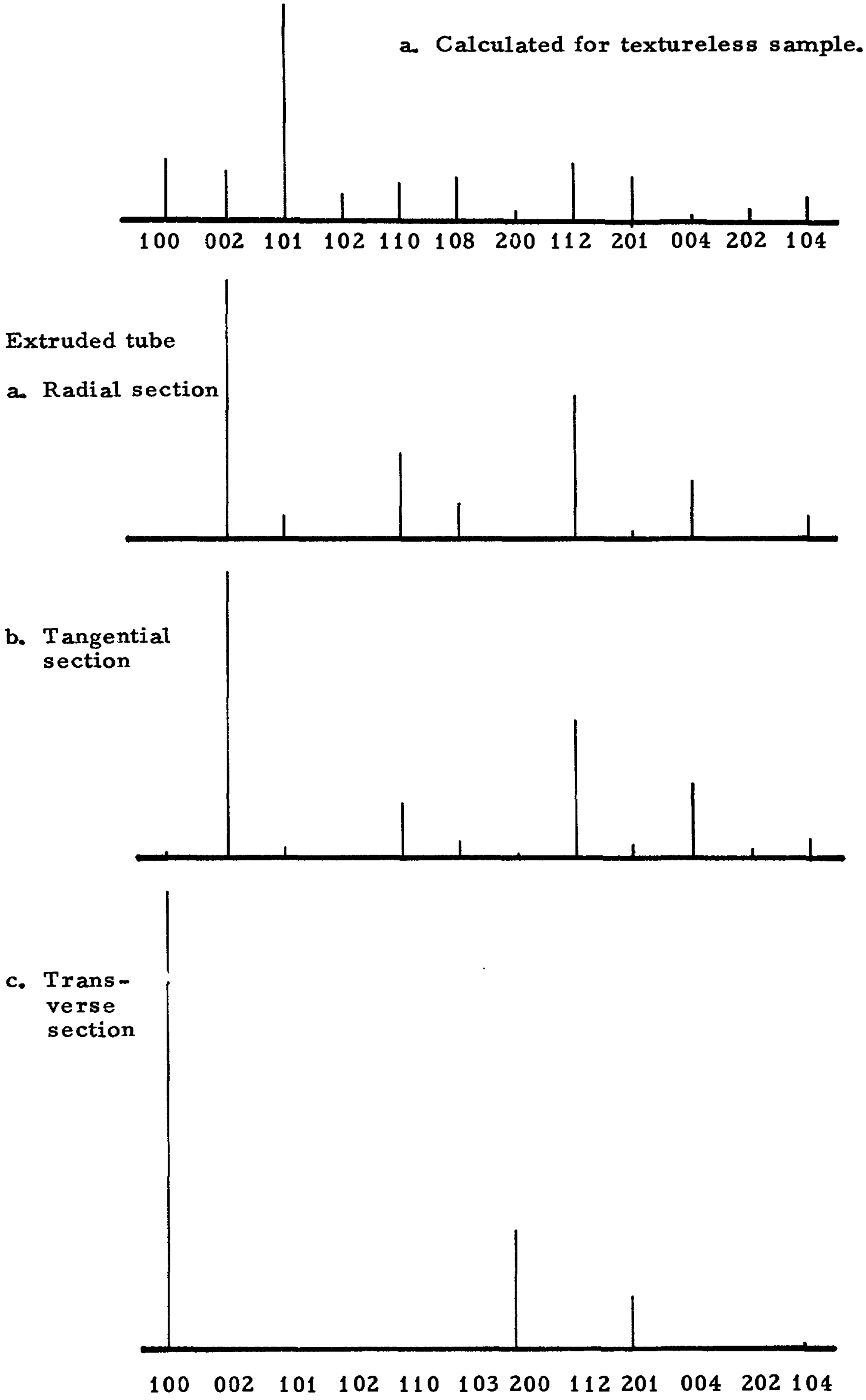


Fig. 11 Extruded tube
Relative X-ray intensities for lattice planes parallell to specimen surface

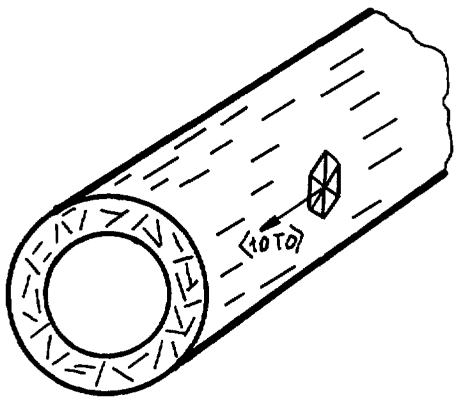


Fig. 12 Basal plane projections in hot extruded beryllium tube

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