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A DENSITY STUDY OF BERYLLIUM

by

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U. S. ATOMIC ENERGY COMMISSION
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Y. M. Johnson - Computing Development

ABSTRACT

Densities were determined for beryllium in the vacuum-cast and wrought vacuum-cast conditions produced as part of the beryllium development program. The average densities of the Rocky Flats as-cast and wrought beryllium were approximately the same as that of commercial powder-pressed beryllium but varied over a smaller range. The density was more uniform in Rocky Flats as-cast ingots and wrought sheets than in commercial powder-pressed material.

INTRODUCTION

...e density of the commercial powder-pressed beryllium currently in use at Rocky Flats must be in the ranges of $1.84\text{--}1.86\text{ g/cm}^3$ or $1.84\text{--}1.87\text{ g/cm}^3$.⁽¹⁾⁽²⁾⁽³⁾ A development program is in progress at Rocky Flats to reclaim beryllium scrap by casting, rolling, and forming techniques. Density data on the Rocky Flats beryllium products was needed for writing specifications and for design data.

The most accurate density value for beryllium in the literature is 1.8477 g/cm^3 at 25°C as calculated from x-ray data.⁽⁴⁾ Voids have occasionally been a problem with powder-pressed beryllium currently in use at Rocky Flats. Voids could also be a problem in as-cast beryllium. The changes in volume during solidification and the phase change from the body-centered cubic to the hexagonal close-packed crystal structure just below the solidification temperature total 7.0 percent.⁽⁵⁾ Fewer voids, or at least voids of a smaller magnitude, would be expected in the wrought material due to the deformation during rolling.

All of the major metallic impurities would tend to increase the measured density of beryllium. Even beryllium oxide, which is the major impurity, would increase the measured density since the density of the oxide is about 3.0 g/cm^3 and the density of beryllium metal is about 1.8 g/cm^3 .

True density, instead of apparent density which is normally used for quality control, was determined during this investigation. True densities are more desirable since the bouyancy effects of air on the samples and weights are compensated for and the data can be compared more accurately with the work of other investigators with different environmental conditions.

(1) Beryllium Material Specifications, Type NW(3%) P11718. As revised 2/2/65.

(2) Specifications for Type WLA Beryllium Shapes, P11736. As revised 3/30/65.

(3) Specifications for Type III Beryllium Shapes, P11512. As revised 1/19/65.

(4) Lillie, D. W., "The Physical and Mechanical Properties of Beryllium", *The Metal Beryllium*, D. W. White, Jr. and J. E. Burke, ed., American Society for Metals, Cleveland, 1955, p 306.

(5) Grosse, A. V. and Cahill, J. A., "The Density of Liquid Beryllium from its Melting Point (1556K) to 2200K and its Expansion on Melting", *Trans. ASM*, 57, 739-42 (1964).

PROCEDURE

The densities were determined by a hydrostatic weighing method. The solid samples were weighed while suspended in air and in a liquid of known density. The mechanics of the calculations are shown on the Procedure for the Evaluation of the True Density of Beryllium in the Appendix. The densities were all adjusted to 20.0°C for comparison purposes by using the thermal coefficient of volume expansion.

The balance could be read to five decimal places but only four places were reproducible and accurate and therefore used. The weighings were accurate to $\pm 0.2\text{ mg}$. The liquid media vapor in the balance case caused the zero point to shift slightly from the normal zero point. It was necessary to reset the zero point whenever the liquid media was put into or removed from the balance case in addition to the normal adjustments during weighing.

Freon-113[®]* trichlorotrifluoroethane was used as the liquid as recommended by Elsheimer⁽⁶⁾. The density versus temperature data for Freon-113[®] was supplied by the Standards Laboratory. The precision of the density data on Freon-113[®] was $\pm 0.2\text{ mg/cm}^3$. Freon-113[®] has a density of 1.57 g/cm^3 which is close to the 1.8 g/cm^3 for beryllium. This increased the accuracy of the data over earlier unreported work using water with its lower density.

The samples were suspended in Freon-113[®] on a polished 24-gage wire. The effect surface tension of Freon-113[®] could have on the wire was eliminated by weighing of the wire suspended in Freon-113[®] both with and without the sample.

Typical analyses for the powder-pressed, vacuum-cast and wrought materials are given in Table I.

(6) Elsheimer, H. N., Suggested Liquid Media for Density Measurements of Beryllium, Plutonium, and Uranium Parts, The Dow Chemical Company, Rocky Flats Division, November 11, 1963, CRDL 94566-3 (internal report).

*Trademark E. I. du Pont de Nemours & Co.

TABLE I
TYPICAL ANALYSES OF BERYLLIUM

	Rocky Flats As-Cast and Wrought (w/o)	Commercial Powder-Pressed (w/o)
Be*	99.4	98.6
BeO*	0.25	1.5
Al*	0.07	0.09
Fe*	0.15	0.15
C*	0.02	0.08
N*	0.01	0.03
Mn	0.01	0.01
Cr	0.02	0.02
Cu	0.05	0.05
Ni	0.02	0.02
Ti	<0.03	0.03
Ca	<0.006	0.01
Cd	<0.003	<0.003
Co	<0.003	<0.003
Mo	<0.003	<0.003
Pb	<0.003	0.003
W	<0.006	<0.006
Zn	0.01	0.01

*Wet Chemical Analyses. The others were by spectrographic techniques.

RESULTS AND DISCUSSION

A statistical analysis of the true density data is shown in Table II. It was not considered necessary to show the actual data from the density determinations. The terminology used in Table II is as follows:

(N) - Number of density determinations

Mean - Simple arithmetical average of (N) density determinations

Standard Deviation - A statistical measure of the deviation of the individual values from the mean. Mathematically it is the square root of the value obtained by dividing the sum of the squares of the differences between the individual values and the mean by (N-1).

Confidence Limits for Individuals - Ninety-five percent of the time an additional data point would be expected to fall between the lower and upper limits.

Confidence Limits for Means - Ninety-five percent of the time the mean for a new set of the same number (N) of data points would be expected to fall between the lower and upper limits.

Items 1 and 2 in Table II show the excellent reproducibility of the data for measurements on one day and on different days. The small difference in the standard deviations for Items 1 and 2 is not significant when calculated from 16 data points. The range for the 16 determinations in Item 1 was 1.8511 to 1.8522 g/cm³. The range for the 16 determinations in Item 2 was 1.8443-1.8455 g/cm³.

The densities of the samples from MR 192 and MR 152, which were used for Items 1 and 2 in Table II, were also determined by the Standards Laboratory to check the accuracy of the data. The Standards Laboratory densities for MR 192 and MR 152 were 1.8525 g/cm³ and 1.8462 g/cm³, respectively, which were slightly higher than our values of 1.8517 g/cm³ and 1.8449 g/cm³. Freon-113[®] used by the Standards Laboratory was from a different shipment than that used for this investigation. We also used the Freon-113[®] used by the Standards Laboratory for single determinations on MR 192 and MR 152 and values of 1.8529 g/cm³ and 1.8456 g/cm³ respectively were obtained. These densities obtained with the same Freon-113[®] agree satisfactorily with the determinations by the Standards Laboratory and substantiate the accuracy of the procedure. It also points out that a variation in density values can result from using different shipments of Freon-113[®].

Items 3, 4, 5, and 6 in Table II show the uniformity of the density in two sheets of Rocky Flats wrought sheet, a Rocky Flats vacuum-cast ingot, and a slab of commercial powder-pressed log of beryllium. By coincidence, the density data for MR 75 had an even lower standard deviation than the 16 determinations on one sample in one day as shown in Item 1. The uniformity of density was considerably better in the Rocky Flats wrought sheets and the Rocky Flats vacuum-cast ingot than in the commercial powder-pressed material. As expected, the density was not as uniform in the vacuum-cast ingot as in the wrought sheets. Beryllium shrinks a total of 7 percent during solidification and the phase change about 15°C below the solidification temperature.⁽⁵⁾ The deformation during rolling would tend to seal the voids in the cast material and thereby make the density more uniform.

Although the statistical sample (N) of Rocky Flats wrought product in Item 7 was greater than for the commercial powder-pressed product in Item 8 in Table II, the data shows that the Rocky Flats wrought product has considerably less variation in density. The

TABLE II
STATISTICAL ANALYSIS OF BERYLLIUM TRUE DENSITY DATA*

Identification	(N)	Mean (g/cm ³)	Standard Deviation	95% Confidence Limits for Individuals		95% Confidence Limits for Means	
				Lower	Upper	Lower	Upper
1. Reproducibility: 1 sample from MR 192 -- 16 determinations on 1 day.	16	1.8517	0.00026	--	--	--	--
2. Reproducibility: 1 sample from MR 152 -- 1 determination a day for 16 days.	16	1.8449	0.00030	--	--	--	--
3. Uniformity in Rocky Flats wrought sheet MR 75: 14 samples from random locations.	14	1.8474	0.00017	1.8471	1.8478	1.8473	1.8475
4. Uniformity in Rocky Flats wrought sheet MR 160: 9 samples from random locations.	9	1.8450	0.00124	1.8421	1.8478	1.8440	1.8459
5. Uniformity in Rocky Flats as-cast ingot: 10 samples from random locations.	10	1.8480	0.00064	1.8465	1.8494	1.8475	1.8484
6. Uniformity in a slab from a commercial powder-pressed log: 12 samples from random locations.	12	1.8473	0.00699	1.8319	1.8627	1.8429	1.8517
7. Typical Rocky Flats wrought product: samples from 48 sheets.	48	1.8470	0.00207	1.8429	1.8512	1.8464	1.8476
8. Typical commercial powder-pressed product: samples from 10 production parts.	10	1.8473	0.00608	1.8336	1.8611	1.8430	1.8517

*g/cm³ at 20.0 °C

means for the Rocky Flats wrought product and the commercial powder-pressed product are the same. The powder-pressed product has a higher BeO content which would increase the density but it had two samples with a density of 1.83 g/cm³ which shows that it also had a greater amount of voids.

It is common to use apparent densities for quality control work. The apparent densities for all 88 samples of Rocky Flats wrought sheet and vacuum-cast ingots were in the range of 1.84-1.85 g/cm³. The densities for 20 of the 22 samples of commercial powder-pressed material were in the range of 1.84-1.86 g/cm³ and the other two samples had a density of 1.83 g/cm³.

CONCLUSIONS

Rocky Flats wrought sheets of beryllium are more uniform in density than the Rocky Flats vacuum-cast ingots. Both the Rocky Flats wrought sheets and vacuum-cast ingots are more uniform in density than commercial powder-pressed material.

The average density for Rocky Flats wrought sheets and commercial powder-pressed material is approximately the same but the range of densities is considerably greater for the powder-pressed material.

The apparent density for Rocky Flats beryllium in wrought sheet and vacuum-cast ingot is 1.84-1.85 g/cm³. The apparent density for commercial powder-pressed beryllium is 1.84-1.86 g/cm³.

APPENDIX

PROCEDURE FOR THE EVALUATION OF THE TRUE
DENSITY OF BERYLLIUM

A. Weight in air	_____
B. Gross weight in liquid	_____
C. Counter balance weight	_____
D. Weight in liquid (B - C)	_____
E. Weight in air correction
factor (A x 0.000399)	_____
F. Corrected weight in air (A + E)	_____
G. Volume of weights ($\frac{D}{8.4}$)	_____
H. Buoyancy correction factor
(G x 0.00095)	_____
I. Corrected weight in liquid
(D - H)	_____
J. Weight displaced by liquid
(F - I)	_____
K. Temperature of liquid	_____
L. Density of liquid at
temperature (K)	_____
M. True density of blank ($\frac{F \times L}{J}$)	_____
N. True density corrected to
20.0°C	_____

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