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BERYLLIUM RESEARCH AND DEVELOPMENT PROGRAM

INFLUENCE OF THE DISTRIBUTION OF OXIDE AND OF THE TOTAL IMPURITY LEVEL ON RECRYSTALLIZATION AND GRAIN GROWTH OF BERYLLIUM

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PECHINEY COMPANY

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FOREWORD

This report was prepared under USAF Contract No. AF 33(616)-7065 with Nuclear Metals, Inc., West Concord, Massachusetts, as the prime contractor. The contract was initiated under Project No. 7351, "Metallic Materials," Task No. 735104, "Beryllium and Beryllium Alloys." The work was administered under the direction of the Metals and Ceramics Division, Air Force Materials Laboratory, Research and Technology Division, with Mr. K. L. Kojola and Capt. P. S. Duletsky acting as project engineers.

The portion of the work covered by this volume was performed under Subcontract No. 10a, "Influence of the Distribution of Oxide and of the Total Impurity Level on Recrystallisation and Grain Growth of Beryllium," by the Pechiney Company, Chambery, France. The authors of this volume are J. Moriceau, J. M. Logerot, M. Croutseilles, A. Saulnier, R. Syre, and P. Vachet of Pechiney Company.

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ABSTRACT

The objective of this program was to study the influence of distribution of oxide and of the total impurity level on the recrystallisation and grain growth of beryllium.

The starting material was either Pechiney SR beryllium or Brush QMV beryllium. Sheets of 1 - 1.5 mm thickness were fabricated by forging and hot rolling, within cans, of cast billets or billets compacted from powder of either -50, +110 mesh or -200 mesh. The sheets were then warm rolled (700°C) to reductions of approximately 50, 100, 180, and 250%. The feasibility of warm rolling beryllium varies in inverse ratio to the total impurity level and the oxide content.

The temperatures of recrystallisation, TR, as defined by X-ray diffraction, decreased with the amount of reduction and varied from 700 - 730°C on the average for SR cast metal, to 760 - 765°C for Brush cast metal, to 800 - 810°C for the powder metallurgy materials. Examinations by optical and electron microscopy were carried out on all the materials after different stages of processing and on the worked sheets after heat treatments in the recovery (one hour at TR -100), recrystallisation (one hour and ten hours at TR), and at grain growth (one hour and 100 hours at TR +200) temperatures. In addition, observations with optical and electron microscopes at temperature made it possible to obtain more precise information on the process of recrystallization.

In the as-cast condition, Brush beryllium can be distinguished from SR by a finer grain size and numerous inclusions. Forging and hot rolling leads to a strong deformation of the grains. In this condition, the SR metal is recrystallized while Brush metal remains worked. Warm rolling decreases this difference. The worked state is characterised by some grains which are slightly deformed, surrounded by a mass of highly distorted material, particularly in the case of Brush metal. The slightly deformed grains consist of slightly disoriented sub-grains containing a small number of dislocations. Between these grains is a confused region in which deformed blocks can be distinguished in the middle of dense veins of dislocations.

The recrystallization of such a structure results from the competition of two processes: on the one hand, the reorganizations in situ of the lightly deformed grains by the elimination of dislocations and the coalescence of subgrains; on the other hand, the appearance of nuclei at the expense of these large grains. This latter process appears preponderantly in the Brush metal. At equal levels of deformation, grain size is finer for Brush metal than for SR. Recovery leads to a more or less complete rearrangement of the substructure. At high temperature, grain growth occurs, the rate of growth being greater the purer the metal.

In the metal of powder origin, the continuous oxide film which surrounds each grain of powder is entirely destroyed by forging and hot rolling, and is broken into particles of 1/100 to several tenths of a micron dispersed in layers parallel to the surface of the sheet. These particles are smaller and more numerous for the -200 mesh powders. These oxide layers no longer constitute insurmountable obstacles for the grain boundaries as shown by the fact

that the powdered and rolled Brush metal exhibits an abnormal grain size comparable to that of cast metal. The SR powder materials, by contrast, preserve a fine grain size throughout the fabrication operations.

These structural differences are found again after warm rolling. During recrystallization, powdered Brush metal changes in the same manner as cast metal. During recrystallization of the powdered SR materials, nuclei appear which grow rapidly to approximately a 20-micron size. In both cases, prolonged heating at high temperature causes only a slight grain growth.

Mechanical properties during tension and bending have been measured on the entire collection of fabricated products and annealed sheets. In the forged state, the powder metals have better mechanical properties than the cast metal, which is fragile. Hot rolling improves the properties of cast metal but lowers slightly those of powdered SR and causes powdered Brush metal to become very fragile because of abnormal grain growth. After working, the best combinations of mechanical properties are obtained in the recrystallized conditions. Recovery treatments have given good results only for Brush cast metal. With regard to grain growth treatments, these seem to improve the bendability of cast metal, but in all cases are catastrophic for the tensile properties. For the SR metal, the mechanical properties improve as the grain size becomes smaller. Such a correlation has not been found for Brush metal of this type.

The following points come out of this study:

(a) Increase of the impurity content in beryllium raises the recrystallization temperature and decreases the tendency for grain growth by restraining the movement of grain boundaries.

(b) At high levels of deformation, the distribution of oxide does not seem to be fundamentally different for the material originating from the different types of powder.

(c) The layers of oxide effectively restrain the motion of grain boundaries to the point of making grain growth negligible but do not always constitute an insurmountable barrier for these boundaries.

(d) In all cases, the conditions of fabrication play a fundamental role in the subsequent behavior of the metal, especially with respect to the mechanical properties.

This technical documentary report has been reviewed and is approved.

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I. INTRODUCTION

The objective of this program was to study:

(a) The distribution of oxide in sheets of different metallurgical origin.

(b) The influence of this distribution and of the total level of impurities on recrystallisation and grain growth of metal during various heat treatments.

The metal used in this study had two origins:

(a) Flakes from electrolytic refining (Pechiney SR).

(b) "Lumps" of commercial quality (Brush QMV).

The consolidation and the fabrication of these two qualities of metal were carried out under as nearly similar conditions as possible using forging and crossed hot rolling of billets from castings or powder compacts (-50, +110 mesh and -200 mesh). Starting with these two qualities of metals and three types of structure, a series of treatments have been applied to the sheet. These treatments consisted of work-recovery, work-recrystallisation, and work-grain growth. The different stages of fabrication which are summarised in Table 1 are presented in Section II of this report.

A detailed metallographic study using X-ray diffraction, optical microscopy at room temperature and at elevated temperature, and electron microscopy was carried out on the metal at all stages of fabrication and after the various treatments (Section III). Finally, the mechanical properties of the metal were measured at room temperature by means of tensile and bend tests (Section IV). The results of these tests will be discussed in Section V.

II. PROCESSING OF WROUGHT SHEET AND THERMAL TREATMENTS

We shall successively examine the different stages of fabrication of wrought sheet. Tables 1 and 2 summarise the operations carried out and present the sample numbering system which was adopted. The starting material consists of:

(a) Pechiney SR from two lots of flake chosen for their excellent purity and similarity.

(b) Brush QMV from a lot of 8 kg of "lumps" furnished by Nuclear Metals, Inc.

Analyses of these materials are given in Table 3.

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TABLE 1. PROCESSING OF SHEETS

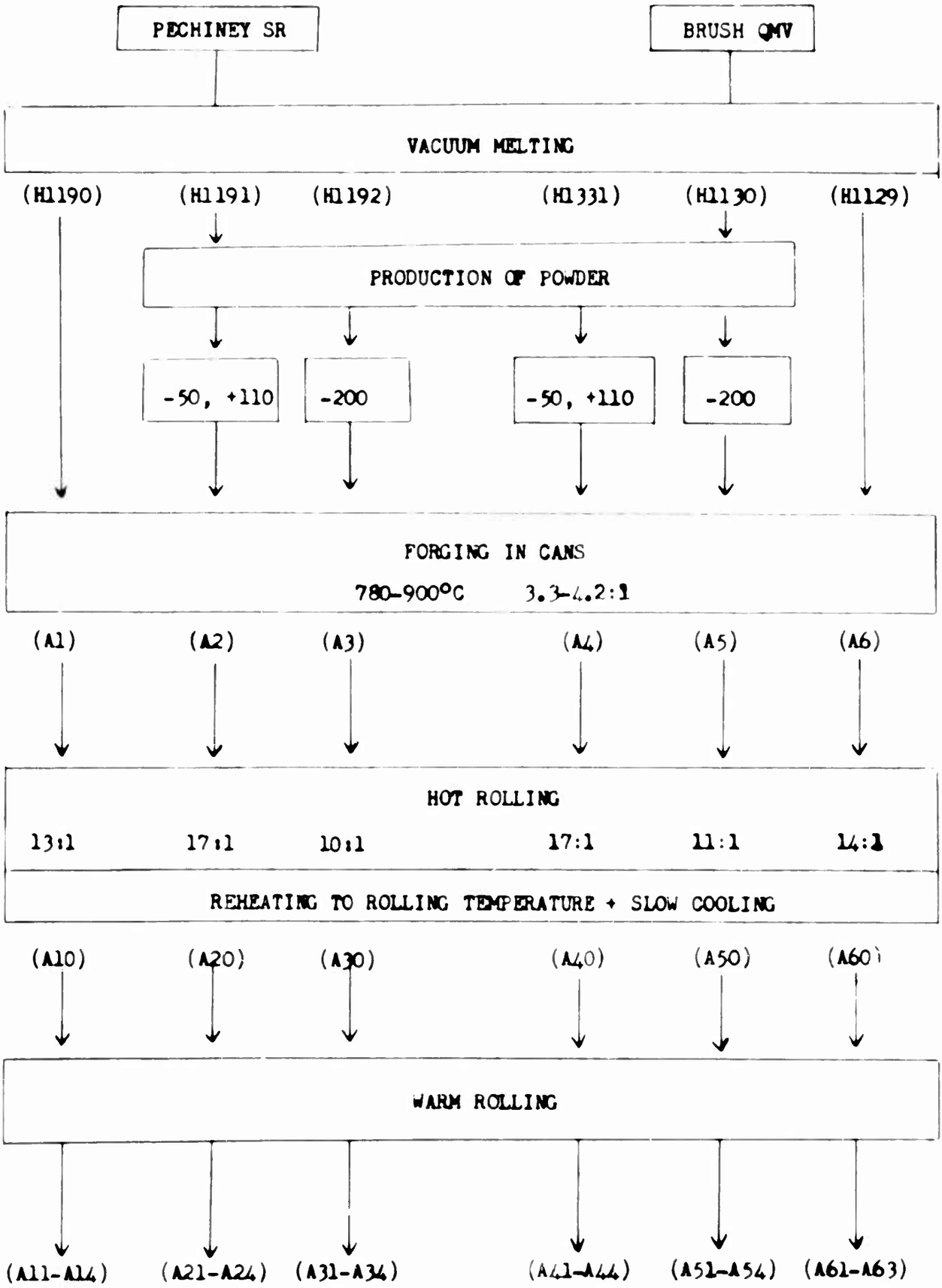


TABLE 2. NUMBERING OF WROUGHT SHEETS

Quality	Type	Number	Total Reduction $100(t_o-t)/t$ %
PECHINEY SR	Cast	A 10*	0
		A 11	50
		A 12	100
		A 13	180
		A 14	255
	Powder -50, +110	A 20*	0
		A 21	8
		A 22	50
		A 23	80
		A 24	235
	Powder -200	A 30*	0
		A 31	50
		A 32	100
		A 33	160
		A 34	225
BRUSH QMV	Cast	A 40*	0
		A 41	50
		A 42	100
		A 43	180
		A 44	245
	Powder -50, +110	A 50*	0
		A 51	50
		A 52	100
		A 53	180
		A 54	250
	Powder -200	A 60*	0
		A 61	50
		A 62	100
		A 63	140

* Hot rolled sheets

TABLE 3. ANALYSES OF MATERIALS

Origin of Metal	Type of Specimen	Number	BeO	Nonmetallic Impurities (ppm)					Metallic Impurities (ppm)													
				C	Cl	N	B	Total*	Fe	Al	Ni	Si	Cr	Mg	Cu	Mn	Ca	Na	Zn	Ti	Mo	Total
Pechiney SR	Flake	Lot 87-89	600	135	410	-	1	< 1000	<10	<30	<30	10	< 2	<10	<5	< 2	<30	150	26	< 2	-	<350
	Cast Ingot	H1190	(1200)	250	<20	-	1	< 1100	35	<30	64	35	<10	25	32	<5	50	<100	<80	<25		<550
	Plate From Forged Casting	H1190	600																			
	Hot Rolled Sheet	A10	1000			35																
	Powder (-50, +110)	A19 SR	2000	250	<20	-	1	< 1700	60	65	44	25	12	36	5	<5	45	<100	<80	<25	<15	<520
	Plate From Forged Powder	A19 SR	3100																			
	Hot Rolled Plate (8% Warm Rolled)	A21	(7600)			95																
	Powder (-200)	A18 SR	(13600)	300	<20		1	< 9000	120	65	50	75	11	60	23	<5	50	<100	<80	<25	<15	<680
	Plate From Forged Powder	A18 SR	11800																			
	Hot Rolled Sheet (52% Warm Rolled)	A31	12600			340																

TABLE 3. ANALYSES OF MATERIALS (Continued)

Origin of Metal	Type of Specimen	Number	BeO	Nonmetallic Impurities (ppm)					Metallic Impurities (ppm)													
				C	Cl	N	B	Total*	Fe	Al	Ni	Si	Cr	Mg	Cu	Mn	Ca	Na	Zn	Ti	Mo	Total
Brush QMV	Lumps	-	4000	-	<20		1	<3500	1180	950	135	400	85	230	80	80	80	<100	<80	-	<15	<3400
	Cast Ingot	H1129	4600	490	<20		1	<3600	1090	760	125	380	95	70	60	80	<30	<100	<80	150	<15	<3100
	Hot Rolled Sheet	A40	5500	330	<20		1	<4000	1180	850	140	400	95	25	60	100	35	<100	<80	160	<15	<3200
	Warm Rolled Sheet	A41-A44	5300			110																
	Powder (-50, +110)	A5	(8900)	310			1	<6000	1200	750	115	350	75	20	38	70	45	<100	<80	160	<15	<3000
	Hot Rolled Sheet	A50	5000			140																
	Warm Rolled Sheet	A536							1250	620	160	-	130	-	40	80	-	-	-	220		
	Powder (-200)	A6	(17700)	290			1	<12000	1300	950	110	350	75	18	38	70	50	<100	<80	265	<15	<3400
	Hot Rolled Sheet	A60	15000			210																

*(O + C + Cl + N)

Note: The values of BeO between ( ) are too large.

A. Vacuum Melting

For each type of material, three castings were made under the following conditions:

(a) Vacuum melting of 10^{-4} mm of Hg in BeO crucibles.

(b) Cast under argon (50 mm) into graphite mold (97 mm diameter).

(c) Casting temperature 1400 - 1450°C, cooled under vacuum.

After cropping, the ingots are machined to a depth of 2 mm and then examined by radiography and chemical analysis. Radiography makes several notable differences apparent:

(a) Stronger absorption of X-rays by QMV metal.

(b) Extensive pipe which is prolonged by cracks toward the bottom of the ingot in the less pure metal.

For each type of material, an ingot was chosen for direct fabrication and a clean piece was taken and saved, the remainder of the material being destined for the production of powder. The fabricated billets had a weight of about 1-1/2 kg.

B. Production of Powder

The cast ingots were turned, under an argon blanket, into chips 0.1 to 0.15 mm thick and 1 to 3 mm long. These chips were then ground under argon in a slow grinder of beryllium with beryllium balls. The powder was removed periodically and manually screened, always under argon, on screens of stainless steel. For each type of material, two fractions of particle size (-50, +110 mesh and -200 mesh) have been obtained by successive removal. The total grinding time was 24 and 200 hours, respectively. The distribution of sizes of powder evaluated at the time of opening of the screens can be estimated as:

(a) -50, +110 fraction: 145 to 360 microns, average 260 microns.

(b) -200 fraction: less than 80 microns, average 65 microns, 15 to 20% smaller than 35 microns.

C. Hot Compaction of Powder

The four lots of powder were compacted by cold pressing, then pressing under argon in a can of soft steel. This operation is carried out in the container of an extrusion press preheated to 450°C under a pressure of 113 kg/mm^2 at 950°C with the pressure being maintained from 20 to 30 seconds. The billets are cooled in air, then decanned and machined to the largest possible size (about 90 mm diameter). Density has been evaluated to be 1.84 ± 0.01 by means of immersion in a dense liquid.

D. Forging in a Can

The billets are first canned in semi-hard steel. Forging consists of upsetting at low speed between the preheated plates of a vertical press under the conditions shown in Table 4. The total reductions obtained and the maximum pressures calculated on the final surface areas are shown in Table 5.

Deformation appears to be more difficult and requires higher pressures for all QMV billets and for the SR -200 mesh billets in Table 5. This behavior can be attributed to the decrease of grain size and the increase in the level of BeO.

The forged plates were examined macroscopically and radiographically.

1. Macro Examination

The visual appearance is approximately the same for each type of plate from both kinds of material. The plates forged from castings had coarser grains with a columnar structure. The plates from forged powder had a relatively homogeneous grain size which was clearly smaller for the -200 mesh material by a ratio of 6:1.

2. Radiography

The observations were as follows:

(a) Mottled aspect on the entire surface of the forged cast plates as a result of diffraction of X-rays by the coarse grains.

(b) Homogeneous appearance of the plates from forged powder but with the presence of inclusions of high absorption whose size was proportional to the size of the powder. In plate A.3 (-200 mesh SR powder), some large inclusions of extraneous origins were marked and subsequently eliminated.

E. Hot Rolling

Hot rolling is carried out with a can under the conditions resulting from previous experience on the commercial Pechiney metal. The can, of the "picture frame" type, is composed of a frame of soft steel machined to the dimensions of the plate, of two sheets of 18-8 stainless steel of 10 mm thickness, and of two soft steel covers welded in place. Rolling is carried out in a Duo rolling mill with rolls preheated to 80°C and with a rolling speed of 25 meters per minute. The passes are 10 to 20% ($100(t_o-t)/t$) between two reheats in an electric furnace and each pass is at 90° to the preceding one. The rolling conditions have been adapted to the dimensions and to the quality of each plate to obtain a sound isotropic sheet of 400 x 400 mm and of a thickness of 1 to 1.5 mm. After the last pass, the sheets are reheated to the temperature of rolling, then rapidly sheared and cooled slowly before being decanned. The conditions are given in Table 6. The maximum temperature is that which existed upon removal from the furnace; the minimum temperature is that measured upon exit from the rolls.

TABLE 4. FORGING CONDITIONS

	SR	QMV
Preheat Temperature	900°C	900°C
Temperature of the Tools	450 - 500°C	450 - 500°C
Temperature at the End of Forging	780°C	750°C

TABLE 5. FORGING REDUCTIONS AND PRESSURES

Origin	Number	Reduction $(\frac{\text{Final Diameter}}{\text{Initial Diameter}})^2$	Pressure (Kg/mm^2)
Pechiney SR	A.1 (cast)	4.2:1	23
	A.2 (powder -50, +110)	3.7:1	27
	A.3 (powder -200)	3.3:1	32
Brush QMV	A.4 (cast)	3.4:1	27
	A.5 (powder -50, +110)	3.6:1	29
	A.6 (powder -200)	3.3:1	32

TABLE 6. HOT ROLLING CONDITIONS

Origin	Number	Rolling Temperature °C	Total Reduction	
			Rolling	Rolling Plus Forging
Pechiney SR	A.10 (cast)	720 - 760	13:1	55:1
	A.20 (powder -50, +110)	750 - 780	17:1	63:1
	A.30 (powder -200)	800 - 850	10:1	33:1
Brush QMV	A.40 (cast)	760 - 830	17:1	60:1
	A.50 (powder -50, +110)	790 - 850	11:1	40:1
	A.60 (powder -200	800 - 880	14:1	46:1

After cleaning, one can make the following observations:

(a) Sheets A.10, A.20, and A.30 apparently are sound.

(b) Sheet A.40 contained a slight defect in the center.

(c) Sheet A.50 contained several severe cracks parallel to a diagonal.

(d) Sheet A.60 was entirely cracked in several directions.

Examination with the naked eye revealed excessive grain coarsening. These phenomena, without doubt interrelated, can be interpreted as the result of a high level of impurities and of the temperature - time conditions during rolling and reheating.

The sound regions were defined by radiography before cutting.

F. Warm Rolling

Sheets A.10, A.20, A.30, A.40, and A.50 were cut into strips 80 mm in width. After several preliminary trials on sheets of present quality, four magnitudes of reduction were obtained while bare rolling of each strip with passes of 2 to 10% in a single direction after preheating to 680 - 710°C. The holding time in the furnace is of the order of 15 to 20 minutes. The temperature upon exit from the rolls varies from 350 to 500°C, depending on the thickness. The magnitude of the true reduction obtained and the numbers adopted for the sheets are presented in Table 2.

It is possible to deduce from these experiments the following remarks:

(1) Warm rolling (that is to say at a temperature below the temperature of recrystallization) is much easier on cast metal than on metal of powder origin, especially with a fine grain size:

(a) Passes attained 10% for the cast against 2 - 3% for the consolidated powders.

(b) Total reduction possible without significant cracks of 150 - 200% for the cast against 40 - 50% for the powder.

(2) A cracking tendency for the powder metal manifests itself through two flaws:

(a) Cracks of "decohesion" appear in the middle of the sheets transverse to the direction of rolling, never observed in the cast sheets.

(b) Spreading cracks at high reductions which propagate themselves in catastrophic fashions following certain directions, while in the cast material these often remain limited in depth.

(3) The pure metal (SR) is the least subject to cracks. On the other hand, the powders (-50, +110) have an intermediate behavior between that of the

cast and of the -200 mesh powder. It seems, therefore, that the suitability for warm rolling is in inverse ratio to the level of impurities and to the oxide content.

(4) The surface condition of the warm rolled sheets is better than that of the hot rolled sheets. For the lower reductions, the surface is smoother, the finer the grain size of the metal. For large reductions, the condition of the roll cylinders becomes predominant in determining the surface condition. In every case, a light film of oxide covers the sheets as a result of heating to 680 - 700°C in air.

G. Analyses

Analyses were carried out by chemical means for the known metallic impurities and for iron by spectroscopy with BeO standards for the metallic elements. The oxygen content has been determined by obtaining residues after solution in bromated methanol. Nitrogen is determined by the Kjehldahl method. The results obtained on the samples of different starting material and after certain stages in processing are presented in Table 3.

1. Metallic Impurities

On cast ingots, the results are very similar on chips removed close to the top or close to the bottom of the ingot, and only the average for each of these qualities is reported. The ratio of the metallic impurity level in the QMV and SR ingots is 5 - 6 against 8 - 10 in the starting materials. The increase in the level of most of the metallic impurities during fabrication of the metal is less obvious in QMV because of fluctuation in distribution of these impurities and of sampling; it is more important in SR, at the ingot stage and at the powder stage, because of the small impurity level in the original flake. The contamination results principally from contact with certain equipment (melting crucibles, turning tools, grinder, etc.).

2. Oxygen and Nitrogen

The levels of BeO shown in Table 3 can, in general, be considered to be too large for two reasons:

(a) The presence of a superficial layer of oxide on the chips and the thin sheets. Lower results are obtained on well-cleaned massive samples.

(b) The persistence in the insoluble residue in bromated methanol considered to be BeO, of metallic particles protected by a thin film of oxide, or of impurities which are themselves insoluble; the excess over the correct value will therefore be greater, the less pure the metal (QMV).

The values of BeO in metal of powder origin are clearly greater than those of cast metal; the increase is approximately the same for SR and QMV (2000 ppm for the -50, +110 mesh powder and 11,000 ppm for the -200 mesh powder). One can relate this increase of the oxygen content to the increase of the specific surface during grinding. A calculation, assuming the powder particles to be spherical, gives specific surfaces of the order of 120 cm^2 per

gram and 600 cm^2 per gram, respectively, in the same ratio as the increase in oxygen content. For the -200 mesh powder, the hypothesis of a uniform film of oxide of 300Å thickness leads to an absolute value of 5000 ppm BeO. Taking into account the cracked and irregular form of the grains of powder, a value double this calculated value is perfectly plausible. This oxidation is considered to be inevitable in the course of production of the powder as a result of repeated contact of a small quantity of powder with the residual impurities (oxygen, water vapor, etc.) in the glove box. The increase in the level of nitrogen can be interpreted in the same fashion.

H. Recrystallisation Temperature

The temperature of recrystallisation, TR, of each sheet has been determined with the help of an X-ray diffraction criterion from the pieces submitted to anneals under vacuum, always for one hour, at increasing temperatures. All the warm rolled sheets produced patterns of continuous rings. The temperature of recrystallisation has been defined as the lowest temperature of annealing which, after an anneal of one hour, leads to diffraction patterns containing spots and streaks with complete disappearance of continuous rings. We shall see in Section III.B.1.f. that optical metallography at elevated temperature combined with electron microscopy has made it possible to define the temperature of recrystallisation more precisely. Figures 10 and 29 show the appearance of X-ray diagrams before and after recrystallisation. Consideration of the following factors has led to the adoption, for sheets worked approximately the same amount, the same temperature for TR:

(a) The necessity of avoiding an excessive number of thermal treatments.

(b) Slow variation of TR as a function of the amount of reduction.

(c) Minor influence of a slight increase of annealing temperature above TR on the properties of the metal, particularly on sintered beryllium.

The temperatures adopted are shown in Table 7.

I. Thermal Treatments

Six thermal treatments were applied to each sheet of the program on both sides of the recrystallisation temperature as is indicated in Table 8. Anneal numbers 1, 2, 3, 5, and 6 were carried out in a furnace having a large thermal inertia with rapid increase of temperature and slow cooling (about four hours to 250°C). The specimens were treated under vacuum for the cast metal, under argon for the powdered metal. Anneal number 4 (flash) was carried out on a limited number of specimens. It consisted of a rapid heat (40 to 70 seconds) to a temperature of 800 to 1050°C. This treatment did not permit obtaining, in reproducible fashion, recrystallisation without grain growth and the results of mechanical tests consequently are of little significance.

TABLE 7. RECRYSTALLIZATION TEMPERATURES

Type	Number	Temperature (°C)
SR cast	A.11 and A.12	730
	A.13 and A.14	700
SR powder	A.21 to A.24	800
	A.31 to A.34	800
QMV cast	A.41 and A.42	775
	A.43 and A.44	760
QMV powder	A.51 to A.54	810

TABLE 8. THERMAL TREATMENTS

Number	Type	Duration	Temperature (°C)
1	Recovery	1 hour	TR -100
2	Recrystallization	1 hour	TR
3		10 hours	TR
4	Rapid anneal at high temperature		
5	Grain growth	1 hour	TR +200
6		100 hours	TR +200

III. METALLOGRAPHIC STUDY

A. Metallographic Techniques

1. Polishing of Specimens for Optical Microscopy

When examination of specimens is carried out with polarized light, it is necessary to obtain an excellent surface condition. Serious difficulties had to be surmounted before obtaining satisfactory photomicrographs, particularly on powdered metal. Mechanical polishing causes the formation of a superficial deformed layer which masks the structure. Electrolytic polishing, regardless of the bath, is always accompanied by an attack of the network of oxide and impurities, which enormously reduces the quality of the photomicrographs. The technique adopted is the following: (a) polishing with light pressure on abrasive papers 240, 400, and 600 of specimens enclosed in araldite; (b) polishing with soft alumina on soft felt; (c) electrolytic polishing on Disa-Electropol with reagent E 2; and (d) light polishing with soft alumina.

In the sintered metals, oxide is not visible at medium magnification after polishing. One can make it apparent with a light etch in a solution of 10% HF in alcohol. It should be pointed out that, except for the sintered specimens, examination on a cross section gives better results than on the surface.

2. Hot Optical Microscopy

A special chamber, Vacutherm Reichert, has been mounted on the optical microscope, which permits continuous examination of the specimens during their heating in a vacuum of 10^{-5} mm of Hg. This apparatus has permitted prolonged observation on sheets of beryllium for several hours at temperatures of 800°C or for one hour up to 1100°C. It does not produce any thermal etching, which makes it possible to follow the movement of boundaries with polarized light.

3. Obtaining Thin Preparations for Electron Microscopy

Thin preparations could be obtained, starting with cast or sintered material in all the conditions, for examination by transmission in the electron microscope. The fragments of sheets or cut foils from massive specimens are thinned mechanically to 8/10 mm. To avoid the introduction of parasitic deformation, the thinning is continued by chemical means down to several tenths of a mm. The thin preparations are then obtained by electrolytic polishing on Disa-Electropol with the following conditions: (a) successive polishes in bath E 2 by 15-second passes alternating on each face (8 to 10 passes) with 30 volts; and (b) fishing out from the electrolytic bath the thin preparations of diameters varying between 50 and 300 microns or cutting from the thin edge of the polished lamellae.

B. Results of the Study

1. Cast Metal

a. As-cast Billets

Optical microscopic examination of specimens cut from the top of the ingots calls for the following remarks: (a) the metal having the most impurities has the finest grain size; and (b) constituents out of solution of 1 to 10 microns are abundantly visible in the QMV metal in the interior of grains and in the grain boundaries (Figure 1(a)). These constituents are rare in SR metal.

b. Forged Plates

Only the plate forged from SR could be examined. The metal consists of bands of lenticular grains. Under polarized light, a rotation of the specimens makes it possible to discern polygonisation in slightly disoriented blocks in peripheral regions of these grains. The large grains are often separated by strings of small grains originating from a primary recrystallisation (Figure 1(b)).

c. Hot Rolled Sheets

(1) X-Ray Diffraction

The SR sheet (A.10) gives a diagram of a recrystallised structure; the QMV sheet (A.40), a non-recrystallized deformed structure.

(2) Optical Microscopy

The structures of the two sheets are clearly different. A sheet of SR consists of essentially equiaxed grains with slightly sinuous contours and a somewhat sufficiently uniform size between 80 and 250 microns (Figure 2(a)). QMV sheet is much more heterogeneous; coarse flattened grains are surrounded by agglomerates of fine grains of 10 to 60 micron size (Figure 2(b)).

(3) Electron Microscopy

Thin sections of SR beryllium (A.10) show grains separated by boundaries of strong disorientation (Figure 3(a)). Thin sections of QMV beryllium (A.40) show polygonised areas with a very variable cell size and blocks of several micron size defined by sub-grain boundaries of substantial disorientation (Figure 3(b)).

(4) Interpretation

We shall see, after having studied the recrystallisation, that the structure of sheet A.40 comes about through recrystallisation initiated by nucleation and growth (formation of fine grains) during rolling. Sheet A.10 seems to have arrived at a more advanced state of complete recrystallisation and grain growth.

Hot rolling involves a succession of reheatings of 50 to 80°C above the recrystallisation temperature, TR, and of rolling passes while the temperature is dropping toward TR. There is competition between the mechanisms

MF 13.898 90X

(a) - QMV Beryllium As Cast (Polarized Light)

MF 13.895 90X

(b) - SR Beryllium Cast - Forged (Polarized Light)

Figure 1 - Microstructure of Cast Beryllium

(a) - A.10 SR Beryllium (Polarized Light)

(b) - A.40 QMV Beryllium (Polarised Light)

Figure 2 - Microstructure of Hot Rolled Sheets of Cast Beryllium (Cross Section)

E.8630 16,000X

(a) - A.10 SR Beryllium

E.10.449 16,000X

(b) - A.40 QMV Beryllium

Figure 3 - Microstructure of Hot Rolled Sheets of Cast Beryllium (Thin Section)

of hot deformations and working on the one hand, polygonization and recrystallization on the other hand. It is difficult to analyse these phenomena subsequently and to separate the real causes of the structural differences between the two kinds of sheet. However, one can think that a role is played by: (a) the level of impurities in QMV sheets which limits or restrains grain growth; (b) a lower rolling temperature of SR sheet and the higher reductions per pass, which permit the accumulation of a greater amount of deformation energy available for inducing recrystallization; and (c) the greater difference between the temperature of rolling and temperature of final reheating for the SR metal, which would be responsible for a more complete recrystallization. For all the conditions, it is important to note the differences in average size and degree of homogeneity of the structure and the presence of substructures in QMV metal.

(5) Impurities

The large constituents out of solution are abundant in QMV metal and are not systematically associated with the grain boundaries. In the thin sections, it appears that some of these have undergone eutectic melting, sometimes in the form of peripheral excrescences and sometimes with spreading along the grain boundaries.

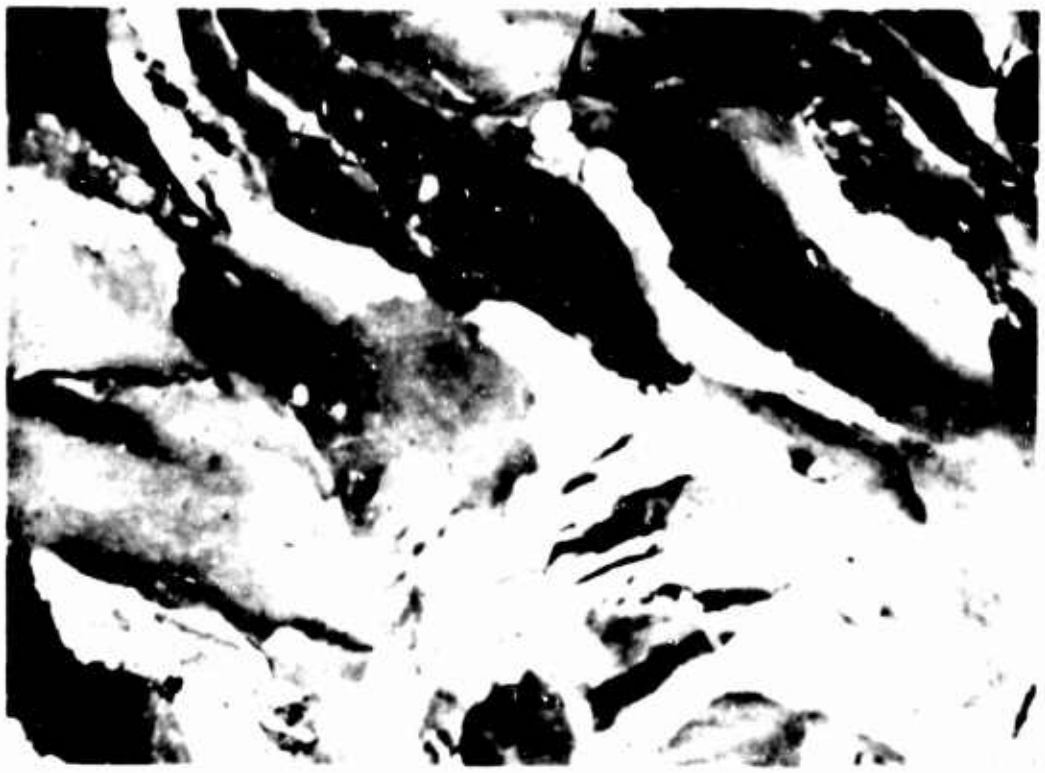
d. Warm Rolled Sheets

(1) The X-ray diffraction patterns are always composed of continuous diffuse rings, which is a criterion of a worked structure. With optical microscopy (Figure 4), the structure revealed with polarized light makes it possible to see strong heterogeneity of deformation. The large grains of the rough, hot rolled material remained individualized but have been deformed by flattening or bending. They are often surrounded by zones of confused appearance. These strongly deformed zones could be formed either by friction or contact of two large neighboring grains or by crushing of regions which were initially fine grained. They have an elongation which is notably greater in QMV metal as a consequence of the non-recrystallized and heterogeneous structure of the rough, hot rolled material. The size of the large less deformed grains (see Table 9) is a function of the initial size of the grains in the rough, hot rolled material and of the magnitude of the deformation. The ratio of the maximum size in the SR and QMV sheets is about 2 or 2.5 to 1 at equal reductions.

(2) The thin sections used in electron microscopic examination of SR metal present a gamut of aspects which we have tried to relate to the microscopic structure:

(a) The interior of the large grains consists of cells with geometric contours separated by parallel sub-boundaries in the simple crystallographic directions ($\langle 10\bar{1}0 \rangle$ or $\langle 1\bar{2}10 \rangle$) and traversed by several freed clusters of dislocations (Figure 5(a)). The disorientation between these blocks is small and one often obtains the same electron diffraction pattern over several tens of microns.

(b) Regions with a structure which is clearly more confused with dense veins of dislocations and small blocks more or less distorted (Figure (5(b)). In the microdiffraction diagrams, a multiplicity of spots caused by rotations of several degrees implies local disorientations.



PC.338 200X

(a) - A.13 SR Beryllium - 180% Reduction
(Polarized Light)

MF 13.786 200X

(b) - A.43 QMV Beryllium - 180% Reduction
(Polarized Light)

Figure 4 - Microstructure of Warm Rolled Sheets of Cast Beryllium (Cross Section)

TABLE 9. EVALUATION OF THE GRAIN SIZE

Treatment Number	Reduction $100\ \frac{t_o - t}{t}$ %	Not Annealed	Recrystallization		Grain Growth	
			1 Hour TR	10 Hours TR	1 Hour TR + 200	100 Hours TR + 200
Cast SR						
A.10*		40 - 250				
A.11	50	30 - 200	30 - 150	--	100 - 400	150 - 800
A.14	255	20 - 100	20 - 70	--	40 - 200	100 - 300
SR Powder -50, +110						
A.20*		2 - 10**				
A.22	50	1 - 8**	35		--	40
A.24	235	1 - 5**	30	--	--	35
SR Powder -200						
A.30*		1 - 10**				
A.31	50	1 - 8**	25	--	--	40
A.34	225	1 - 5**	20	--	--	35
Cast QMV						
A.40*		5 - 55				
A.41	50	7 - 55	12 - 75	12 - 80	60 - 200	100 - 240
A.44	245	2 - 30	6 - 40	7 - 50	50 - 110	50 - 130
QMV Powder -50, +110		5 - 70				
A.50*		25 - 120				
A.51	50	10 - 85**	10 - 80	10 - 80	15 - 90	10 - 90
A.54	250	10 - 55**	10 - 55	--	12 - 60	10 - 65
QMV Powder -200		4 - 25				
A.60*		3 - 120**				

* Hot rolled sheets ** Thickness of longitudinal grains
For values of TR, see Table 7.

E.854.2 16,000X

(a) - A.13 SR Beryllium - 180% Reduction

E.8512 16,000X

(b) - SR Beryllium - 180% Reduction

Figure 5 - Microstructure of warm Rolled Sheets of Cast Beryllium (Thin Section)

(c) Conditions between these two extremes are found. Several areas are separated into two regions, each having a structure which is lightly deformed, by very sinuous boundaries of large disorientation (Figure 6(a)).

(3) The crystallographic orientation of the thin sections always departs slightly from being parallel to the basal plane, making a maximum angle of 20° with it. This is in good agreement with textures measured on warm rolled sheet. One frequently observes hexagonal networks of dislocations corresponding to rotations as large as several degrees about a principal axis. The thin sections of QMV beryllium have a similar structure (Figure 6(b)).

e. Recovery Anneal*

No significant change is discernible in either the X-ray diffraction patterns or in the appearance observed with optical microscopy. With the electron microscope (Figure 7), the recovery is manifested by a polygonization into cells of very variable size arriving at very diverse degrees of perfection. These two characteristics are related to the magnitude and to the disorientation of the cells in the worked condition as well as to the density of the dislocations. After annealing, only a few isolated dislocations remain. However, the limited duration of the anneal does not permit an extensive rearrangement of the structure.

The thin samples have been heated in the body of the microscope (Figure 8). One thus observes, starting at 300°C, a reorganization of the worked cells by displacement of internal dislocations toward the walls without important modification of the dimensions nor the form of these blocks. The limit of heating at 600°C (oxidation and evaporation) prevents the observation of an eventual growth of the cells by coalescence or migration of sub-boundaries. It is likely, nevertheless, that the phenomena observed correspond to the initial stages of recovery of a massive specimen.

In the QMV beryllium, in addition to the gross inclusions which are always present, there exists fine precipitates of the order of 1/10 micron associated with the dislocations or the sub-boundaries (Figure 9). In the SR beryllium, no precipitation has ever been observed.

f. Recrystallization of Worked Sheets

(1) Preliminary Experiments

(a) At first, we proceeded with experiments with the hot optical microscope conducted in parallel with examinations of thin sections in the electron microscope. It is apparent that the structure defined as recrystallized according to the X-ray diffraction criterion results from the competition of two processes:

1. Germination, only in the highly deformed regions, and growth of these nuclei at the expense of the deformed large grains of the matrix.

---

* One hour duration. Temperature 600 to 670°C. See Table 8.

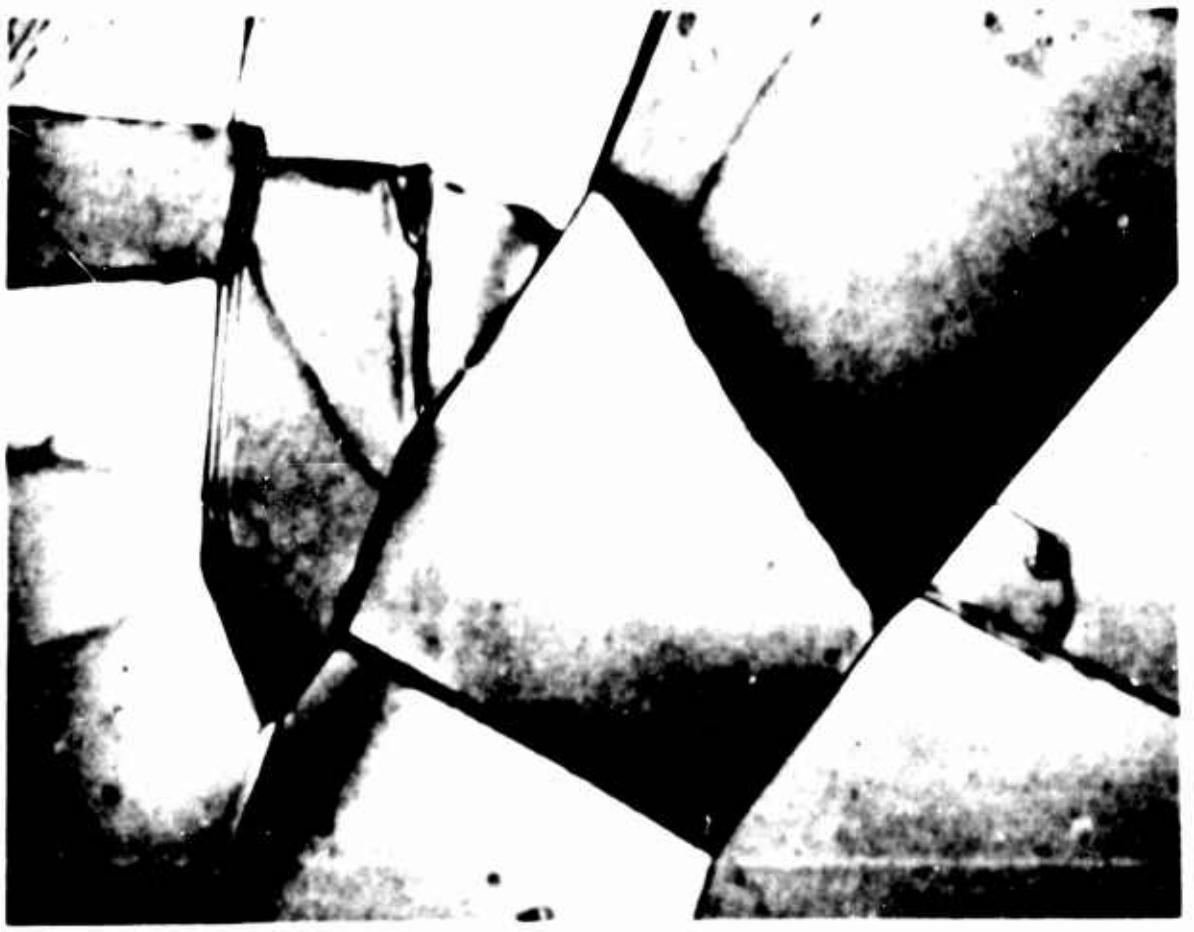
E.9987 40,000X

(a) - A.13 SR Beryllium - 180% Reduction

E.10.428 16,000X

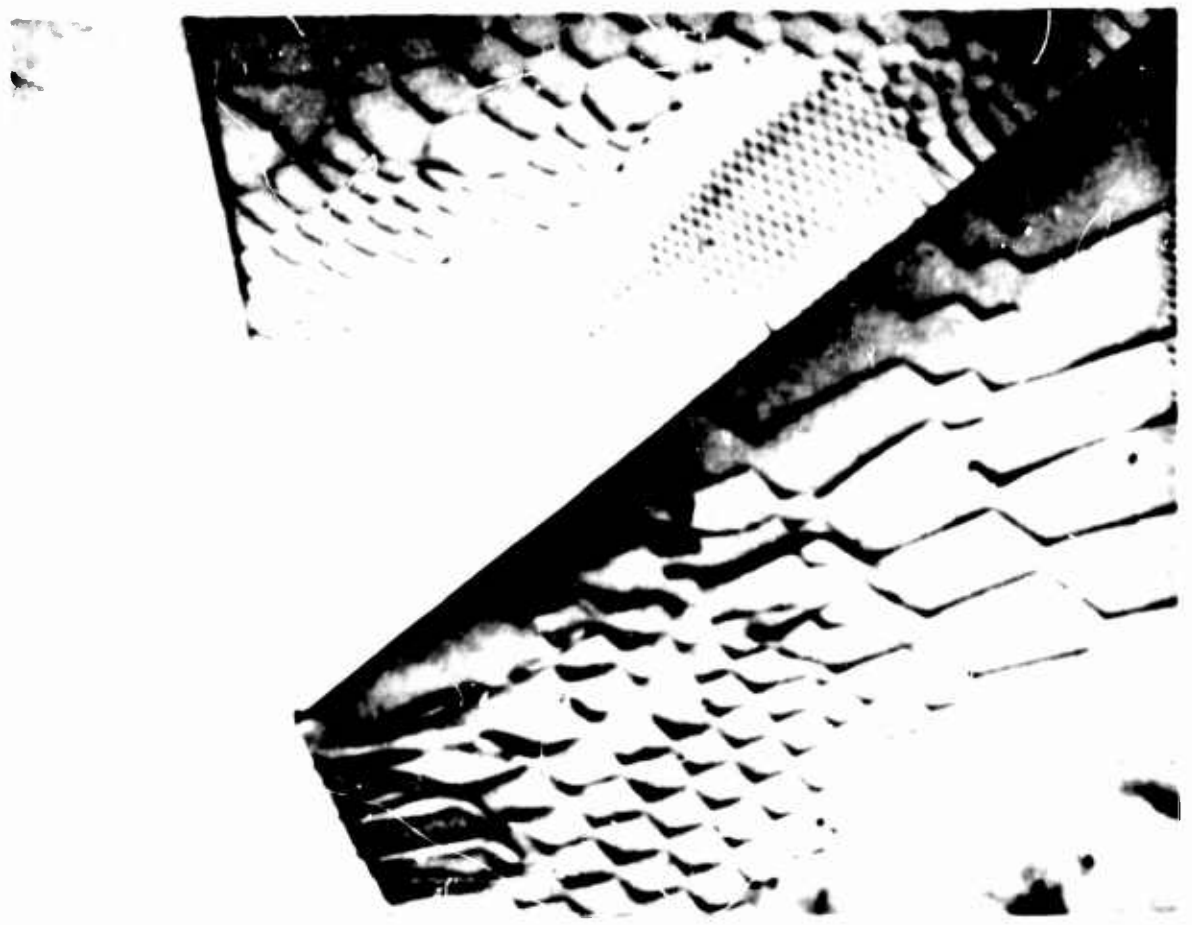
(b) - A.43 OMV Beryllium - 180% Reduction

Figure 6 - Microstructure of Warm Rolled Sheets of Cast Beryllium (Thin Section)



E.9937 40,000X

(a) - A.131 SR Beryllium - 1 hour at 600°C



E.8937 40,000X

(b) - A.131 SR Beryllium - 1 hour at 600°C

Figure 7 - Microstructure of Recovery Annealed Worked Sheets of Cast Beryllium (Thin Section)

E.8566 16,000X

(a) - A.13, Original Condition

E.8568 16,000X

(b) - A.13, 5 Minutes at 300°C

E.8570 16,000X

(c) - A.13, 5 Minutes at 400°C

E.8573 16,000X

(d) - A.13, 5 Minutes at 600°C

Figure 8 - Microstructure of Warm Rolled Cast Beryllium Heated in the Electron Microscope (Thin Section)

E.10.478 16,000X

(a) - A.411 QMV Beryllium Treated 1 Hour at 670°C

E.10.476 16,000X

(b) - A.411 QMV Beryllium Treated 1 Hour at 670°C

Figure 9 - Microstructure of Recovery Annealed Worked Sheets of Cast Beryllium (Thin Section)

2. Simultaneous rearrangement by polygonisation and reorganisation in situ without change of angular orientation in the heart of the less deformed large grains.

(b) Figure 10 makes it possible to follow this evolution in an SR sheet simultaneously with the change of the X-ray diffraction patterns during the course of three isothermal heatings on the same specimen:

1. Figure 10(a): worked condition with a pattern with continuous rings.

2. Figure 10(b): nucleation and appearance of the first spots in streaks on the pattern.

3. Figure 10(c): growth of the nuclei and a pattern indicating a recrystallisation which has just terminated.

4. Figure 10(d): growth of new grains - pattern of a structure freely recrystallized. Only a part of the volume of the metal is comprised of new grains originating from primary recrystallisation.

(c) We were able to observe in a thin sample, by electron microscopy, a region of nucleation noted with the optical microscope. Figure 11 shows two examples of primary nucleation (regions free of dislocations) in process of growing toward the interior of large grains (regions still containing dislocations and numerous substructures). It appears that the nuclei themselves result from the growth of certain highly disoriented blocks in the worked state which have first of all undergone an internal reorganisation.

(d) In the QMV metal, as a result of the relatively greater importance of large regions with large deformation, the number of nuclei is greater. The rate of growth is slower than in the SR metal at equal temperatures (at 750°C, 4 microns per minute against 18 microns per minute). However, because of the larger number of these nuclei and the smaller volume of large initial grains, the nuclei absorb almost all the volume of metal before the large grains are able to achieve recrystallisation in situ. In other terms, the first process becomes preponderant relative to the second and the larger part of the recrystallised metal is comprised of new grains.

(2) Examination of Sheets Annealed One Hour or Ten Hours at TR*

(a) Recrystallisation produces reasonably uniform grains whose size (see Table 9 and Figures 1, 2, 12, and 13) is a function of several factors:

1. Size of the grains of the hot rolled piece, that is to say indirect influence of the purity and the conditions of rolling.

2. Magnitude of the warm deformation: the size of the grains remains smaller than that of the large grains in the worked state.

3. Duration of treatment: the anneal for 10 hours manifests itself by a slight growth of average grain size.

---

* TR = 700 to 775°C. See Table 7.

PC 376-15 180X

(a) - As Rolled (Polarized Light)

PC 381-15 180X

(b) - Heated 20 Minutes at 640°C (Polarized Light)

PC 390-15 180X

(c) - Heated 25 Minutes at 660°C (Polarized Light)

PC 397-15 180X

(d) - Heated 50 Minutes at 680°C (Polarized Light)

Figure 10 - Microstructure of Recrystallized Warm Rolled Sheet of Cast SR Beryllium (Hot Microscopy)

E.10.524 2,000X

(a)

E.10.526 8,000X

(b)

Figure 11 - Microstructure of Cast SR Beryllium Showing Growth Nuclei During Recrystallisation (Thin Section)

NF 13.780 200X

(a) - A.132, 1 Hour at 700°C

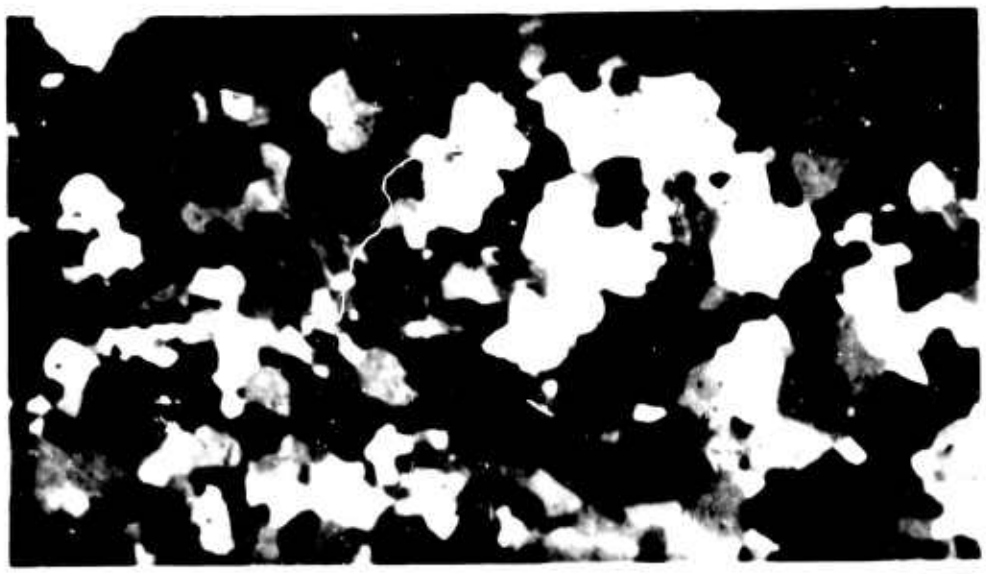
NF 13.916 200X

(b) - A.133, 10 Hours at 700°C

NF 13.901 200X

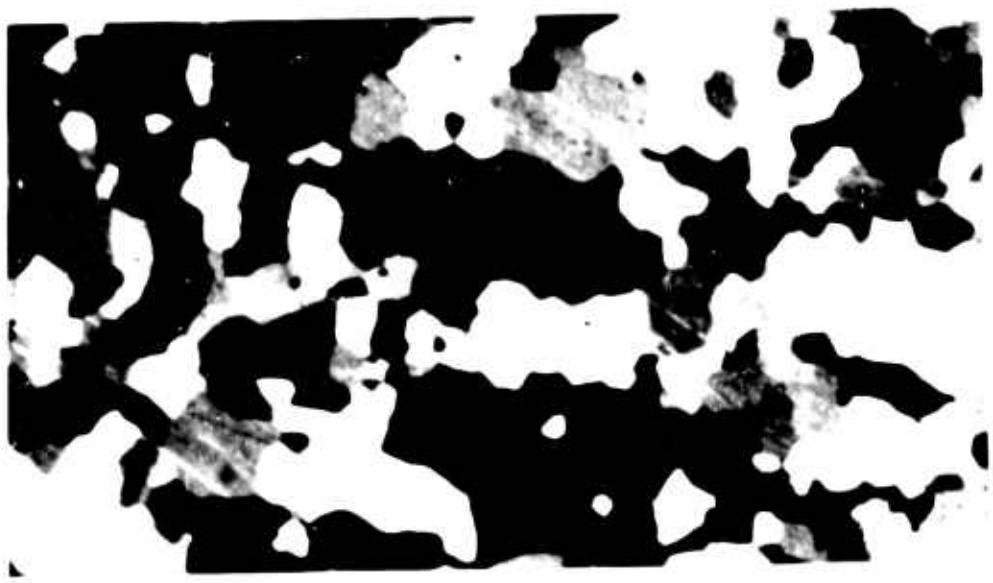
(c) - A.136, 100 Hours at 900°C

Figure 12 - Microstructure of Cast SR Beryllium Showing Recrystallization and Grain Growth



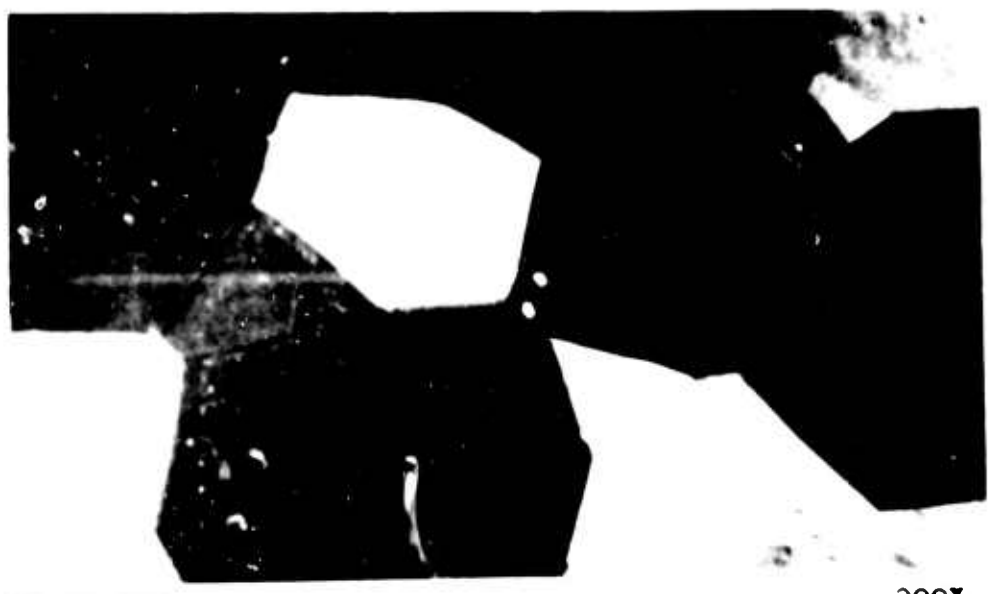
NF 13.864 200X

(a) - A.432, 1 Hour at 760°C



NF 13.865 200X

(b) - A.433, 10 Hours at 760°C



NF 13.918 200X

(c) - A.436, 100 Hours at 960°C

Figure 13 - Microstructure of Cast QMV Beryllium Showing Recrystallization and Grain Growth

(b) With electron transmission microscopy (Figures 14 and 15), one finds:

1. Grains defined by high angle boundaries which must be the grains from primary recrystallization. The boundaries are visible thanks to the interference fringes when the boundaries are oblique to the direction of the electron beam. They are very often associated with a small number of dislocations distributed along one or two well defined directions. Certain dislocations appear to be emitted or absorbed by the boundary (Figure 14(a)). This seems to substantiate the hypothesis of emission or absorption of dislocations by the boundary by certain privileged dislocations. These dislocations seem on the other hand to be situated not in the boundary but in the immediate neighborhood.

2. Other regions where substructures exist which one can consider as the interior of large grains which were initially reorganized "in situ" (Figure 14(b)).

(c) The thin samples starting with SR metal enclose very few dislocations: several constituents out of solution originate from the cast state and several rare precipitates in the boundaries. The thin preparations of QMV metal are, on the contrary, sprinkled with constituents out of solution (Figure 15):

1. Several gross constituents originating from the cast ingot are situated in the grains or are associated with the boundaries.

2. Fine constituents of the order of 1/10 micron are precipitated during the course of the anneal in the interior of the grains that are often associated with dislocations which one can put in contrast by varying the orientation of the thin section.

3. With respect to the other constituents, of intermediate number and formation, it is difficult to know if these arise from a coalescence of precipitates or if these already existed before the anneal.

g. Grain Growth*

(1) The anneals at high temperature cause extensive grain growth. The size of the grains thus produced are subject to several factors:

(a) Amount of initial working: the size of the grain is smaller as the reduction increases. They must, without doubt, feel the influence of the thickness of the sheet as a result of the interaction with the free surface. The ratio of sizes is by a factor of 2 for the extremes of reduction.

(b) The duration of anneal: when one prolongs the anneal from 1 to 100 hours, the grains grow in the ratio of 1 to 2 for SR and 1 to 1.2 for QMV.

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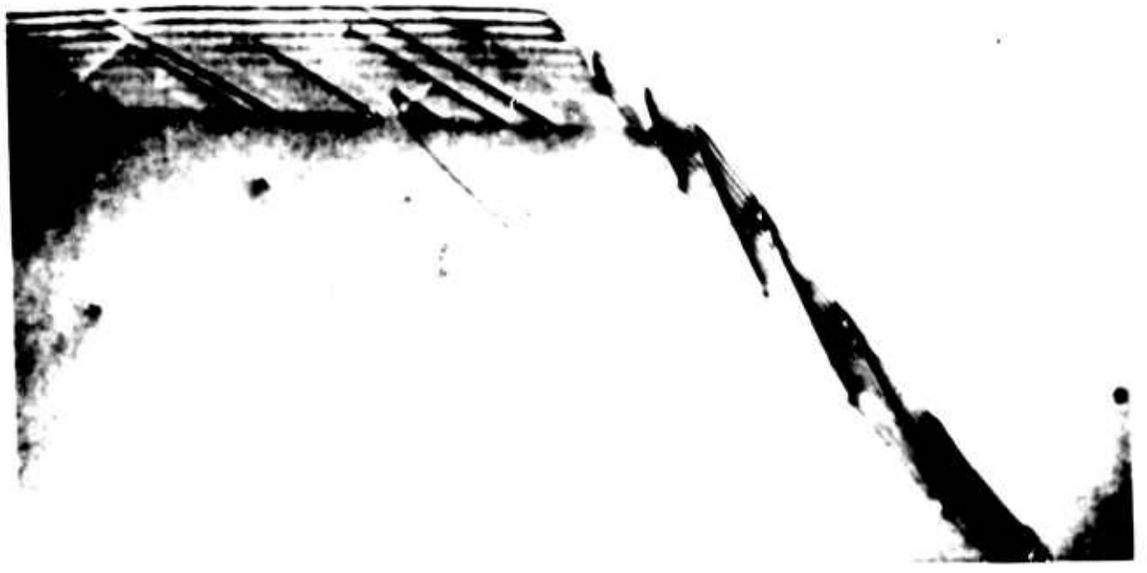
* One hour or one hundred hours. T = 900 to 970°C. See Table 8.



E.9125 16,000X

(a) - A.132, Treated 1 Hour at 700°C

E.9840 16,000X

(b) - A.132, Treated 1 Hour at 700°C

Figure 14 - Microstructure of Cast SR Beryllium After Recrystallization Annealing (Thin Section)

E.10.490 16,000X

(a) - A.442, Treated 1 Hour at 760°C

E.10.495 16,000X

(b) - A.442, Treated 1 Hour at 760°C

Figure 15 - Microstructure of Cast QMV Beryllium After Recrystallization Annealing (Thin Section)

(c) The purity of the metal: the level of impurities in QMV metal reduces considerably the speed of grain growth. Growth during an anneal of 100 hours leads to an increase of size by a factor of 4 for SR metal and a factor of 3 for QMV.

(2) The average size of the grains attained in a sheet of SR is, all other things being equal, in a ratio of 2.5 to 1 as compared with the grains of a similar sheet of QMV. This relative limitation of the size of grains in the metal more highly loaded with impurities (QMV) must be brought about by the restraint of the boundaries by inclusions or Cottrell atmospheres or perhaps by draining of impurity atoms by the boundaries during the course of their migration.

2. Powdered Metal

a. As Sintered Billets

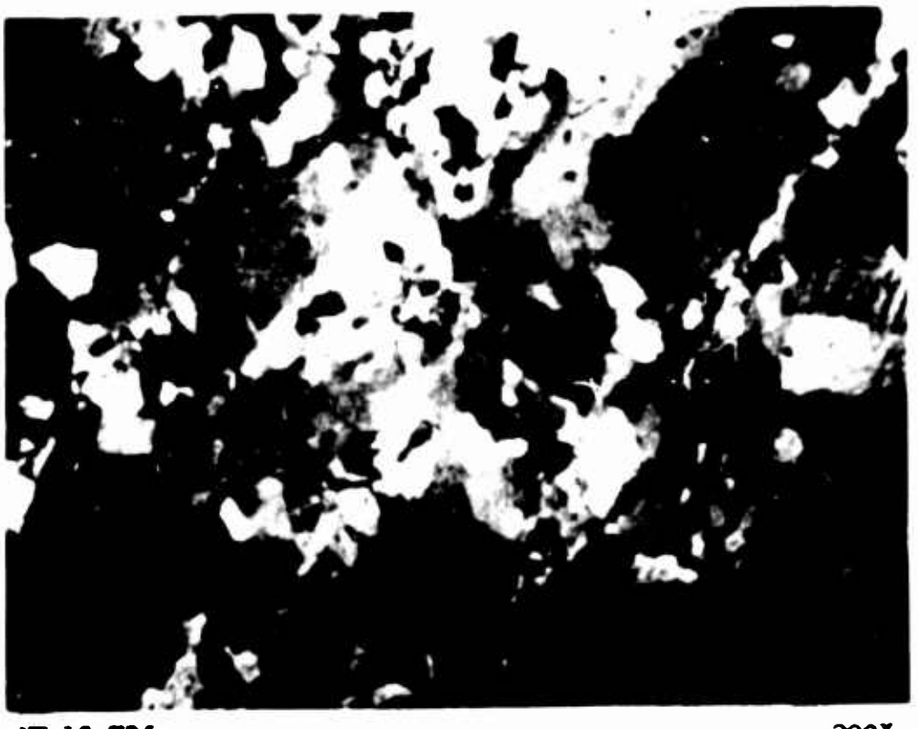
The sintered billets, of QMV beryllium only, were examined in polarised light. The sintered metal originating from powder of -50, +110 mesh consists of polygonized equiaxed grains of a size attaining 70 microns surrounded by bands of fine grains (Figure 16(a)). The sintered metal originating from -200 mesh powder has a similar structure but with finer grains (Figure 17(a)).

A light etch in a solution of 10% HF in alcohol reveals the oxide most satisfactorily. This oxide is distributed along a network corresponding to the contour of the initial grains of powder and to the cracks which penetrated them. In the interior of these networks, the metal is recrystallised, the fine grains being localised at the periphery in the neighborhood of the beds of oxide. One never observes a grain crossing the beds of oxide. The film of oxide surrounding the original particles is therefore, at this stage, a barrier to all migration of boundaries (Figures 16(b) and 17(b)). This distribution of oxide has been confirmed with carbon replicas. The etch also brings into relief a large number of inclusions dispersed in the interior of the grains, just as in cast QMV.

In the thin sections viewed with the electron microscope, one can observe more or less oblique sections of these beds of oxide (Figure 18). These are beds of plate-like particles of some tens to some thousands of Å in diameter. The largest ones give rise to fine spots on the electron diffraction patterns - they are therefore crystalline. In the thick beds, these particles sometimes seem to be cemented by a mass of oxide which appears to be amorphous. One observes boundaries associated with the beds of oxide and others traversing regions free of oxide (recrystallisation boundaries).

b. Forged Plate

Let us recall that the magnitude of upsetting varied from 3 or 4 to 1. Under the optical microscope, the sections give the appearance of a structure derived from that of the sintered billet by flattening of the networks of oxide and the grains. In the interior of the oxide networks, the



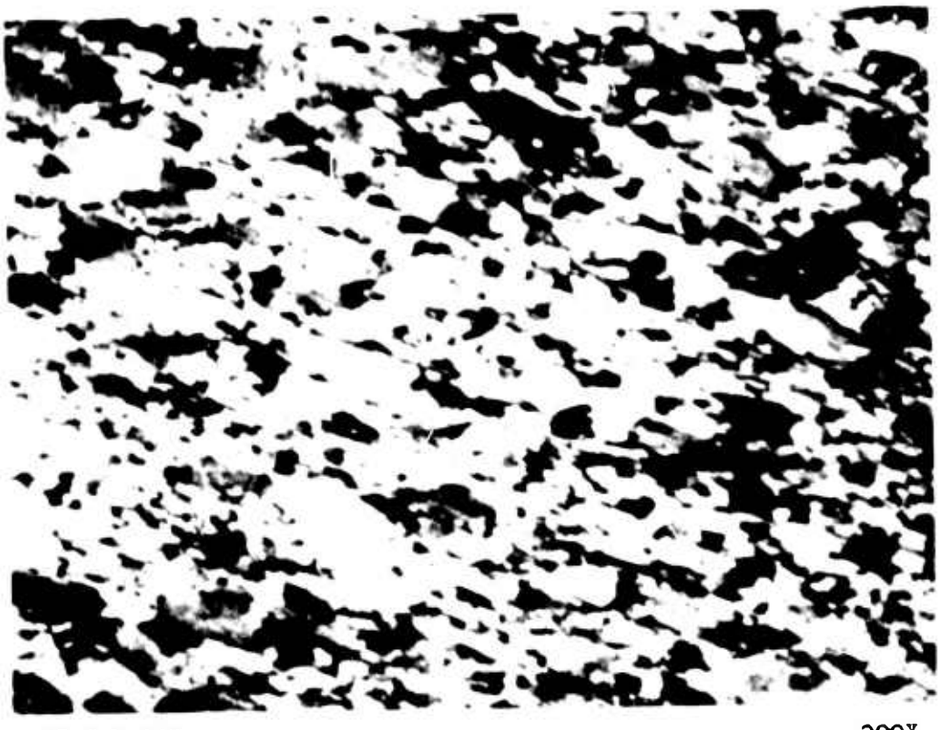
NF 12.725 200X

(a) - QMV Beryllium, as Compacted
(Polarized Light)

NF 13.307 200X

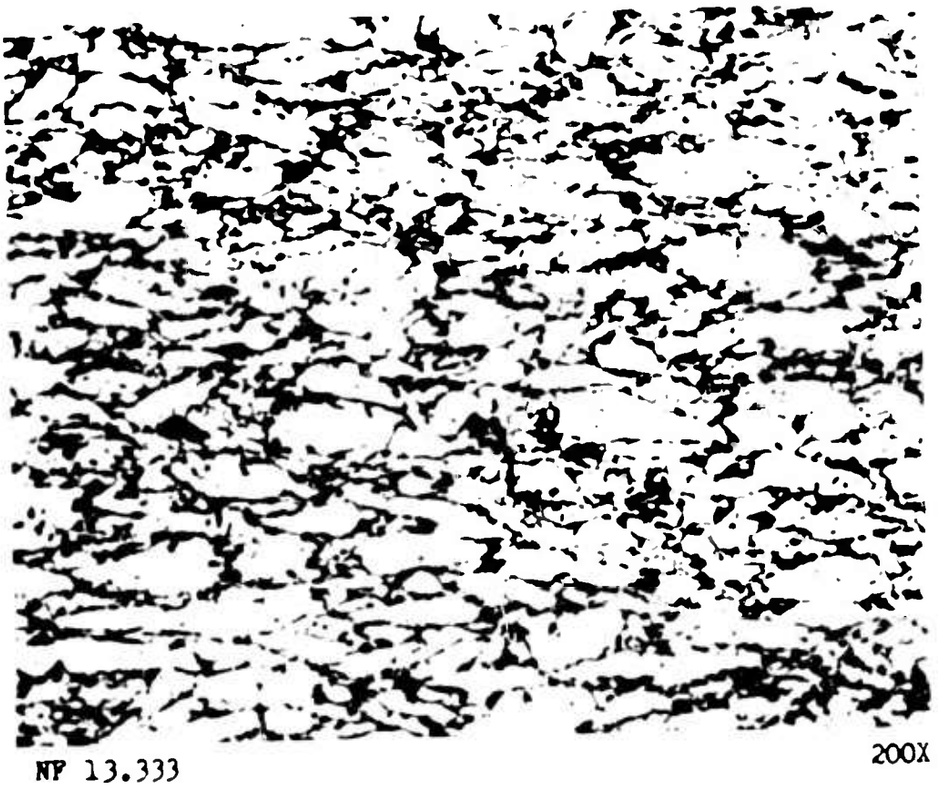
(b) - QMV Beryllium, as Compacted
(Etched 10% HF)

Figure 16 - Microstructure of Powder Beryllium (-50, +110) as Sintered



NF 13.337 200X

(a) - [illegible]ryllium, as Compacted
(Polarised Light)

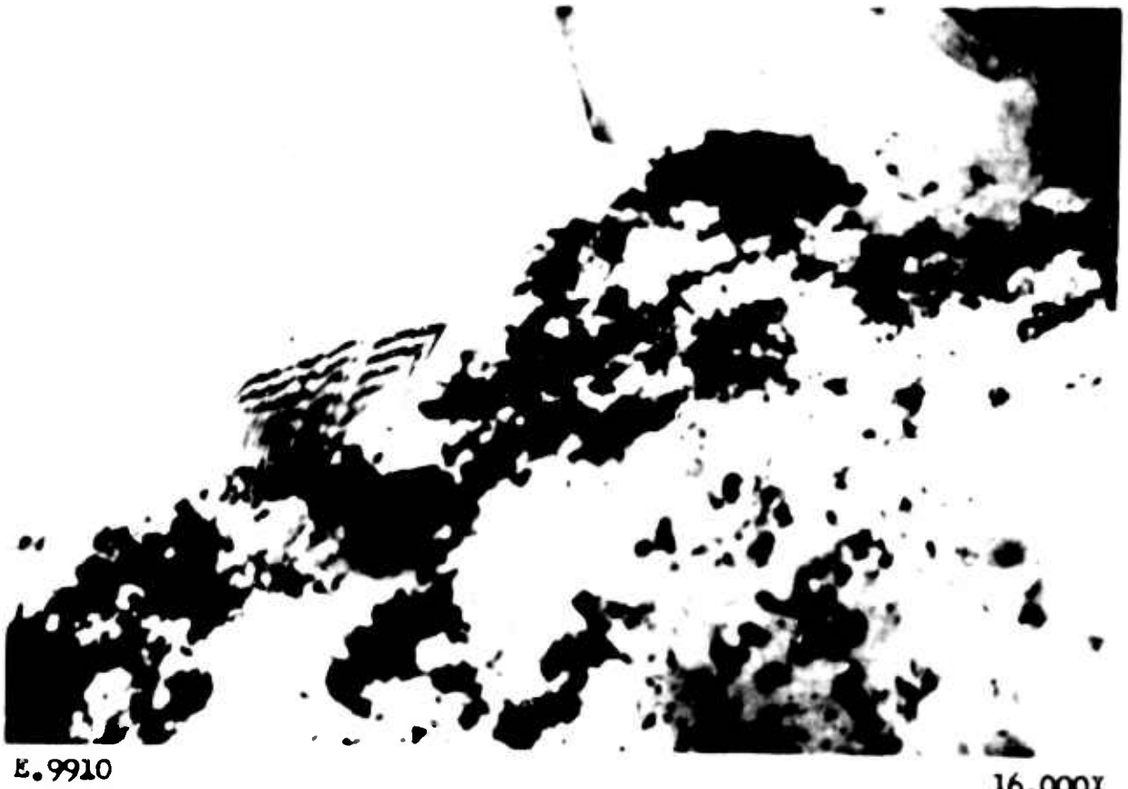


NF 13.333 200X

(b) - QMV Beryllium, as Compacted
(Etched 10% HF)

Figure 17 - Microstructure of Powder Beryllium (-200) as Sintered

(a) - -50, +110 (Thin Section)



(b) - -200 (Thin Section)

Figure 18 - Microstructure of As Sintered Beryllium Powder

metal is polygonized or recrystallized, depending upon the deformation experienced locally. Some grains cross the beds of oxide. Under the electron microscope in thin sections of slices parallel to the direction of forging, the beds of oxide appear in cross section (Figure 19). The frictional forces between grains have lightly dispersed the particles. In Figure 19(b), one sees a small grain crossing a bed of oxide.

c. Hot Rolled Sheets

(1) The X-ray diffraction patterns reveal a worked structure in all cases.

(a) Optical Microscopy

1. An etch with an alcoholic solution with 10% HF on the cross section of the sheets brings into evidence the oxide in the form of very fine serrated beds parallel to the surface as a result of crushing the network of oxide films. The beds are more serrated in the metal from -200 mesh powder than in the metal from -50, +110 mesh powder. We have not noted any significant difference between sheets from SR or QMV with regard to the distribution of oxide. On the other hand, observation with polarized light reveals essential differences between the sheets of SR and QMV.

a. Sheet A.20 (SR; -50, +110) Figure 20(a). The fine grains (1 to 10 microns) of lenticular shape were formed by crushing the grains of the forged plates. They are almost always surrounded by beds of oxide. A few rare large flattened grains at the surface or in the interior are the result of abnormal growth.

b. Sheet A.30 (SR; -200) Figure 20(b). The size of the grains is approximately the same. The beds of oxide, more serrated than the preceding, cross the grains. The rolling therefore has crushed the grains and induced a small simultaneous growth.

c. Sheet A.50 (QMV; -50, +110) Figure 21(a). An important grain growth during rolling has produced large equiaxed grains of 25 to 120 microns with very sinuous contours crossing a large number of beds of oxide (the beds of oxide have been made visible by an etch of 10% HF).

d. Sheet A.60 (QMV; -200) Figure 21(b). Grain growth of a special type has given flat grains with a thickness of 40 to 120 microns extending over several hundreds of microns (we have sometimes seen 1 to 2 mm) and occupying the major portion of the volume. The remaining material has very fine grain structure identical to that of Sheet A.30.

2. Since the distribution of oxide is the same for the two qualities of SR and QMV, the abnormal grain growth of the QMV must be attributed to the higher rolling temperature which was necessary to avoid cracking.

(b) Electron Microscopy

1. The thin preparations give very similar appearances in the four sheets: polygonization into subgrains leading to very diverse

E.8860 16,000X

(a) - -50, +110 (Thin Section)

E.8865 16,000X

(b) - -200 (Thin Section)

Figure 19 - Microstructure of Forged Beryllium Powder

PC 642 450X

(a) - A.20, -50, +110 (Polarised Light)

PC 663 450X

(b) - A.30, -200 (Polarised Light)

Figure 20 - Microstructure of SR Beryllium Powder (Hot Rolled Sheets)

MF 13.322 200X

(a) - A.50, -50, +110, Etched 10% HF
(Polarised Light)

MF 13.879 200X

(b) - A.60, -200 (Polarised Light)

Figure 21 - Microstructure of QMV Beryllium Powder
(Hot Rolled Sheets)

degrees of reorganization or grains with different orientations formed by recrystallization (Figure 22 for the SR and Figure 23 for the QMV). The oxide appears in crystalline platelets dispersed across the thin preparation. The particles clearly are more numerous and smaller in the -200 mesh material. One sees some interactions between sub-boundaries and isolated dislocations but the aggregation of oxide platelets does not have a relation with the structure. Finally, the oxide is sometimes assembled in the agglomerates of platelets similar to that of Figure 25(a). It is convenient to make several remarks here:

a. Calculations show that the surface area of initial grains of powder is multiplied by a factor of 10 to 20 after forging and rolling; because of this fact, and thanks to friction, the particles are strongly dispersed as is shown in the photomicrographs.

b. In certain areas, one sees few particles. This arises from the fact that the thin precipitations, thickness of several thousands of Å, could come from the interior of the band of metal in between two beds of oxide.

d. Warm Rolled Sheets

(1) The diagrams are those of a worked structure.

(a) Optical Microscopy

1. The structure stems from that of each hot rolled sheet:

a. SR sheets. All the grains have been crushed or flattened a little (Figure 24(a)).

b. QMV sheet (-50, +110) Figure 24(b). The size of the grains of the hot rolled piece A.50 is of the same order of size as that of the grain of the rolled cast piece. As a result of this, after warm rolling, the morphology is in fact similar to that described previously for cast metal.

c. QMV sheet (-200). The zones of fine grains behave in the same way for the SR sheets. The coarse, flat grains are elongated and lightly deformed.

(b) Electron Microscopy

As seen in Figures 25 and 26, the structure is similar to that of cast beryllium; cells defined by sub-boundaries and crossed by dislocations. The magnitude of the cells and the density of dislocations varies by large amounts. It seems that the cells are, in the aggregate, less well formed than in the cast metal and are smaller in metal with a fine grain size. The oxide is always in crystalline platelets in contours which are frequently geometric. Except for some agglomerates (Figure 25(a)), the dispersion is better than after hot rolling. Only some of these platelets appear to be associated with sub-boundaries or dislocations.

E.8639 16,000X

(a) - A.20, -50, +110

E.8628 16,000X

(b) - A.30, -200

Figure 22 - Microstructure of Thin Sections of SR Beryllium Powder (Hot Rolled Sheets)

E.9651 16,000X

(a) - A.50, -50, +110

E.9682 16,000X

(b) - A.60, -200

Figure 23 - Microstructure of Thin Sections of QMV Beryllium Powder (Hot Rolled Sheets)

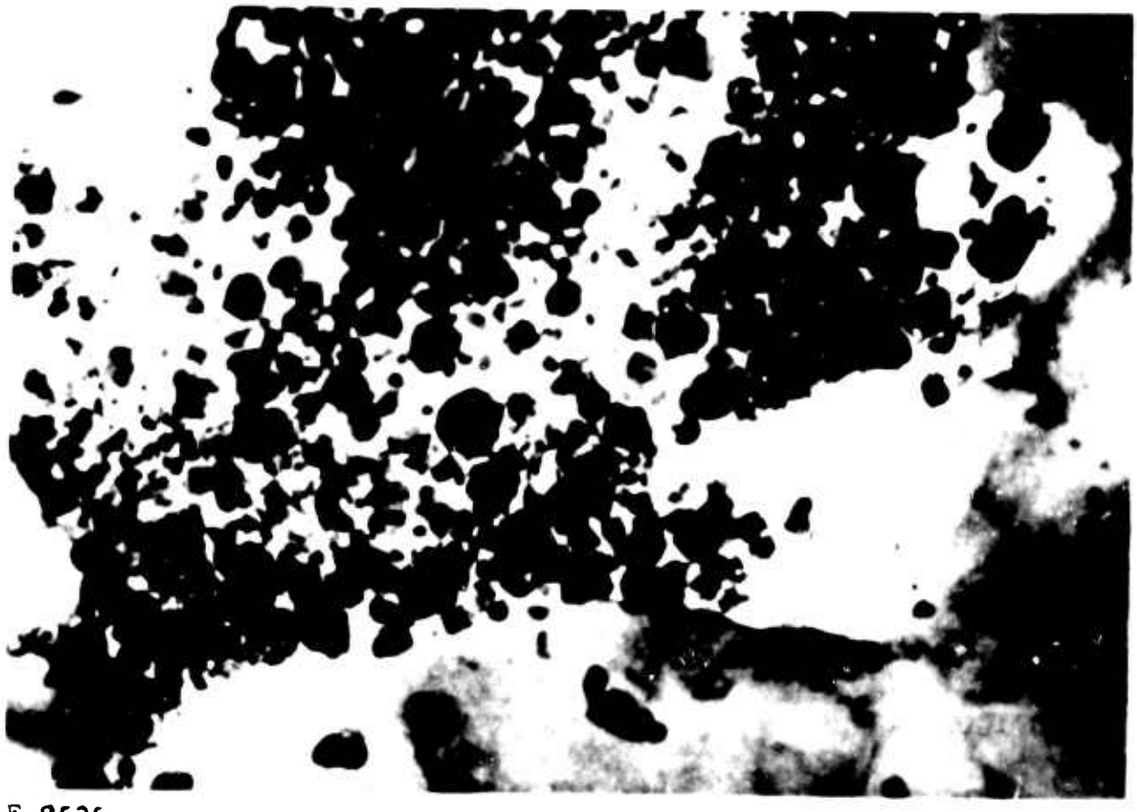
MF 13.921 450X

(a) - A.32, SR Beryllium, 100% Reduction
(Polarised Light)

MF 13.876 200X

(b) - A.52, QMV Beryllium, 100% Reduction

Figure 24 - Microstructure of Warm Rolled Sheets of Beryllium Powder



E.8535 16,000X

(a) - A.23, 80% Reduction (Thin Section)

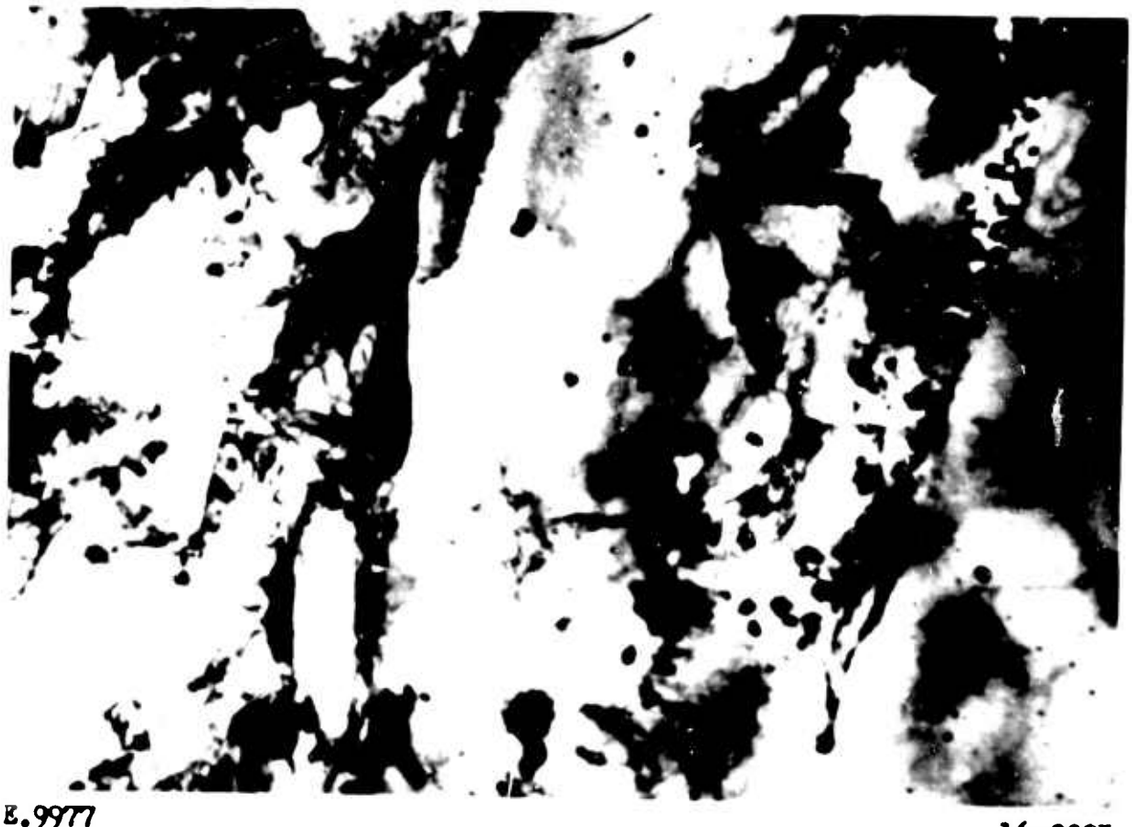
E.8561 16,000X

(b) - A.23, 80% Reduction (Thin Section)

Figure 25 - Microstructure of Warm Rolled SR Beryllium Powder

E.8532 16,000X

(a) - A.32, SR Beryllium, 100% Reduction
(Thin Section)



E.9977 16,000X

(b) - A.52, QMV Beryllium, 100% Reduction
(Thin Section)

Figure 26 - Microstructure of Warm Rolled Beryllium Powder

e. Recovery Treatment*

No change is discernible in the X-ray diffraction patterns or in the appearance with the optical microscope. With the electron microscope (Figures 27 and 28), one observes, as in cast metal, a polygonization more or less advanced. There remain clearly more dislocations than in the cast metal given the recovery treatment in spite of the fact that the annealing temperature is higher. This shows that the particles of oxide impede the motion of dislocations and that a certain amount of anchoring is effected. In the SR metal with fine grain size, the sub-grains are smaller and more disoriented with respect to each other (Figure 27). In the QMV metal, the anneal has caused the precipitation of several fine constituents which one can distinguish from the oxide platelets (Figure 28). These precipitates appear to be less abundant than in the cast QMV metal annealed one hour at approximately the same temperature. They are associated with isolated dislocations or sub-boundaries.

f. Recrystallization

(1) Preliminary Experiments

(a) Several experiments with hot optical microscopy were tried:

1. SR beryllium (Figure 29): In a worked sheet heated to the neighborhood of its TR temperature, one sees appearing a certain number of new grains which grow quickly to a size attaining about 20 microns. A limited optical magnification does not make it possible to locate clearly the nuclei which appear. The number of nuclei increases up to the point that all the metal consists of new grains of irregular form, larger than the original grains, but rarely exceeding 30 microns. Primary recrystallization is complete and rapid and produces a grain size larger than that of the initial grains of the worked metal. Once this phase has been terminated, however, one rarely sees motion of boundaries and one cannot cause a grain growth even upon heating for 1 hour at 1000°C.

2. QMV beryllium (-50, +110): The analogy with cast metal due to the similarity of grain size persists. The recrystallization is of the type described for cast metal previously; recrystallization by nucleation and growth in the regions most highly deformed and reorganization in situ in the original large grains having small deformation. The number of nuclei is no greater and the speed of growth is smaller than in cast metal. We have never observed appreciable grain growth after recrystallization during the course of heating up to 1000°C.

3. QMV beryllium (-200): The regions of fine grain size recrystallize as in the SR beryllium; the large grains do not appear to be modified. They must recrystallize themselves in situ.

(2) Examination of Recrystallized Sheets**

The dimensions of the grains obtained after the recrystallization treatments on the worked sheets are presented in Table 9. Observations

* 1 hour at 700 or 710°C.
** 1 hour or 10 hours at 800 or 810°C.

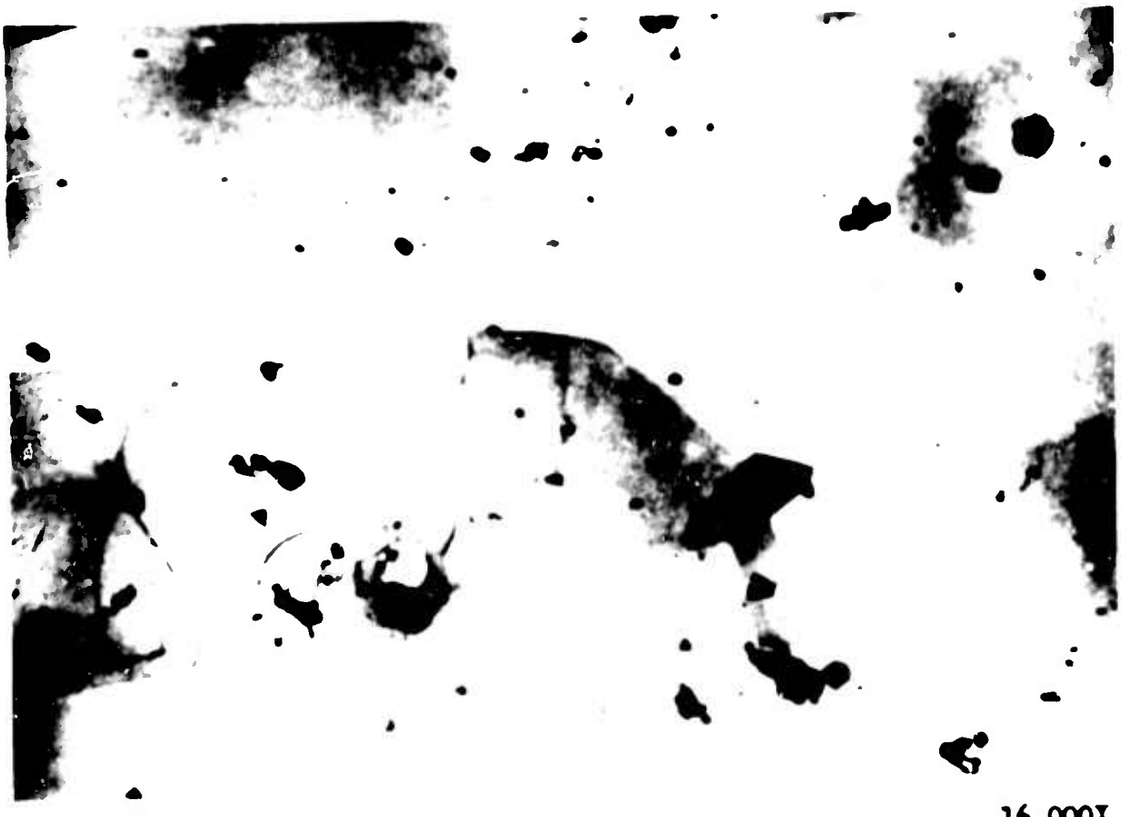
E.9193 16,000X

(a) - A.231, -50, +110, 1 Hour at 700°C
(Thin Section)

E.9376 16,000X

(b) - A.341, -200, 1 Hour at 700°C
(Thin Section)

Figure 27 - Microstructure of Recovery Annealed SR Beryllium Powder



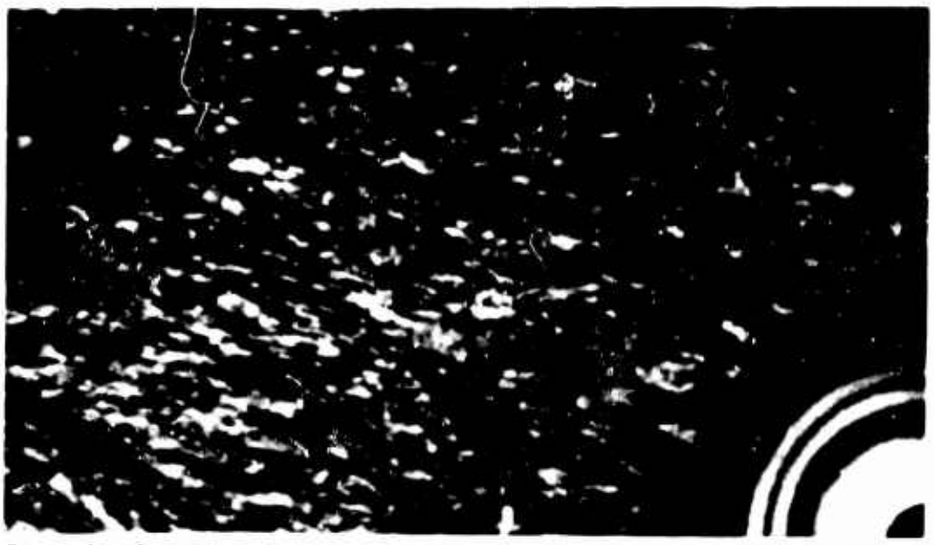
E.10.023 16,000X

(a) - A.521, -50, +110, 1 Hour at 710°C
(Thin Section)

E.10.032 40,000X

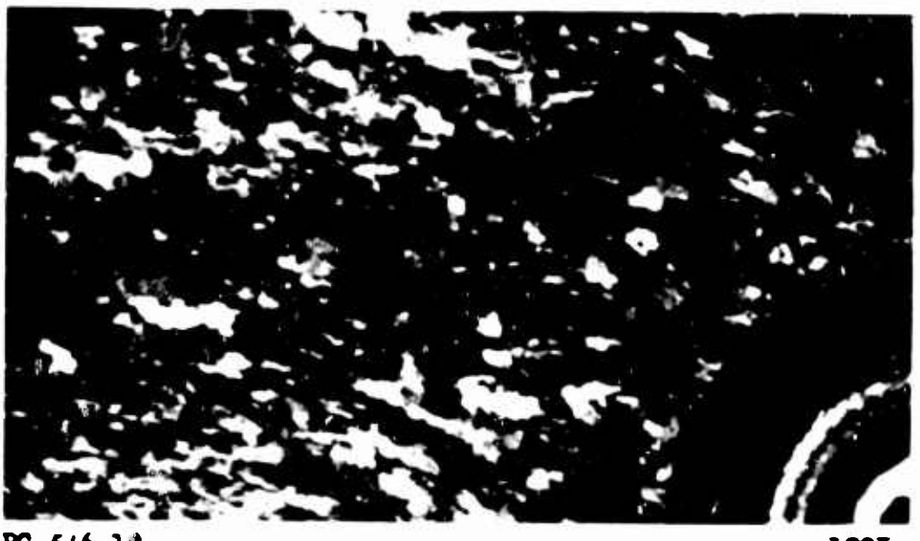
(b) - A.521, -50, +110, 1 Hour at 710°C
(Thin Section)

Figure 28 - Microstructure of Recovery Annealed QMV Beryllium Powder



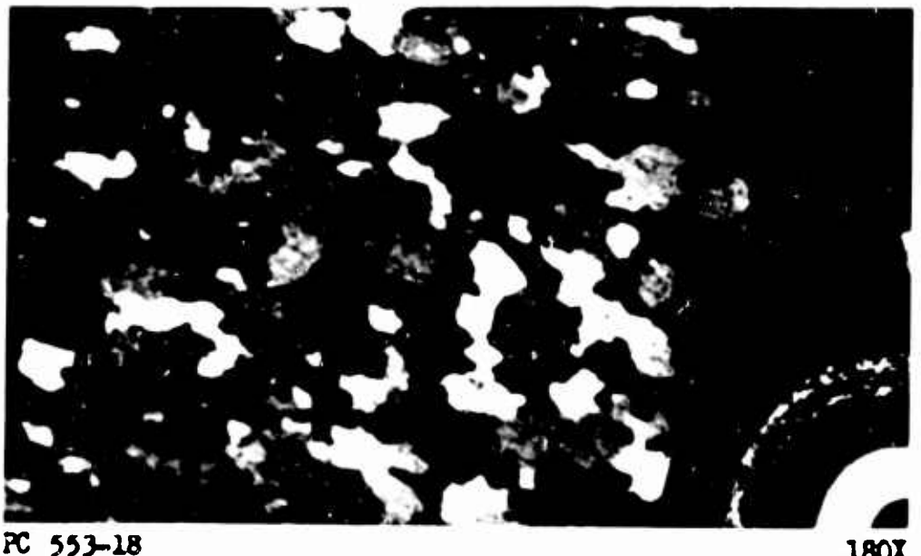
PC 527-18 180X

(a) - A.23, Worke Condition



PC 546-18 180X

(b) - A.23, 6 Minutes at 730°C



PC 553-18 180X

(c) - A.23, 2 Minutes at 800°C

Figure 29 - Microstructure of Recrystallised Warm Rolled Sheet of SR Beryllium Powder (Hot Microscopy)

with the optical microscope agree with the results of the preliminary experiments. The SR beryllium (-50, +110) is recrystallized with fine grains with average size of 18 to 21 microns (Figure 30(a)). The SR beryllium (-200), containing more oxide, has a finer grain size (average of 12 to 15 microns) and a more irregular form (Figure 31(a)). This arises from the more numerous interactions with the beds of oxide: the grain boundaries follow the beds of oxide over a portion of their length or sometimes pass from one bed to another perpendicularly to these beds.

The QMV beryllium (-50, +110) has recrystallized giving a coarser grain with average size 35 to 50 microns (Figure 32(a)). The QMV beryllium (-200) consists of large flat grains and of regions of fine grains (Figure 33).

With the electron microscope (Figures 34(a), 35(a), 36(a), and 37(a)), one sees grains of variable size separated by high angle boundaries. Some dislocations often remain in the interior, especially in the fine grained SR. These dislocations often are anchored to fine particles of oxide. One observes some interactions between the grain boundaries and the oxide either in the form of fine particles or in the form of agglomerates.

In the QMV metal, there always exist large inclusions identical to those observed in the cast or powdered metal in every condition. There does not seem to have been precipitation of fine constituents from solid solution either because the temperature of annealing is too high or because a coalescence makes it impossible to distinguish them from the particles of oxide.

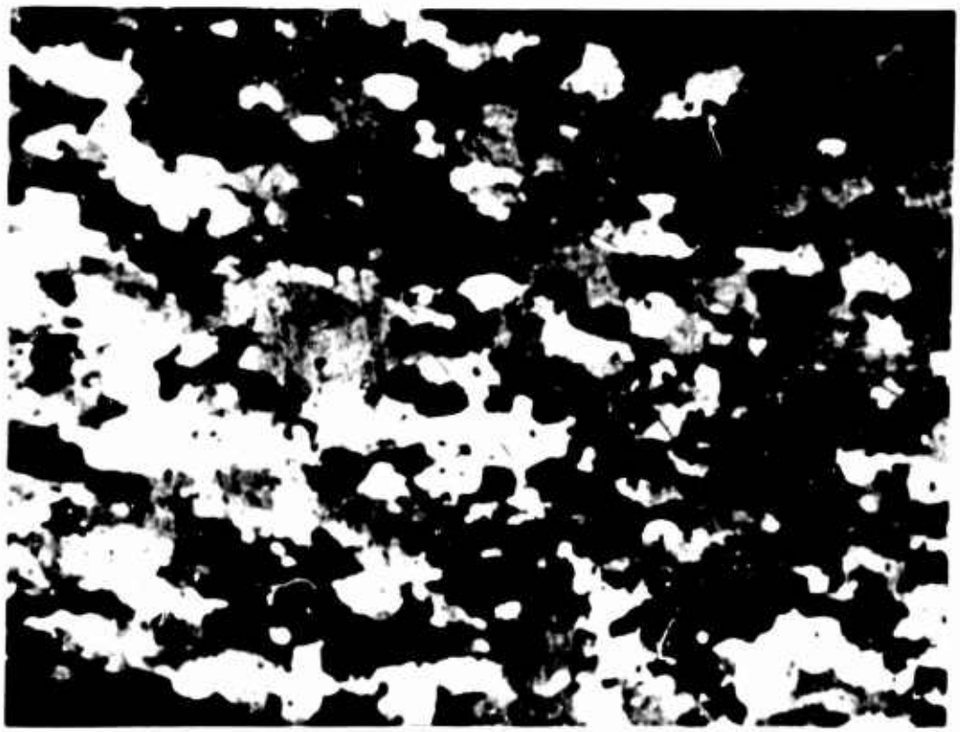
g. Grain Growth

An essential characteristic of the powdered metal consists of the absence of notable grain growth, regardless of the previous history, during the course of annealing at 1000°C even after 100 hours. In the SR (-50, +110), the growth does not exceed 20% and the grains become more equiaxed (Figure 30(b)). In the SR (-200), the growth sometimes attains 50%, but the grains remain irregular and grow more easily parallel to the beds of oxide and thus elongate themselves (Figure 31(b)).

In the QMV (-50, +110), the grain size does not increase more than 20%. It does become uniform (Figure 32(b)). The thin sections reveal little difference in the recrystallized metal (Figures 34(b), 35(b), 36(b), and 37(b)). In the QMV metal, the lenticular constituents rich in aluminum are formed at the boundaries.

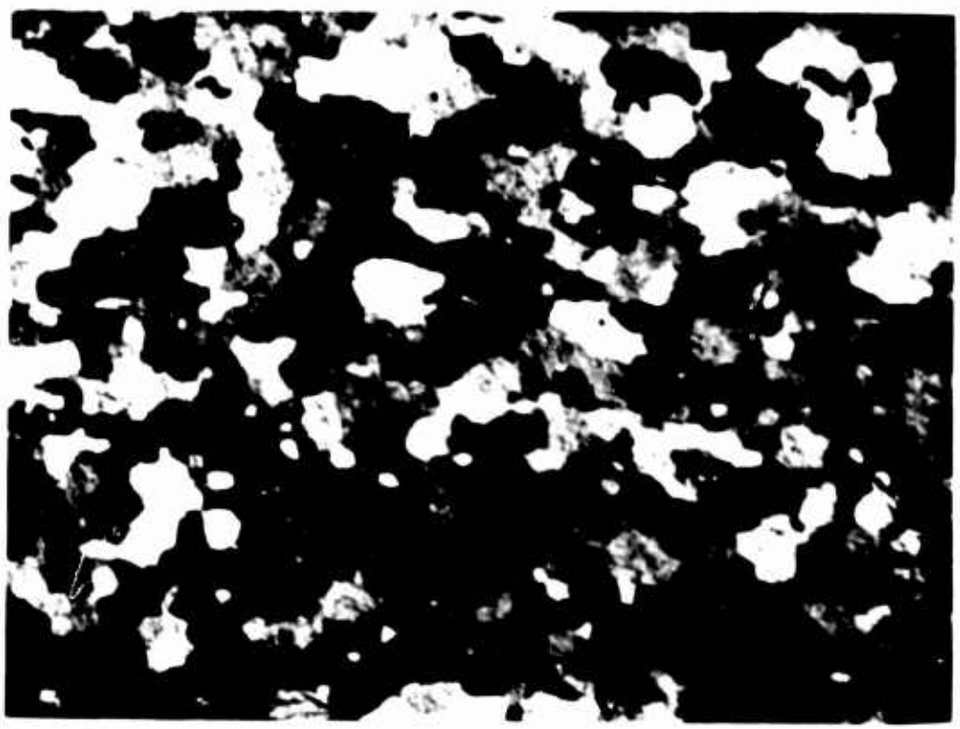
IV. MECHANICAL PROPERTIES

A. Measurement of Properties



PC 478 200X

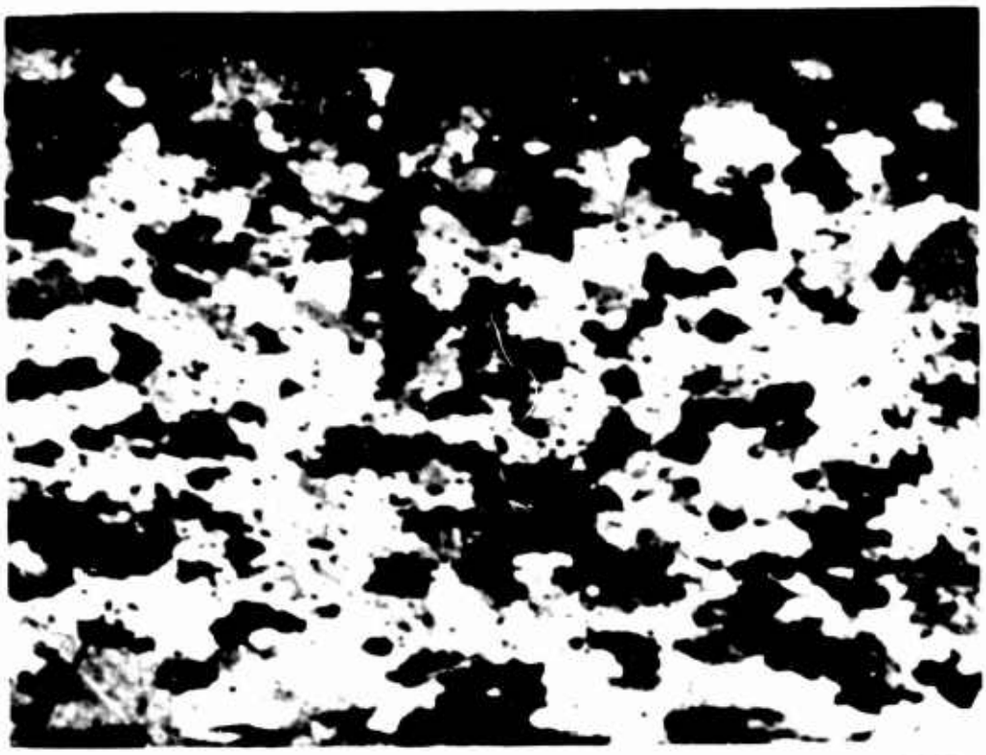
(a) - A.222, 1 Hour at 800°C (Polarised Light)



PC 482 200X

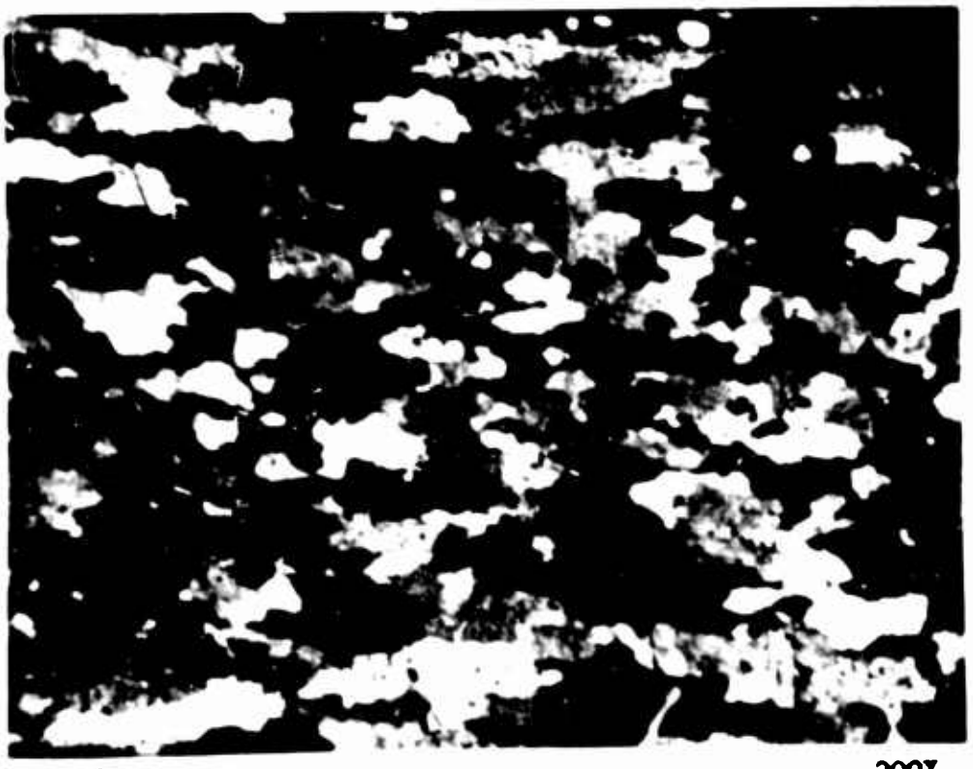
(b) - A.226, 100 Hours at 1000°C (Polarised Light)

Figure 30 - Microstructure of Recrystallised SR Beryllium Powder (-50, +110)



PC 562 200X

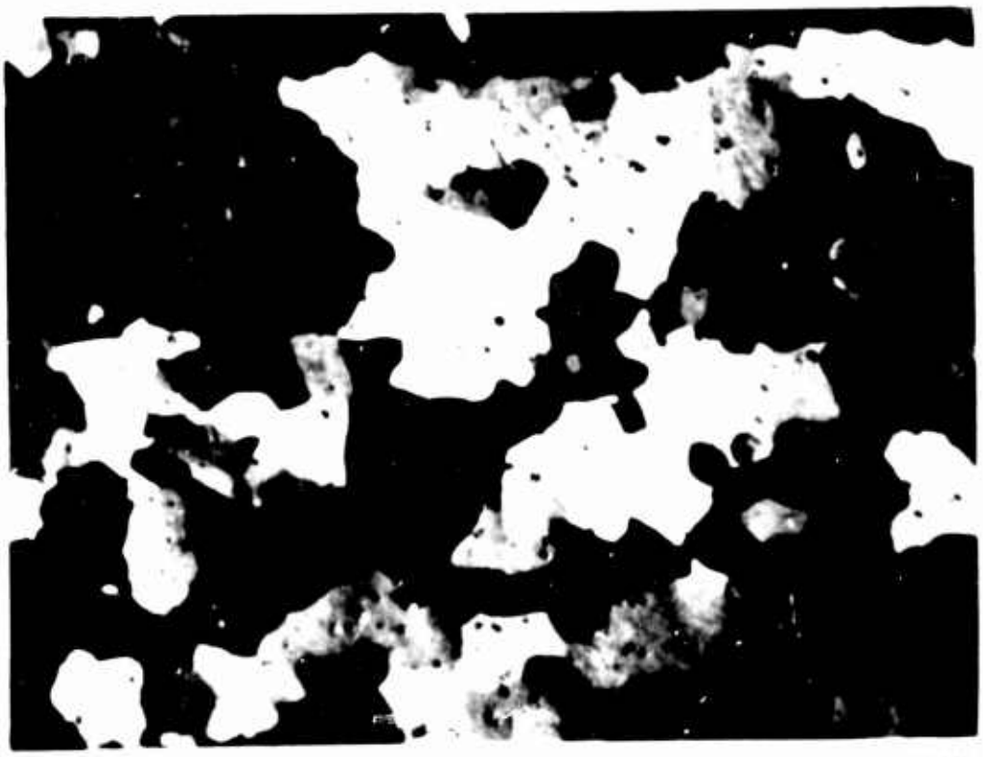
(a) - A.332, 1 Hour at 800°C (Polarised Light)



PC 560 200X

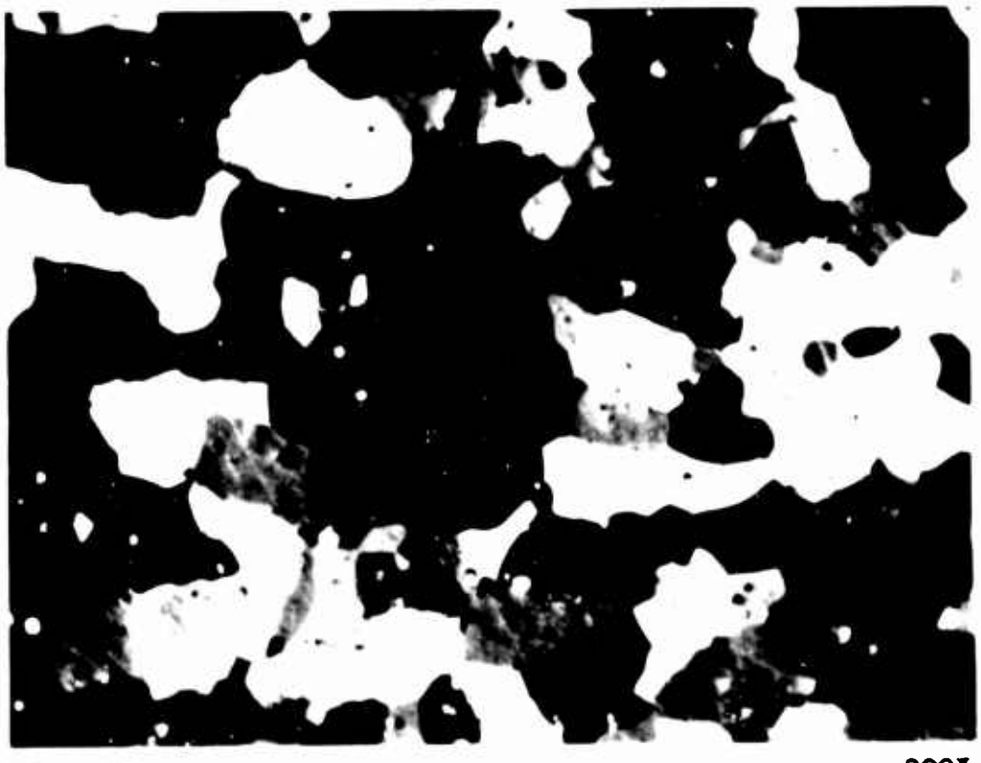
(b) - A.336, 100 Hours at 1000°C (Polarised Light)

Figure 31 - Microstructure of Recrystallised SR Beryllium Powder (-200)



MF 13.870 200X

(a) - A.522, 1 Hour at 810°C (Thin Section)



MF 13.871 200X

(b) - A.526, 100 Hours at 1000°C (Thin Section)

Figure 32 - Microstructure of Recrystallised JMV Beryllium Powder (-50, +110)

MF 13.382 200X

(a) - A.642, 1 Hour at 850°C (Polarised Light)

Figure 33 - Microstructure of Recrystallized HV Beryllium Powder (-200)

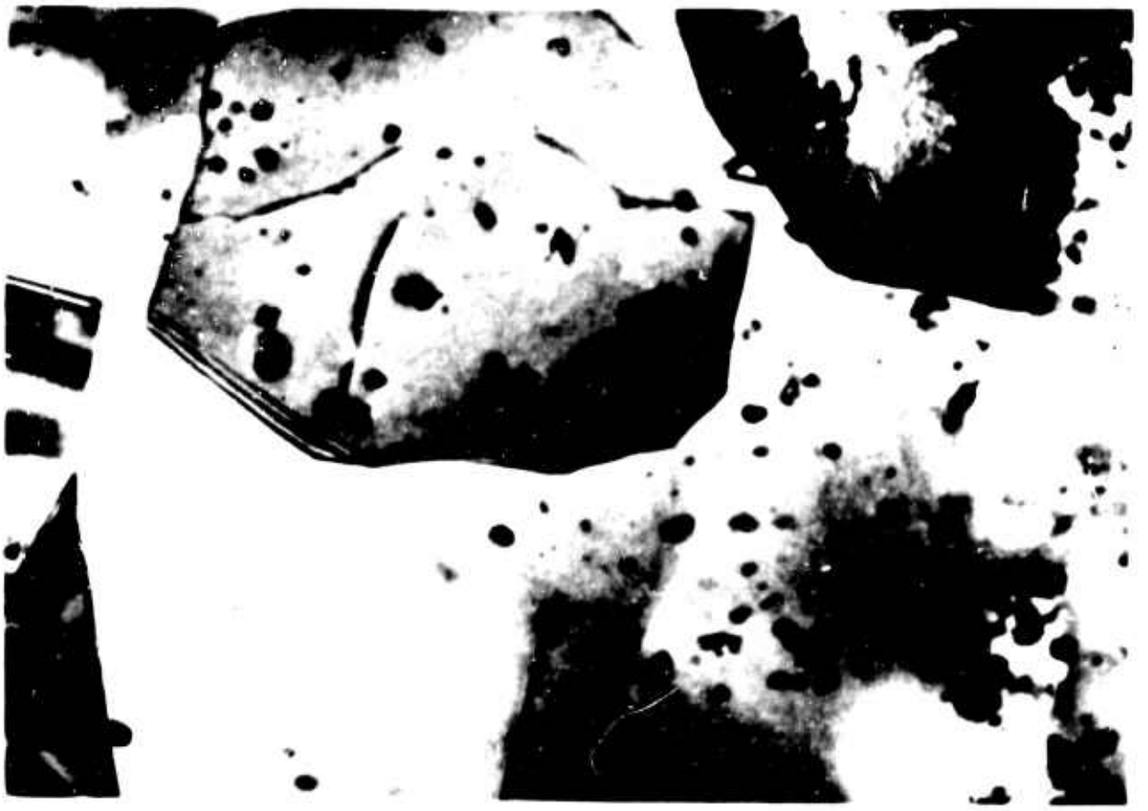
E.9940 16,000X

(a) - A.242, 1 Hour at 800°C

E.9151 40,000X

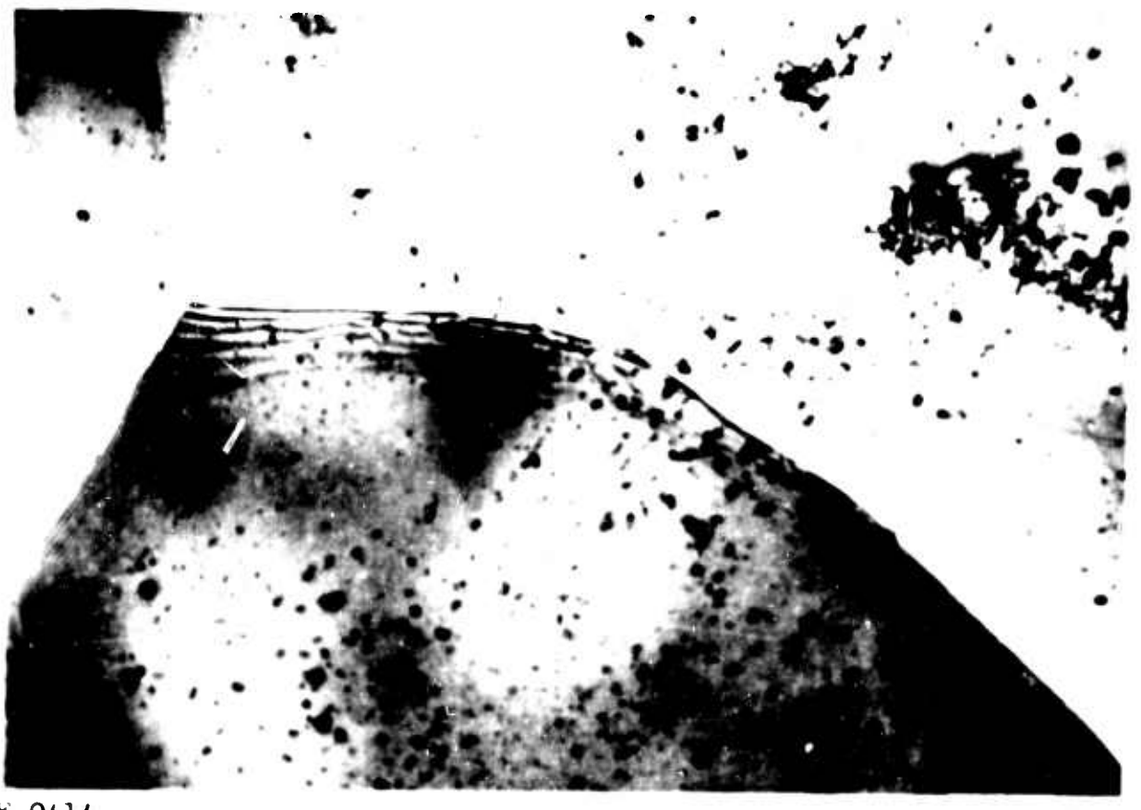
(b) - A.246, 100 Hours at 1000°C

Figure 34 - Microstructure of Thin Sections of Recrystallised SR Beryllium Powder (-50, +110)



E.9435 16,000X

(a) - A.312, 1 Hour at 800°C



E.9414 8,000X

(b) - A.326, 100 Hours at 1000°C

Figure 35 - Microstructure of Thin Sections of Recrystallised SR Beryllium Powder (-200)

E.10.225 40,000X

(a) - A.532, 1 Hour at 810°C

E.9823 16,000X

(b) - A.526, 100 Hours at 1000°C

Figure 36 - Microstructure of Thin Sections of Recrystallized QMV Beryllium Powder (-50, +110)

E.9959 16,000X

(a) - A.636, 100 Hours at 1000°C

E.9961 40,000X

(b) - A.646, 100 Hours at 1000°C

Figure 37 - Microstructure of Thin Sections of Recrystallized MV Beryllium Powder (-200)

1. Tensile Tests

a. The tensile specimens for use at room temperature are of the type T2 or T7 (Figure 38), depending on the thickness of the sheet. They were machined in two perpendicular directions of the sheet: that of rolling (L) and the transverse direction (T). The dimensions of these specimens were calculated with the purpose of utilizing the minimum amount of sheet while maintaining a ratio of usable length to cross sectional area approximately constant at a value of 10. The useful part of the specimens was milled laterally with tungsten carbide tools, then polished manually to eliminate all trace of working using metallographic papers of increasing fineness. No other treatment of the surface was carried out on the specimens before or after heat treatment in order to avoid disturbing the observation of the effect of working. The marking of the gage length was accomplished by spots of special ink and measurement of distance between spots (before pulling and after bringing together the two parts of the specimen) was done with a Huet binocular microscope to a precision of about 0.001 mm.

b. Tension is produced with a universal Instron machine (type CM) under the following conditions:

(1) Sensitivity: 100, 200, or 500 kg, depending on the specimen.

(2) Pulling speed: 0.5 mm per minute for all specimens.

(3) Amplification of recording: 5 mm/minute.

c. The properties measured are:

(1) Breaking load R referred to the cross section of the specimen before rupture.

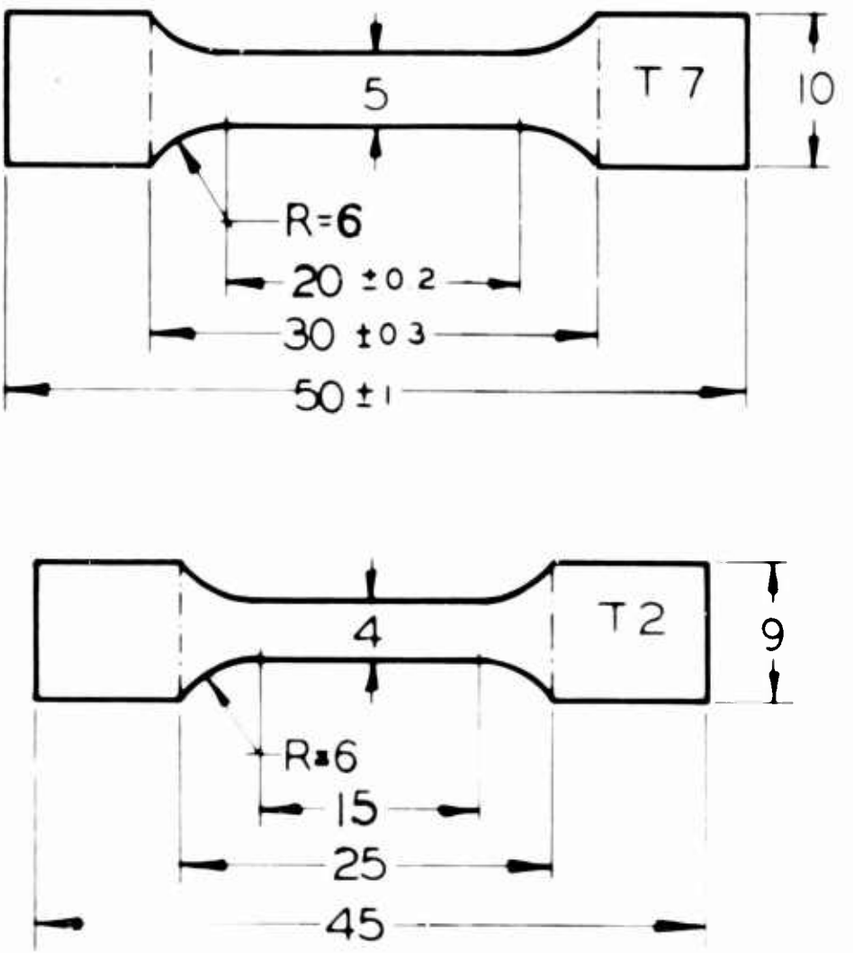
(2) Elastic limit at 0.2%, or at fracture when the range of plasticity is too small.

(3) Reduction of area at the break: $100 \frac{a_0 - a}{a}$%.

(4) Total elongation over the gage length (15 to 20 mm depending on the thickness of specimen).

2. Bending

The bend tests were carried out with a similar type of specimen (width 10 mm, length 50 mm) on which the edges have been mechanically polished under the same conditions as for the tensile specimens. The apparatus is of the three-point load type with two fixed rolls of 30 mm diameter and a punch with an end rounded to a radius of 5 mm. The distance between the axes of the rolls is 40 + 3t (t being the thickness of the specimen). The apparatus is mounted on a standard Amsler machine with a maximum sensitivity (100 kg). The application of load is effected with a minimum opening of the hydraulic valve and the test is interrupted at the appearance of the first crack corresponding to the maximum load.



NOTE: T2 = Specimen for sheet thicknesses 0.2 to 0.7 mm
T7 = Specimen for sheet thicknesses greater than 0.7 mm

Figure 38 - Room Temperature Tensile Specimens for Beryllium Sheet (Dimensions in Millimeters)

One then measures the bend angle obtained according to the following sketch:

The load causing rupture is always very small (1 to 10 kg) and has not been taken into consideration.

B. Results

We tried first to follow the evolution of mechanical properties, measured at room temperature, during the course of the high temperature processing. Our principal effort was carried out on the evaluation of warm rolled sheets which were submitted to different anneals. However, the lack of metal in certain cases or various incidents during fabrication have not permitted us to realize all the specimens that would have been desirable. Considering the customary scatter of results of mechanical tests on beryllium, we have eliminated the values which were obviously aberrations in order not to falsify the interpretation of the entire group of data; this is the reason why some of the tables appear to be incomplete.

1. Forged Condition

Some cylindrical specimens were machined from the forged plates, then annealed under vacuum for 1 hour at 800°C, and then polished electrolytically.* The results are presented in Table 10. Let us remember that the value of upsetting varied for 3.3 to 4.2 to 1.

The cast metal, with very coarse grains, is fragile and weak. The powdered metal possesses, on the contrary, improved characteristics, particularly the metal with fine grains (-200). This behavior can be related to the size of the grains and, to a large extent, to an isotropic texture.

2. Hot Rolled Condition

The specimens of type T7 (Figure 38) were subjected either to an anneal of 10 minutes at 850°C or an electrolytic polish. The results appearing in Tables 11 and 12 are the average values obtained in the two directions of rolling.

Sheet A.21, which received a light reduction (8%), is set apart.

The characteristics obtained after annealing are slightly better than after electrolytic polishing, but the anneal at 850°C has slightly modified the structures. One can verify that the hot rolled sheets were practically isotropic. The influence of purity is masked by the interdependent factors of grain size and condition of fabrication. For the cast sheets, the smaller grain size and the presence of substructure in A.40 suffices to explain the better

* Bath - 52% H_3PO_4, 16% H_2SO_4, 16% Glycerol, 16% Ethanol; Intensity - 2 amperes/cm^2; Time - 30-40 seconds.

TABLE 10. MECHANICAL PROPERTIES OF FORGED SHEETS

Origin	Type	Elastic Limit (kg/mm^2)	Rupture (kg/mm^2)	Elongation (%)
Pechiney SR	Cast	17.0	18.4	0.9
	Powder (-50, +110)	25.8	37.8	5.4
	Powder (-200)	37.8	51.7	9.9
Brush QMV	Cast	18	18.7	0.4
	Powder (-50, +110)	25	42.5	5.9
	Powder (-200)	34.6	55.0	7.7

TABLE 11. MECHANICAL PROPERTIES OF HOT ROLLED SHEETS
(AS ROLLED, ELECTROLYTIC POLISH)

Origin	Number	Elastic Limit (kg/mm^2)	Rupture (kg/mm^2)	Elongation (%)
Pechiney SR	A.10	17.2	18	1.3
	A.21	54	57	3.1
Brush QMV	A.40	25.7	34.4	4.1
	A.50	27.6	28.1	0.5

TABLE 12. MECHANICAL PROPERTIES OF HOT ROLLED SHEETS
(ANNEALED 10 MINUTES AT 850°C)

Origin	Number	Elastic Limit (kg/mm^2)	Rupture (kg/mm^2)	Elongation (%)
Pechiney SR	A.10	14.9	18.6	2.4
	A.21	32.3	45.1	8.2
	A.30	35.2	42.2	3.7
Brush QMV	A.40	23.8	33.4	4.5
	A.50	27.2	29.6	1.4

ductility and better strength of this sheet (QMV). The powder sheet of QMV is very fragile. This can be related to the abnormal grain growth which occurred during rolling.

3. Sheets Worked and Annealed

a. Tensile Tests

(1) As was done for the metallographic study, we are going to treat the case of the cast and powdered sheets separately. The results, reported in Table 13, have been presented in the form of graphs. Figures 39, 40, and 41 show, after each thermal treatment, the mechanical properties (L and T) as a function of the previous reduction. Figure 42 shows, for an average reduction, the properties of the different sheets in the longitudinal direction as a function of the temperature of anneal. We shall try to separate the influence of the different parameters: anisotropy, reduction, thermal treatment, and purity.

(a) Cast Metal

1. Anisotropy of Rolling (See Figure 39)

In every case, the longitudinal properties are significantly higher than the transverse properties. In general, the anisotropy is more extreme as the initial reduction is greater and as the annealing temperature is higher and as the length of the anneal is longer. The SR sheets are, all other things being equal, more isotropic than the QMV sheets.

2. Influence of Reduction (See Figure 39)

One can examine for a given treatment the influence of the magnitude of the initial reduction on the mechanical properties of the annealed sheet. For the SR sheet, except for the recovery anneal, the three properties become higher as the magnitude of reduction is greater. One can relate this behavior to the decrease of the grain size parallel to the working direction. For the QMV sheets, on the contrary, the mechanical properties are not influenced significantly by the initial reduction.

3. Influence of Thermal Treatment (See Figures 39 to 42)

a. The recovery anneal* leaves the SR metal in a very fragile condition, while it leads to a notable ductility in the QMV sheets, as is shown by the summary results in Table 14. The duration of the anneal was perhaps too short for the SR metal, in consideration of the low temperature of 600 to 630°C. The effective recovery of the mechanical properties of the QMV sheets could be due to the more elevated temperature of 670°C which permitted a more complete polygonisation and could be, on the other hand, caused by the fine precipitation observed in the thin sections.

b. The recrystallisation anneal of one hour** leads to better ductility and a decrease of elastic limit as shown in Table 15. The results are more uniform on the QMV sheet.

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* 1 hour - Temperature 600 to 670°C, see Table 8.
** TR = 700 to 775°C, see Table 7.

TABLE 13. TENSILE PROPERTIES OF WORKED AND ANNEALED SHEETS

Origin	Total Reduction, %	Number		Recovery: 1 Hour at TR -100° (1) LE	R	A	Recrystallisation: 1 Hour at TR (2) LE	R	A	Recrystallisation: 10 Hours at TR (3) LE	R	A	Grain Growth: 1 Hour at TR +200° (5) LE	R	A	Grain Growth: 100 Hours at TR +200° (6) LE	R	A
Cast SR	50	A.11	T	23.6	23.6	0.2	19.5	22.4	1.0	15.4	18.3	1.9	11.9	13.4	1.5	7.1	7.2	0.7
			L	30.0	31.0	0.5	19.3	27.7	3.3	—	—	—	--	—	—	9.8	10.1	1.1
	100	A.12	T	29.6	29.6	0.1	15.0	18.1	1.5	14.4	16.8	1.4	11.1	11.6	1.0	8.3	8.6	0.8
			L	35.2	36.1	0.64	15.7	30.9	4.8	16.4	29.8	4.7	13.7	20.8	2.3	12.4	14.9	1.4
	180	A.13	T	26.6	28	1.0	17.2	23.3	2.1	17.5	21.4	2.1	13.2	15.9	1.6	10.9	11.0	1.1
			L	25.6	37	3.6	18.7	34.4	4.1	—	—	—	17.5	27.5	3.0	16.5	19.7	1.8
	255	A.14	T	20.8	24.1	1.5	18.3	25.5	2.4	16	18.6	1.7	--	--	—	9.1	9.5	1.2
			L	31.0	31.4	0.4	21.5	40.4	5.6	—	—	—	—	--	--	15.4	21.7	2.3
SR Powder (-50, +110)	50	A.22	T	54.6	56	2.1	23.8	39.8	5.75	22.9	40.2	8.3	21.8	38.4	5.9	25.5	41.7	6.3
			L	54	54	0	24.9	42.3	5.9	--	--	—	24.5	42.4 (23)	7.2 (0.5)	24.3	38.5	3.8
	80	A.23	T	57	57.5	1.8	28.4	41.5	6.0	25.5	41.1	8.2	--	--	--	24.6	36.7	3.5
			L	53.0	53.2	0.25	--	—	—	--	—	--	--	--	--	24.9	38.7	4.3
	235	A.24	T	--	39.6	0	27	42.0	5.3	27.3	41.3	5.6	--	—	—	22.2	23.9	0.6
			L	45.6	45.8	0.5	29.8	44.3	5.5	30.8	47.1	10.7	—	—	—	--	--	—
SR Powder (-200)	50	A.31	T	54.2	54.2	0.15	--	30.6	0.7	37	37.2	0.4	29.4	29.4	0.39	26.1	26.1	0.34
			L	58.5	59.5	0.34	—	29.7	0	18	18.6	0.1	—	--	—	24.5	36.1	3.4
	100	A.32	T	51.8	52.5	0.36	25.4	39.5	3.2	28.7	41.2	3.9	23.4	31.6	2.36	23	34.3	2.8
			L	57.0	57.8	0.32	28	45.8	10.6 (0.5)	30.6	35.2	1.4	28.2	28.2	0.58	25.8	37.2	2.8
	160	A.33	T	59.8	59.4	3.9	29	42.5	6.9	—	—	—	—	--	—	24	40.7 (21)	5.4 (0.4)
			L	55.3	56.3	0.9	35 (29)	47.5	12.8 (1)	—	—	—	—	—	—	26.4	30.2	1.1
	225	A.34	T	--	54	0.1	30.2	42.9	6.0	--	—	—	—	—	--	26.5	31.2	1.8
			L	--	45	0.1	30	44	6.4	28.8	45.7	11.2	—	—	--	27.4	30.2	0.9

TABLE 13. TENSILE PROPERTIES OF WORKED AND ANNEALED SHEETS (Continued)

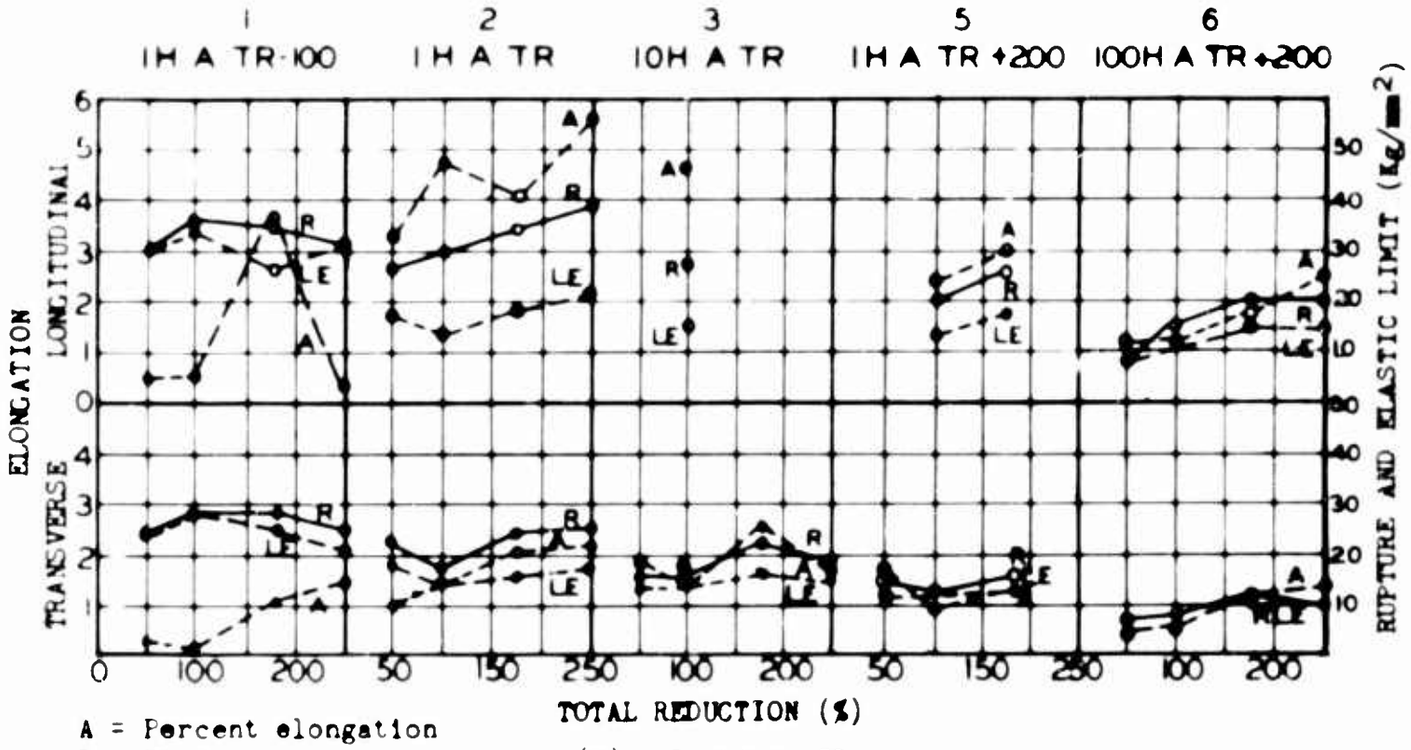
Origin	Total Reduction, %	Number		Recovery			Recrystallisation						Grain Growth					
				1 Hour at TR -100°			1 Hour at TR			10 Hours at TR			1 Hour at TR +200°			100 Hours at TR +200°		
				1			2			3			5			6		
				LE**	R	A	LE	R	A	LE	R	A	LE	R	A	LE	R	A
Cast QMV	50	A.41	T*	34.4	40.9	2.5	21.5	34.0	4.7	17.3	29.9	4.3	17.0	20.5	1.9	16.9	16.9	0.7
			L	35.9	44.1	3.7	21.7	36.2	5.8	17.7	31.9	4.5	17.6	21.9	1.4	16.5	16.6	0.6
	100	A.42	T	34.3	39.8	2.0	21.7	32.7	3.9	17.7	28.5	4.1	16	18.3	1.2	14.6	14.6	0.4
			L	35.3	43.0	3.2	21.4	34.5	4.7	18.3	35.3	5.3	17.2	23.5	2.2	18.5	20.5	1.3
	180	A.43	T	32.9	41.4	3.6	18	29.7	3.4	17.6	32.0	5.3	15.5	18.2	0.95	11.5	11.6	0.6
			L	33.7	47.2	4.6	20.3	31.8	3.9	20.1	35.1	4.4	18.1	24.7	2.9	18.8	21.9	1.4
	24[illegible]	A.44	T	33.1	42	5.2	17.1	32.6	5.3	17.8	32.2	4.4	15.7	17.5	1.0	17.8	13.9	0.6
			L	33.2	45.8	3.8	19.1	35.9	6.4	20.3	39.1	6.4	18.3	23.6	2.5	18.3	21.9	1.5
QMV Powder (-50, +110	50	A.51	T	16.7	16.7	--	22.4	28.9	2.2	--	--	--	--	--	--	17.6	22.3	1.5
			L	--	--	--	19	19	0.4	--	--	--	23.3	23.3	0.9	23.7	28.9	3
	100	A.52	T	32.5	32.5	0.1	20.7	24.4	1.8	20.8	24.8	1.6	18	19.1	1.1	19.9	22.8	2.0
			L	34.7	34.7	0.1	20.8	28.8	2.3	21.8	31.1	2.6	21	27	2.3	20	21.3	1.2
	180	A.53	T	19	19	0	21.3	24.3	1.3	18.1	24.6	1.3	21.8	22.5	0.2	20.0	22.6	1.6
			L	37.5	37.5	0.1	22.1	28.6	1.7	22.9	35.5	4.3	23.4	23.4	0.6	25.5	30.4	3.4
	250	A.54	T	24	24	0	11.7	20.6	0.6	18.8	23.0	1.5	18.6	26.8	1.9	17.8	19.8	1.1
			L	41.6	41.6	0	21.4	38.9	5.2	22.7	36.8	0.5	22.4	36.6	5.3	20.0	25.7	1.8

* T = Transverse L = Longitudinal

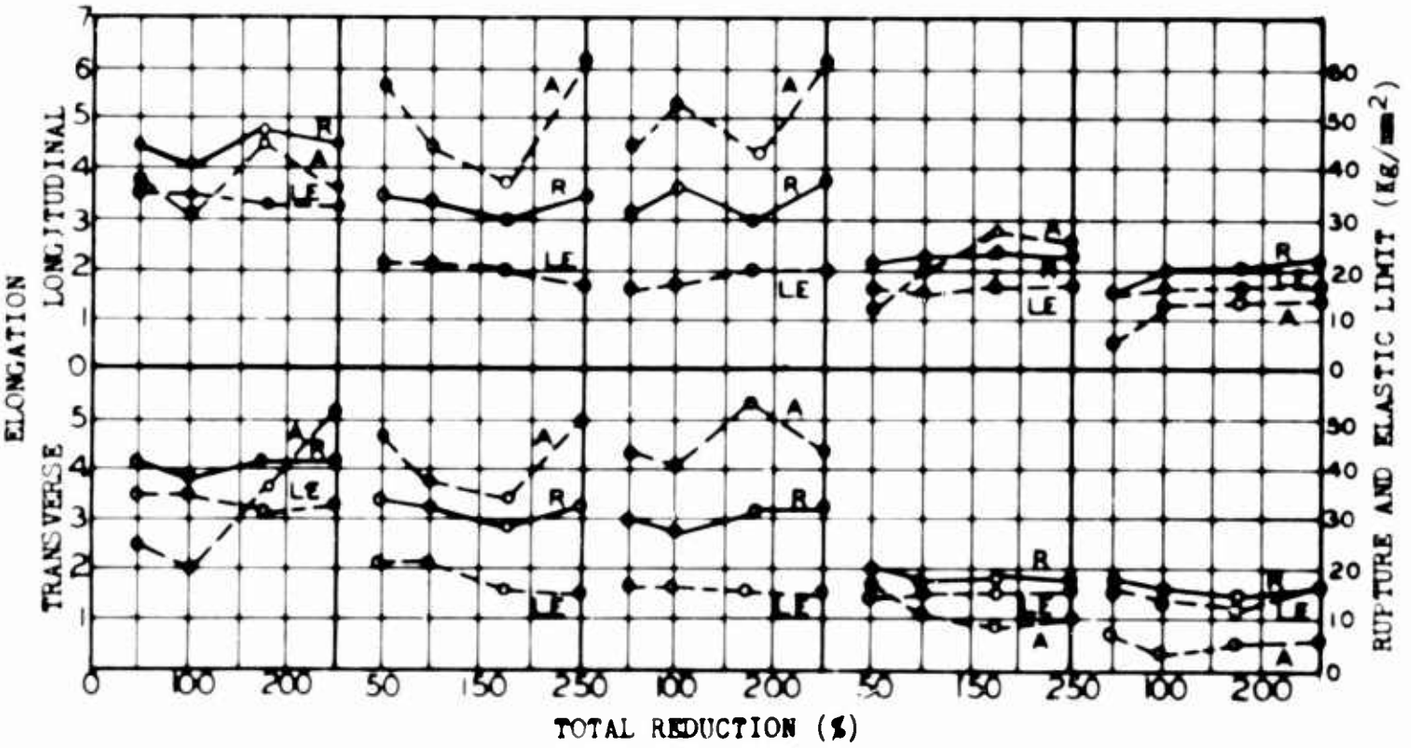
** LE = 0.2% Elastic Limit (Kg/mm^2)
R = Rupture (Kg/mm^2)
A = Percent Elongation

TR = 700°C for sheets A.13 and A.14 (cast SR)
TR = 740°C for sheets A.11 and A.12 (cast SR)
TR = 800°C for sheets A.21-A.24 and A.31-A.34 (powder SR)

TR = 760°C for sheets A.43 and A.44 (cast QMV)
TR = 775°C for sheets A.41 and A.42 (cast QMV)
TR = 810°C for sheets A.51-A.54 (powder QMV)

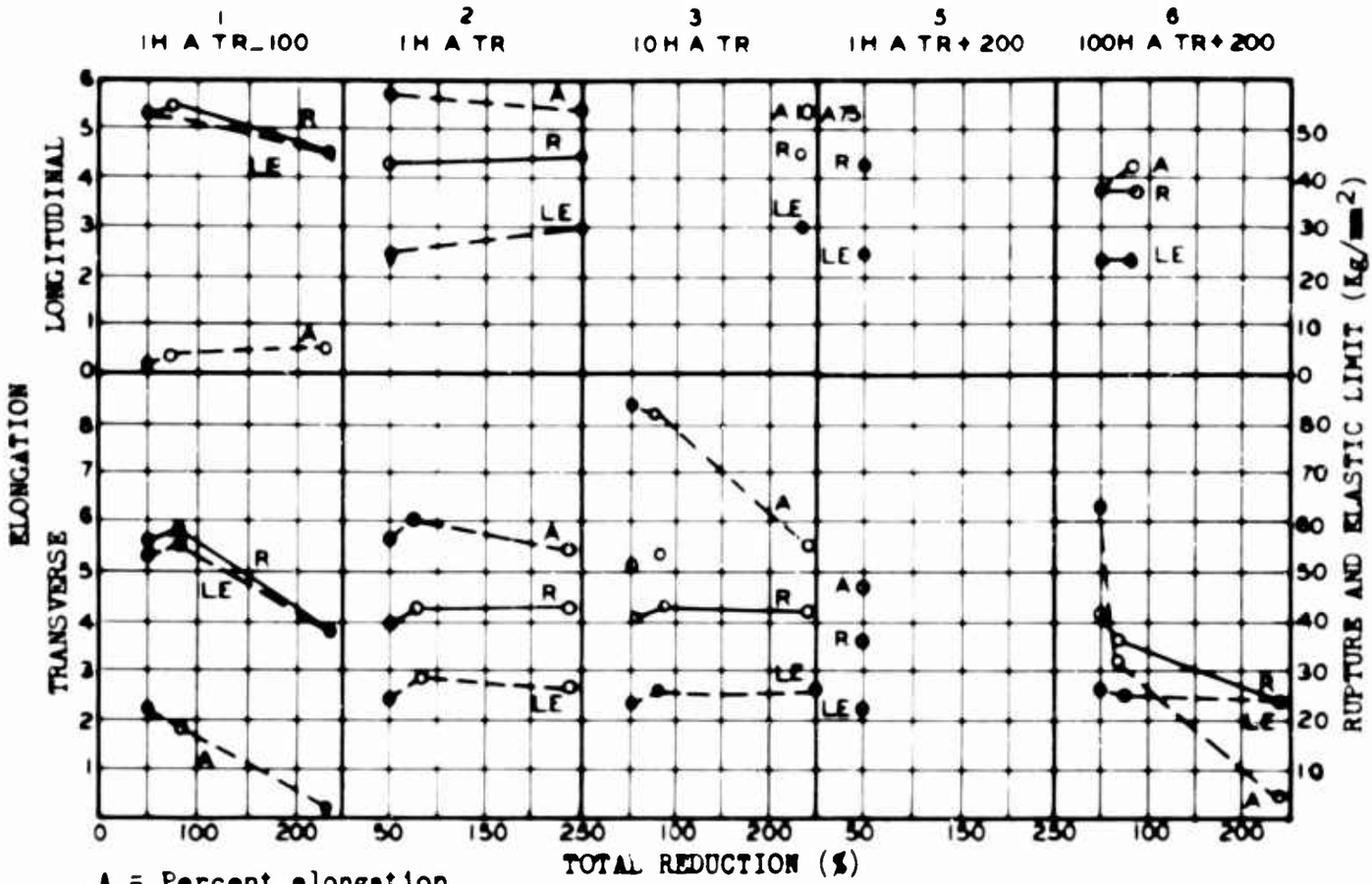


(a) - Pechiney SR

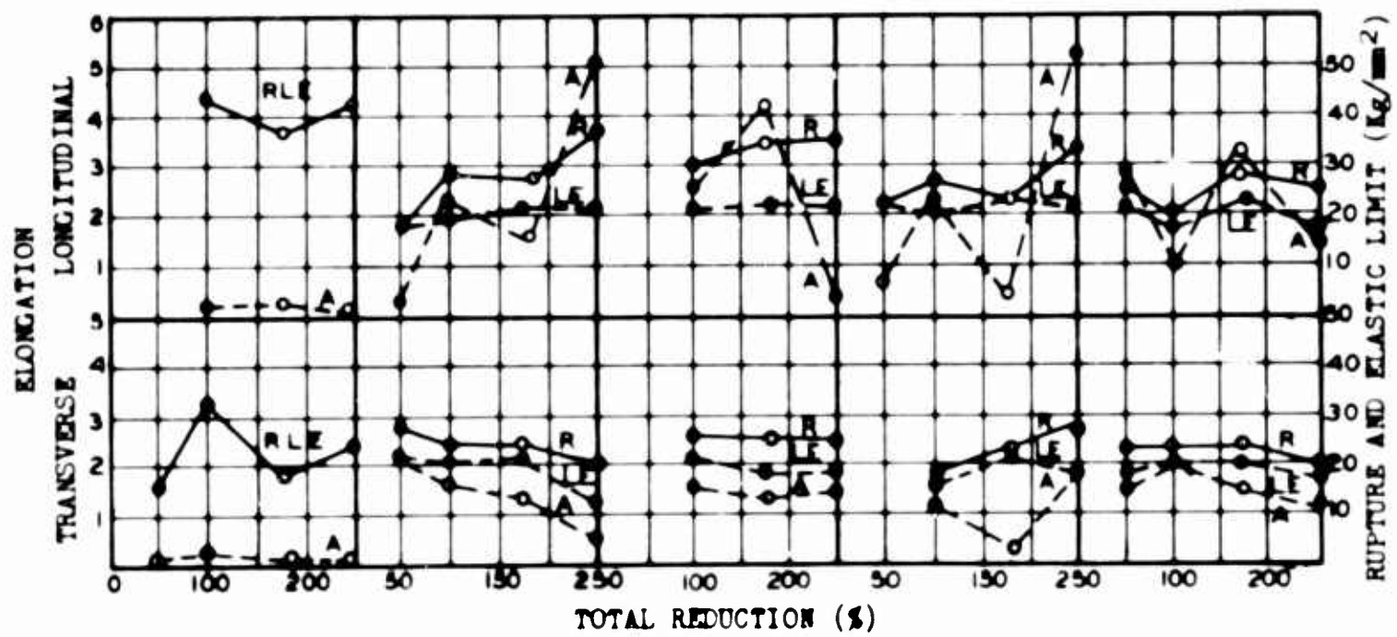


(b) - Brush QMV

Figure 39 - Mechanical Properties of Cast Beryllium as a Function of Reduction (Tensile Tests)

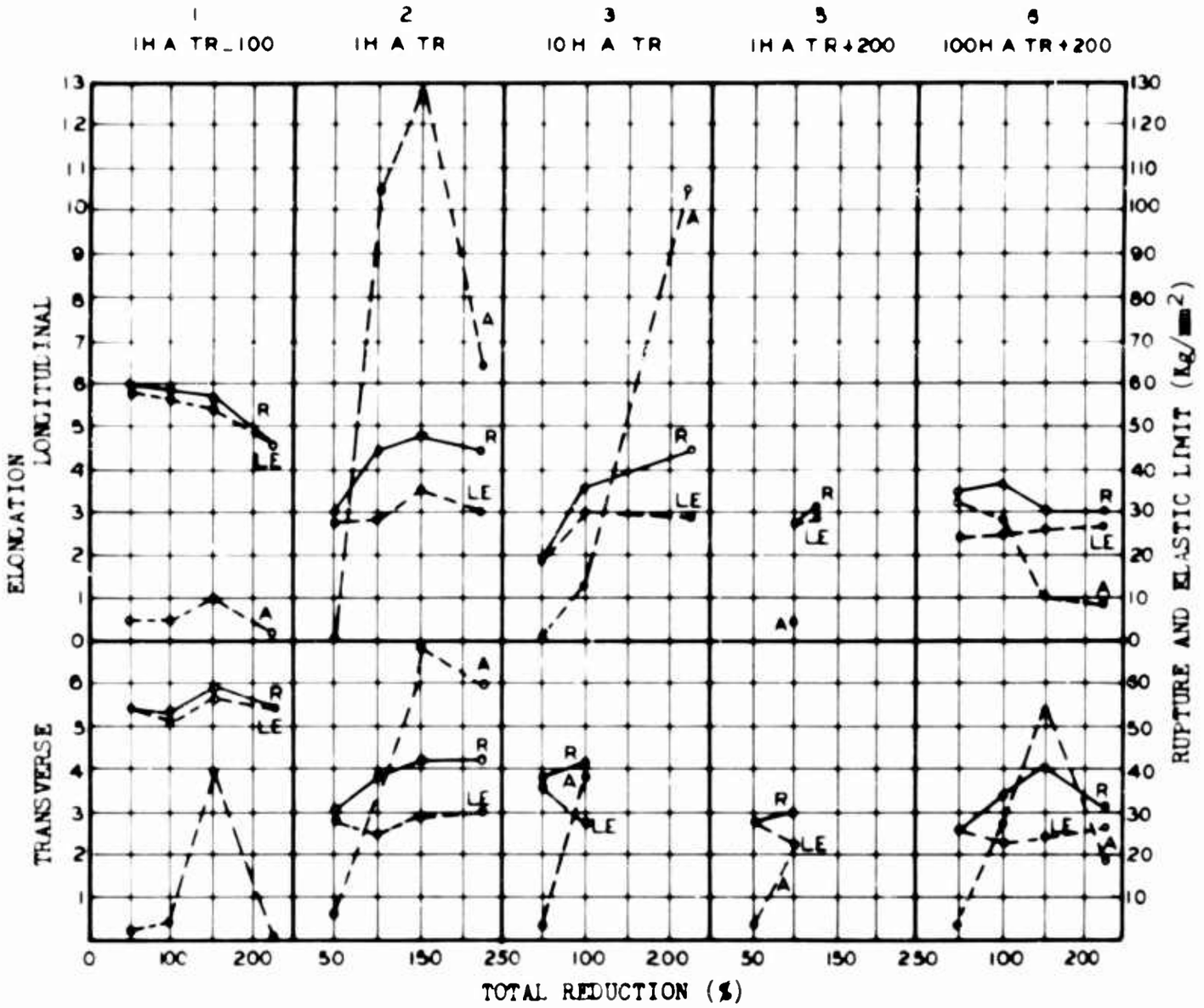


(a) - Pechiney SR



(b) - Brush QMV

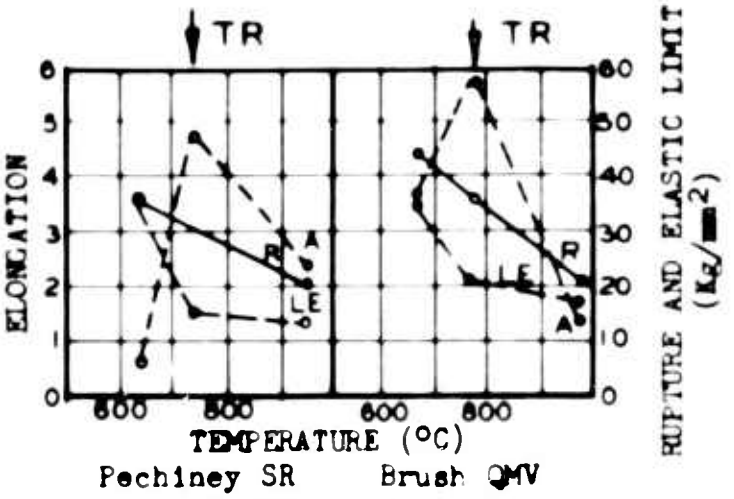
Figure 40 - Mechanical Properties of Powder Metal (-50, +110) as a Function of Reduction (Tensile Tests)



A = Percent elongation
R = Rupture
LE = Elastic Limit (0.2%)

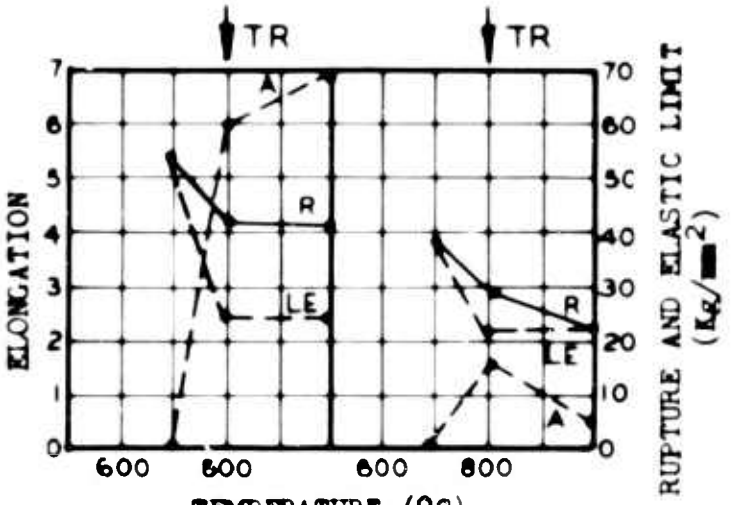
Pechiney SR

Figure 41 - Mechanical Properties of Powder Metal (-200) as a Function of Reduction (Tensile Tests)



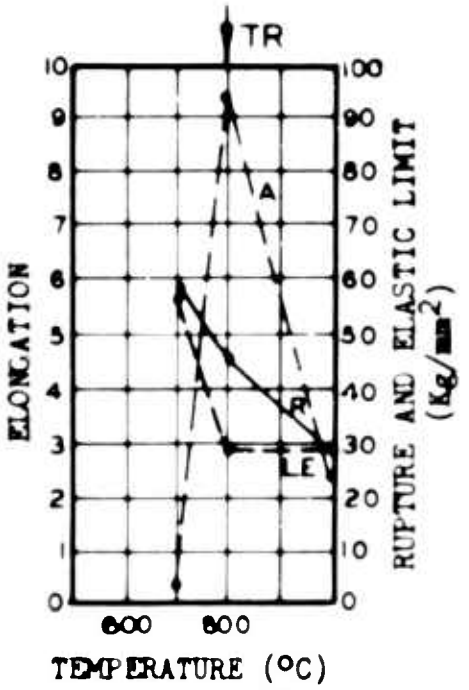
Pechiney SR Brush QMV

(a) - Cast



Pechiney SR Brush QMV

(b) - Powder (-50, +110)



Pechiney SR

(c) - Powder (-200)

A = Percent elongation
R = Rupture
LE = Elastic Limit (0.2%)

Figure 42 - Comparison of Mechanical Properties of SR and QMV Cast and Powdered Sheets as a Function of Reduction (Tensile Tests)

TABLE 14. MECHANICAL PROPERTIES OF WORKED AND ANNEALED CAST SHEETS (RECOVERY ANNEALED

Metal	Elastic Limit (Kg/mm^2)	Rupture (Kg/mm^2)	Elongation (%)
SR	24 to 35	24 to 36	0.2 to 1.5 (3.6)
QMV	33 to 36	40 to 47	2 to 5

TABLE 15. MECHANICAL PROPERTIES OF WORKED AND ANNEALED CAST SHEETS (RECRYSTALLIZATION ANNEALED)

Metal	Elastic Limit (Kg/mm^2)	Rupture (Kg/mm^2)	Elongation (%)
SR	15 to 21	18 to 40	1 to 5.6
QMV	17 to 22	33 to 36	3.4 to 6.4

c. Holding 10 hours at the temperature TR improves the ductility a little at the price of a slight reduction of the elastic limit.

d. The anneals at high temperature* cause a definite deterioriation of all properties. This behavior seems to be related above all to the large grain growth.

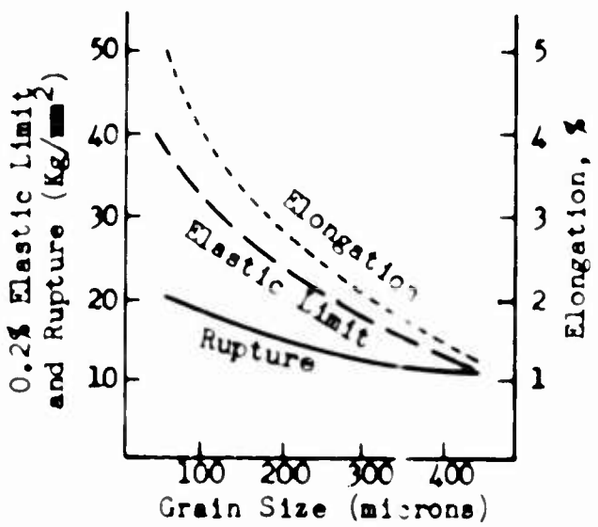
4. Influence of the Impurity Level and of the Size of the Grains

a. The level of impurities makes itself apparent in two ways:

(1) Indirectly by the effect on the grain size.

(2) Directly by the phenomenon of the hardening in solid solution or by precipitation.

For the SR metal, if one considers all the recrystallized sheets with large grains, one determines, by plotting the mechanical properties as a function of the grain size (as shown below) that there exists a correlation independent of annealing between grain size and mechanical properties. The grain size factor seems therefore to be the essential parameter for metal of high purity.



b. For the QMV metal, one finds for the sheets recrystallized at 760 or 775°C a value for the mechanical properties independent of the size of the grains and of the reduction. On the other hand, the recrystallized SR sheet reduced 250% and the recrystallized QMV sheet reduced

* 900 to 970°C, see Table 8.

50% have an average grain size of 45 microns and identical mechanical properties. A disappearance of the effect of the grain size below 45 microns in Brush metal seems to be improbable. It is therefore reasonable that the improvement of properties related to the decrease of grain size is compensated by a phenomenon which decreases the three properties as the reduction increases. It seems reasonable to us to think that this phenomenon is related to impurities. We shall recall here that one observes in recrystallized QMV metal a precipitation of fine constituents. On the other hand, after the anneal at high temperature, the very low properties cannot be interpreted uniquely as the effect of grain growth in the QMV metal. It is possible that the poor surface condition, produced by evaporation or oxidation of metal, played an equal role.

(b) Powdered Metal

1. Interpretation of the results is made difficult because of the scatter of the results and because of premature fracture of the specimens (internal flaws, or serration in the grips).

a. Anisotropy Due to Rolling (See Figures 40 and 41)

Anisotropy is not very marked in the SR metal of fine grain size and it appears principally in the elongations. On the other hand, anisotropy is definite for the QMV metal and it is accentuated with the reduction.

b. Influence of Reduction (See Figures 40 and 41)

It is hardly possible to separate a significant influence of the amount of reduction on the mechanical properties of the annealed sheets. In view of the limited number of specimens, particularly on the SR metal (-50, +110), and of the frequent premature failure (internal flaws or serration in the grips), one can only discern in the SR metal (-200) a small increase of the elastic limit and of the rupture strength as the reduction increases. This could be the consequence of the parallel reduction of the grain size. On the other hand, the large reductions manifest themselves by a formation of microcracks or microfissures at the network of beds of oxide. This causes the premature ruptures to become more numerous.

c. Influence of Annealing (See Figures 40, 41, and 42)

(1) The recovery anneal* leaves the metal very fragile with the highest values for breaking strength and elastic limit (45 to 60 kg/mm^2 for SR).

(2) The recrystallization anneals** give an interesting ductility for the SR material, which is less for QMV, which is expressed as a very pronounced lowering of the elastic limit (Table 16).

(3) The anneal at high temperature (1000°C) lowers slightly the elastic limit and reduces the ductility. It accentuates

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* 1 hour at 700 or 710°C, see Table 8.
** 1 hour and 10 hours at 800 or 810°C, see Table 8.

the internal flaws (cracks or fissures) related to oxide under the effect of thermal strains. On the other hand, the state of the surface is poor (oxidation or evaporation). These two factors provoke a large number of premature failures which makes the interpretation of results questionable. It appears to us, after detailed examination of the specimens, that the powdered SR beryllium (-50, +110) preserves its strength and even has its ductility increased by the anneal of 1 hour at 1000°C. On the other hand, the SR beryllium (-200) has always given very small elongations.

d. Comparison Between SR and QMV Metal

(1) The QMV sheets have, in the aggregate, a very low ductility and an average strength. It is necessary above all to see the important effect of the grain size: each grain is crossed by a large number of beds of oxide which multiply the number of possible sources of transgranular rupture. The influence of the grain size on the properties after an anneal is perhaps masked, as has been proposed for the cast metal, by a phenomenon related to impurities. The decrease of ductility after annealing at high temperature does not correspond to any increase of grain size and is perhaps a consequence of taking into solution certain impurities.

(2) The recrystallized SR sheets have given the highest properties. The grain size factor makes it possible to interpret to a large extent these values. The metal with finest grain size (-200) has the best properties, but the scatter of results and the frequency of premature failures is greater.

b. Discussion of Bend Tests

(1) The interpretation of the results of the bend tests is rendered particularly delicate by the large number of parameters and the habitual scatter of results (angle of bend measured at the appearance of the first crack). For a punch of a given radius and the same width of specimen, the parameters are the following:

(a) Thickness of specimen

(b) Reduction

(c) Thermal treatment

(d) Grain size

(2) These parameters evidently are not independent. Furthermore, the mode of testing the specimen is not the same, depending on the relative dimensions of the specimen:

(a) For a relatively narrow specimen (width/thickness < 10), the strains are essentially uniaxial (in a transverse direction relative to the direction of rolling).

(b) For a wide specimen (width/thickness > 15), the strains are multiaxial.

(3) The observations that one can make based on the results of Figure 43 are the following:

(a) Cast Metal

1. The bendability of SR beryllium appears in the aggregate to be superior to that of QMV beryllium, regardless of the thermal treatment. The angle of bend increases in significant fashion as the reduction increases for each of the treatments. It increases likewise (particularly for SR) with the grain size at equal reductions. These two apparently contradictory phenomena show that the favorable factor is probably the geometric factor (radius of punch/thickness) which increases in proportion to the decrease of thickness.

2. A recovered state is confirmed as being the most fragile. The recrystallization treatment gives the best ductility to QMV beryllium. The treatment at high temperature which causes a grain growth of the worked sheets is favorable for SR metal but not for QMV metal because, without doubt, of the return to solid solution of certain precipitates.

(b) Powdered Metal

1. The results obtained are more irregular and different from those of cast metal for the same conditions of rolling and thermal treatment:

a. The degree of thickness of the specimen is not favorable in all cases for SR powder (-50, +110).

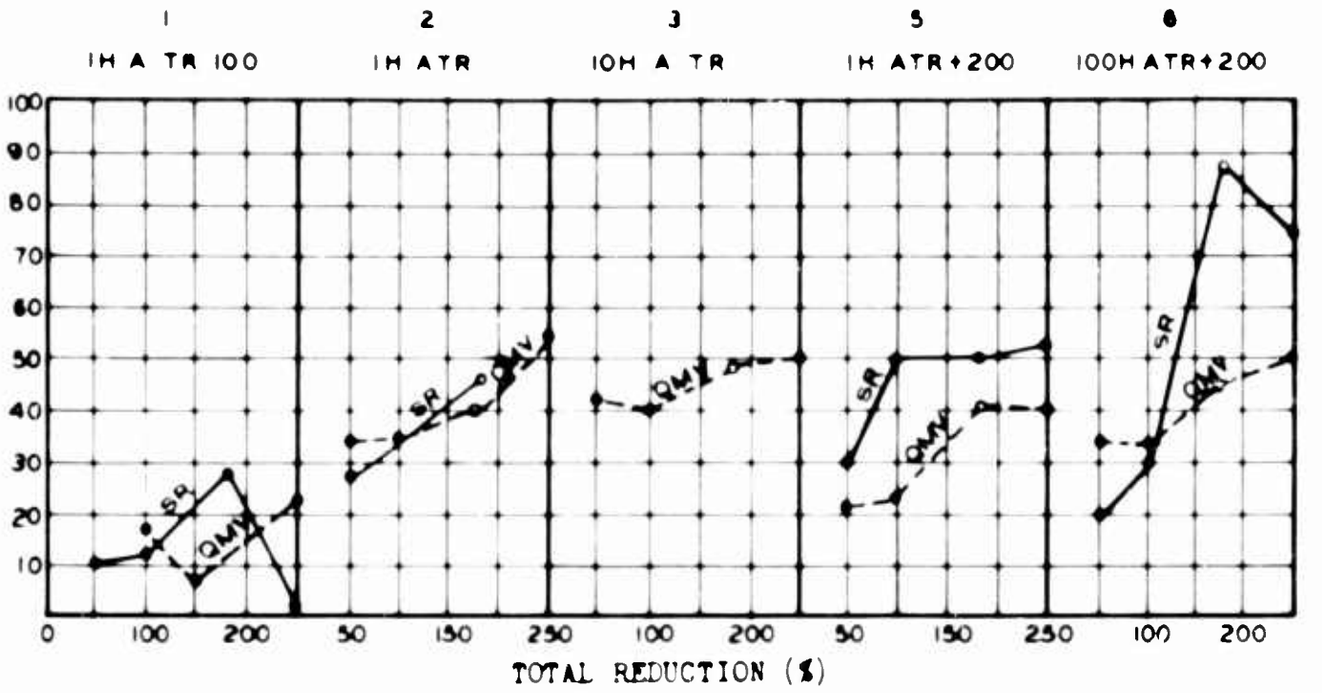
b. The recrystallization treatment gives the largest bend angles for the two types of material, while the grain growth treatment appears to be very unfavorable this time for the SR sheets.

2. A correct interpretation of these phenomena would require many more bend specimens and would need to take into consideration the state of the surface, the shape of the grains, and equality of texture.

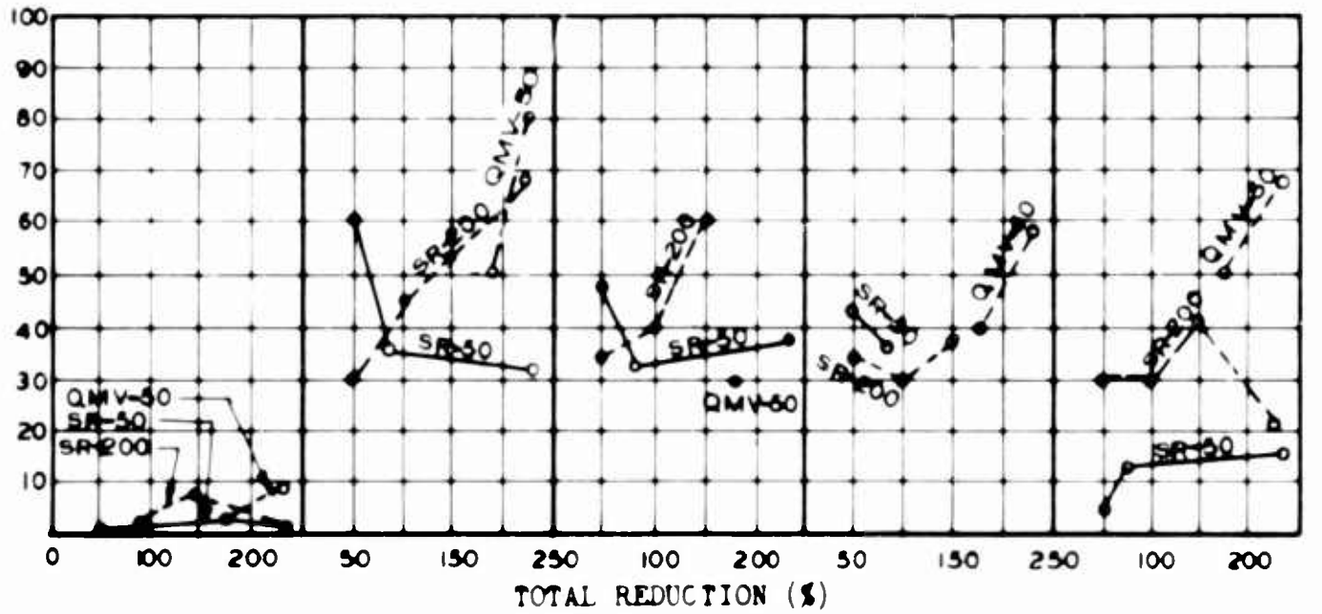
V. SUMMARY AND DISCUSSION OF RESULTS

We shall first try to present some general remarks on the distribution of oxide and of impurities before seeing if it is possible to interpret the behavior of the metal with the aid of this information. The initial billets show these differences:

(a) With regard to the distribution of oxide, between the cast metal in which there is no dispersed oxide (except perhaps in association with the large metallic inclusions) and the powdered metal containing a cell-like network corresponding to the film of oxide which covers the grains of powder. The



(a) - Cast SR and QMV



(b) - Powder SR and QMV

Figure 43 - Bendability as a Function of Reduction

distribution and the amount of oxide introduced by powdering are identical in the SR and QMV billets.

(b) With regard to the distribution of impurities between the very pure SR metal, in which the elements are practically all in solid solution, and the QMV metal, where a good part of the impurities are assembled in inclusions of several micron size, the rest being in solid solution.

The operations of forging and of hot rolling leading to a deformation of 30 to 60 to 1 profoundly modify the distribution of the oxide. The cocoons of oxide which surround the grains of powder are crushed and broken. The oxide finds itself dispersed in very fine beds, in the form of crystalline platelets, of several hundredths to several tenths of microns, very sparsely distributed except for some agglomerates. This can be understood readily since the applied deformation increases the surface of each grain of the sintered billet by a factor of 10 to 20.

The large metallic inclusions of the QMV metal are little modified by the mechanical or thermal treatments if there are no eutectic fusions. On the other hand, the thermal treatments at temperatures between 650 and 800°C lead to a precipitation of fine inclusions (of the order of 1/10 micron or finer) starting from the solid solution in the QMV metal. The purity of the SR metal does not permit such precipitations. Finally, the treatments at high temperature cause the formation in the grain boundaries of lenticular constituents rich in aluminum.

The growth of the impurity networks, and/or the amount of oxide, notably decreases the hot plasticity of beryllium. It has therefore been necessary to modify the working conditions at high temperature in going from SR to QMV. These modifications have played an important role:

(a) For the cast metal where, joined with the difference of purity, they have led to a deformed structure of fine grains in QMV and a recrystallized structure of distinctly coarser grains in the SR.

(b) For the powdered metal, where they have caused a very important grain coarsening in the QMV sheets. Since it has never been possible to reproduce such grain coarsening by working and annealing, it seems that the simultaneous application of mechanical strains and of elevated temperature (850°C) enormously facilitates the migration of boundaries.

The warm rolling works the metal while producing, in every case, a very heterogeneous localised deformation essentially at the periphery of coarse grains which are only lightly deformed.* These grains consist of sub-grains slightly misoriented containing a few dislocations. In the regions of large deformation, deformed blocks can be distinguished in the middle of dense veins of dislocations. The extensions of these zones and the distribution of dislocations depend more on the grain size of the hot rolled sheet than on the purity of the metal. The presence of oxide platelets does not disturb or

* The presence of these well differentiated lightly deformed grains allows us to speak, in the following, of a grain size in the worked state.

exaggerate the structure unless this is in making more difficult the formation of regular blocks of substructure in the regions where the density of oxide is greatest.

The temperature of recrystallization for anneals of 1 hour is increased, at equal reduction, as the purity diminishes or when one passes from cast to powdered metal. There is no difference in recrystallization temperature for the three sheets made from powder. The anneals of 1 hour at 100°C below the temperature TR were too short in general to restore the mechanical properties of the sheets which thus remained very fragile, except for the sheets of cast QMV (under the combined effects of a higher anneal temperature than for SR, of fine grains, and perhaps of a phenomenon associated with the precipitation of fine constituents). This treatment caused a polygonization in cells reaching very diverse degrees of perfection, by rearrangement of dislocations in the existing cells, then growth of these sub-grains by coalescence or migration of sub-boundaries. The elimination of dislocations appears to be more difficult in the powdered metal where the particles of oxide exert a restraint.

The recrystallization of beryllium appears to us to be the result of the competition of two processes operating simultaneously:

(a) Primary recrystallization by nucleation and growth at the expense of lightly deformed grains.

(b) Reorganization in situ, by polygonization and coalescence of sub-grains, in these large grains.

The final outcome of this competition depends largely on the amount of the regions of high deformation relative to the amount of lightly deformed grains; that is to say, in fact, to the size of the grains of the hot rolled sheet. We have determined, by hot optical microscopy, that the higher level of impurities in the QMV metal retards notably the migration of boundaries; but it is probably that the two processes are retarded in the same proportion. On the other hand, in the powdered metal with fine grain size (SR), the number of nuclei seems to be reduced compared to the size of the grains, perhaps because of the reduced formation of zones of large deformation. In the cast SR and the powdered QMV (-50, +110) with large grains, the two processes of recrystallization have a comparable importance. In the cast QMV, with initially a medium grain size, the first process clearly predominates. It is important to note that the powdered QMV metal (-50, +110) has recrystallized following the same processes as the cast, in spite of the presence of numerous beds of oxide crossing each grain. The particles of oxide therefore do not constitute, during recrystallization, insurmountable barriers to the migration of boundaries, but only restraint. Nevertheless, they contribute to the formation of an irregular shape to the grains because of a certain number of anchorages. Finally, for the SR metal with finer initial grain size, it seems that the recrystallization occurs uniquely by the first process. In the series of sheets, the grain size of the recrystallized metal is coarser than the initial grains. This could come about through a reduced number of nuclei compared to the size of the grains. The finer grain size of the sheet containing the most oxide (-200) could result from the increased number of nuclei as well as through the

restraint exerted by the particles of oxide. It appears that the level of impurities and the presence of oxide plays mostly an indirect role in the recrystallization through their previous effect on the grain size of the hot rolled material.

After examination with electron transmission microscopy of a large number of thin sections, it now appears reasonable to state that the primary recrystallization nuclei are nothing other than certain blocks of substructure in the worked state which, after having eliminated their internal dislocations, can grow thanks to a sufficiently large disorientation relative to their surroundings.

The high temperature anneals cause more important differences to appear between the cast and powdered metal. In the cast metal, there is produced a very important grain growth. The phenomenon is more pronounced in the SR metal where only the thickness of the sheet seems to limit the size of the grains, than in the QMV beryllium where the impurities impede the migration of the boundaries and even block them. Grain growth occurs in a reduced amount in QMV and leads to a much finer grain size. The blocking could result from an anchoring by the inclusions or from a draining of impurities by boundaries to an extent that the concentration becomes sufficient to immobilize them. The presence of numerous constituents in the boundaries after a new anneal of several hours at 700°C seems to attest to this latter hypothesis.*

In the powder metal, on the contrary, one can cause only a small grain growth even by annealing for 100 hours at 1000°C. This greatly reduced grain growth after recrystallization results from an anchoring by the oxide. We have shown that only a small part of the oxide plays a role in this anchoring of the boundaries which can be caused either by agglomerates of particles or by the fine particles. After recrystallization, the major part of the oxide is found dispersed in the interior of the grains. It must be noted that in spite of the five-fold greater oxide content of the sheet (-200), the sheets of powdered SR originating from medium sized or fine powder have essentially the same grain size.

The mechanical properties depend on the structural condition and the level of impurities either directly or indirectly (through the influence of the grain size). In the cast metal, the highest tensile properties are obtained with a fine grained recrystallized structure. A correlation independent of thermal treatment between grain size and tensile properties has been established for the SR metal. In the QMV metal, the dependence of properties as a function of the working necessitates the hypothesis of a phenomenon related to impurities which lower the properties as the working increases, annulling the effect of grain size. For the powdered metal, it must be pointed out that the forged plates have interesting tensile properties which one no longer finds after rolling. This must depend on the texture.

The worked QMV beryllium remains fragile and rather weak regardless of the thermal treatment because of the abnormal grain size. For the worked SR metal, recrystallization develops higher properties than for the cast metal, which

* See ASD-TDR-62-509 Volume VII

again can be attributed to the important role of the reduced grain size. The metal containing the most oxide (-200) apparently has the best properties (without doubt because of the somewhat finer size of its grains) but contains a large number of internal defects (flaws or cracks) associated with the oxide which, especially after a heavy reduction or anneal at high temperature, leads to a number of premature failures.

VI. CONCLUSIONS

The revelation, in the different qualities of metal, of the influence of the distribution of oxide, and of the level of impurities on the mechanisms of recrystallization and grain growth of beryllium, is distorted by the differences of structure at the end of the high temperature conversion operations, demonstrating the fundamental importance of these fabrication conditions and of the impossibility of realizing identical conditions in products which do not have the same hot plasticity. We have been able, nevertheless, to separate out the following results. In the cast metal, the level of impurities controls the growth and the size of the grains in all the conversion operations or during the anneals. The size of the grains is an essential parameter for the tensile properties which, in general, are all increased, and particularly the ductility, when one makes the grain size finer. In the powder metal, the cocoons of oxide which surround each grain of powder have been completely dispersed into beds of fine particles sufficiently dispersed by the substantial reductions which were employed (50 or 60 to 1). The particles of oxide do not present an insurmountable resistance to the motion of boundaries since one has obtained:

(a) An important grain growth during rolling at elevated temperature.

(b) A growth of nuclei across the beds of oxide during primary recrystallization of the worked powder sheets.

The oxide particles constitute a restraint which does not permit increase of grain size by annealing at high temperature of a recrystallized sheet.

Only a portion of the oxide serves in the blocking of the boundaries. For this reason, a ratio of 1 to 5 in the content of oxide of the powdered SR metal which was initially powder of -50, +110 mesh or -200 mesh is far from being reflected by the slight difference in the resulting grain size. The favorable effect by the reduction of grain size, obtained by increasing the oxide content, on the mechanical properties and particularly on the ductility of powdered beryllium is greatly attenuated by the multiplication of internal defects.

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13. ABSTRACT

The objective of this program was to study the influence of distribution of oxide and of the total impurity level on the recrystallisation and grain growth of beryllium.

The starting material was either Pechiney SR beryllium or Brush QMV beryllium. Sheets of 1 - 1.5 mm thickness were fabricated by forging and hot rolling, within cans, of cast billets or billets compacted from powder of either -50, +110 mesh or -200 mesh. The sheets were then warm rolled (700°C) to reductions of approximately 50, 100, 180, and 250%. The feasibility of warm rolling beryllium varies in inverse ratio to the total impurity level and the oxide content.

The temperatures of recrystallisation, TR, as defined by X-ray diffraction, decreased with the amount of reduction and varied from 700 - 730°C on the average for SR cast metal, to 760 - 765°C for Brush cast metal, to 800 - 810°C for the powder metallurgy materials. Examinations by optical and electron microscopy were carried out on all the materials after different stages of processing and on the worked sheets after heat treatments in the recovery (one hour at TR -100), recrystallisation (one hour and ten hours at TR), and at grain growth (one hour and 100 hours at TR +200) temperatures. In addition, observations with optical and electron microscopes at temperature made it possible to obtain more precise information on the process of recrystallisation.

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In the as-cast condition, Brush beryllium can be distinguished from SR by a finer grain size and numerous inclusions. Forging and hot rolling leads to a strong deformation of the grains. In this condition, the SR metal is recrystallised while Brush metal remains worked. Warm rolling decreases this difference. The worked state is characterised by some grains which are slightly deformed, surrounded by a mass of highly distorted material, particularly in the case of Brush metal. The slightly deformed grains consist of slightly disoriented sub-grains containing a small number of dislocations. Between these grains is a confused region in which deformed blocks can be distinguished in the middle of dense veins of dislocations.

The recrystallisation of such a structure results from the competition of two processes: on the one hand, the reorganisations in situ of the lightly deformed grains by the elimination of dislocations and the coalescence of sub-grains; on the other hand, the appearance of nuclei at the expense of these large grains. This latter process appears preponderantly in the Brush metal. At equal levels of deformation, grain size is finer for Brush metal than for SR. Recovery leads to a more or less complete rearrangement of the substructure. At high temperature, grain growth occurs, the rate of growth being greater the purer the metal.

In the metal of powder origin, the continuous oxide film which surrounds each grain of powder is entirely destroyed by forging and hot rolling, and is broken into particles of 1/100 to several tenths of a micron dispersed in layers parallel to the surface of the sheet. These particles are smaller and more numerous for the -200 mesh powders. These oxide layers no longer constitute insurmountable obstacles for the grain boundaries as shown by the fact that the powdered and rolled Brush metal exhibits an abnormal grain size comparable to that of cast metal. The SR powder materials, by contract, preserve a fine grain size throughout the fabrication operations.

These structural differences are found again after warm rolling. During recrystallisation, powdered Brush metal changes in the same manner as cast metal. During recrystallisation of the powdered SR materials, nuclei appear which grow rapidly to approximately a 20-micron size. In both cases, prolonged heating at high temperature causes only a slight grain growth.

Mechanical properties during tension and bending have been measured on the entire collection of fabricated products and annealed sheets. In the forged state, the powder metals have better mechanical properties than the cast metal, which is fragile. Hot rolling improves the properties of cast metal but lowers slightly those of powdered SR and causes powdered Brush metal to become very fragile because of abnormal grain growth. After working, the best combinations of mechanical properties are obtained in the recrystallised conditions. Recovery treatments have given good results only for Brush cast metal. With regard to grain growth treatments, these seem to improve the bendability of cast metal, but in all cases are catastrophic for the tensile properties. For the SR metal, the mechanical properties improve as the grain size becomes smaller. Such a correlation has not been found for Brush metal of this type.

The following points come out of this study:

(a) Increase of the impurity content in beryllium raises the recrystallisation temperature and decreases the tendency for grain growth by restraining the movement of grain boundaries.

(b) At high levels of deformation, the distribution of oxide does not seem to be fundamentally different for the material originating from the different types of powder.

(c) The layers of oxide effectively restrain the motion of grain boundaries to the point of making grain growth negligible but do not always constitute an insurmountable barrier for these boundaries.

(d) In all cases, the conditions of fabrication play a fundamental role in the subsequent behavior of the metal, especially with respect to the mechanical properties.

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