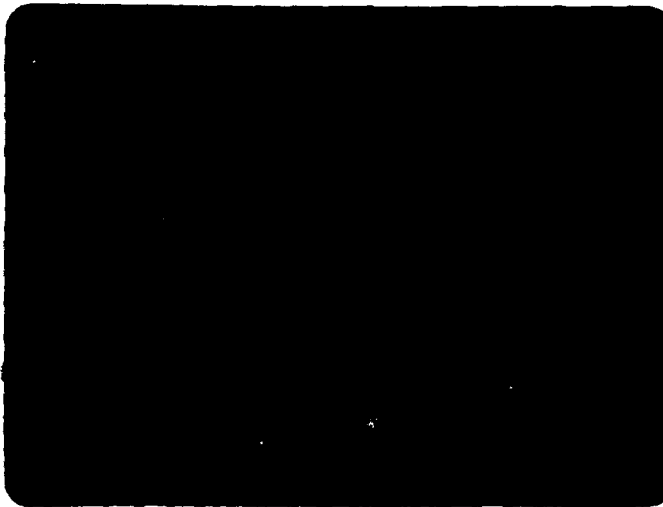


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AMMRC TR 68-08

**SOME EFFECTS OF POWDER PARTICLE SIZE ON  
THE PHYSICAL BEHAVIOR OF PRESS-FORGED  
BERYLLIUM**

Technical Report by

*JACOB GREENSPAN*

May 1968

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D/A Project 1C024401A328  
AMCMS Code 5025.11.294  
Metals Research for Army Materiel  
Subtask 38166

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ARMY MATERIALS AND MECHANICS RESEARCH CENTER

SOME EFFECTS OF POWDER PARTICLE SIZE ON THE PHYSICAL BEHAVIOR  
OF PRESS-FORGED BERYLLIUM

ABSTRACT

Powder metallurgy beryllium generally contains an oxide dispersoid, due to particle surface scale, and thus the material actually is a system subject to particle strengthening. The present report shows a dependence of strength on raw powder particle size and also on thermal history.

## INTRODUCTION

Powder metallurgy beryllium differs from ingot beryllium particularly in oxide content and, as a result, microstructures of the massive materials differ significantly.<sup>1</sup> In general, the oxide appears as a dispersoid in network-like configuration, distributed throughout. Principally, it has been known to act as a barrier to grain growth, characterizing powder metallurgy material by grain size considerably finer than that of ingot material. An important result has been grain refinement strengthening and, in addition, dispersed particles have shown some influence on fundamental deformation behavior of the matrix material. Thus, the dispersoid, which actually occurs inadvertently as a result of powder surface oxidation, is known to be particularly relevant to mechanical behavior and is recognized as an important materials parameter. However, more exact characterization is necessary for more effective utilization and greater understanding of the particle-matrix relationship will be helpful to further guidance. The present report gives some experimental data that contribute in part to the subject.

## PROCEDURE

The material examined in this investigation was an industrial-grade electrolytic powder, having the impurity analysis shown in Table I. Powders

Table I. IMPURITY ANALYSIS OF  
ELECTROLYTIC BERYLLIUM POWDER

Impurity	ppm	Impurity	ppm	Impurity	ppm	Impurity	ppm
Iron	300	Sodium	<100	Silicon	25	Copper	7
Carbon	260	Zinc	< 80	Titanium	<25	Silver	<3
Chlorine	200	Calcium	30	Lead	<15	Cadmium	<2
Nickel	140	Chromium	25	Molybdenum	<15	Boron	<1
Aluminum	110	Magnesium	25	Manganese	11		
BeO 1.91% for -43 micron powder; 2.2% for -20 micron powder							

were attritioned by conventional procedure, and then were sized nominally to fractions of -43 micron and -20 micron particles. The object in this case was to alter oxide distribution without large change in oxide content. The actual increase was from 1.91 to 2.2 percent (Table I) by removing the 43 to 20 micron fraction of metal powder in this way. The powders were hot pressed to block, and then cubes of 2-inch dimension were press forged to 1/2-inch plate as shown in Figure 1. Forging was performed at specific temperatures as given in Table II, and then additional heat treatment, associated with solution and precipitation of certain metallic impurities, was applied.<sup>2-7</sup> Possible changes of this kind were followed by precision electrical resistivity measurement along the gage length of tensile coupons of 1-inch length (about 2.5 cm) and about 0.030-square inch cross section, (about 19 sq mm) later tested in tension. Measurement was at room temperature only, the object being to note relative changes that might associate metallic impurity with mechanical behavior. The resistivity measurement was accomplished with a Kelvin bridge capable of  $10^{-6}$  ohm resolution, but it was found that general experimental deviation of the order of 1 percent of the mean could occur.

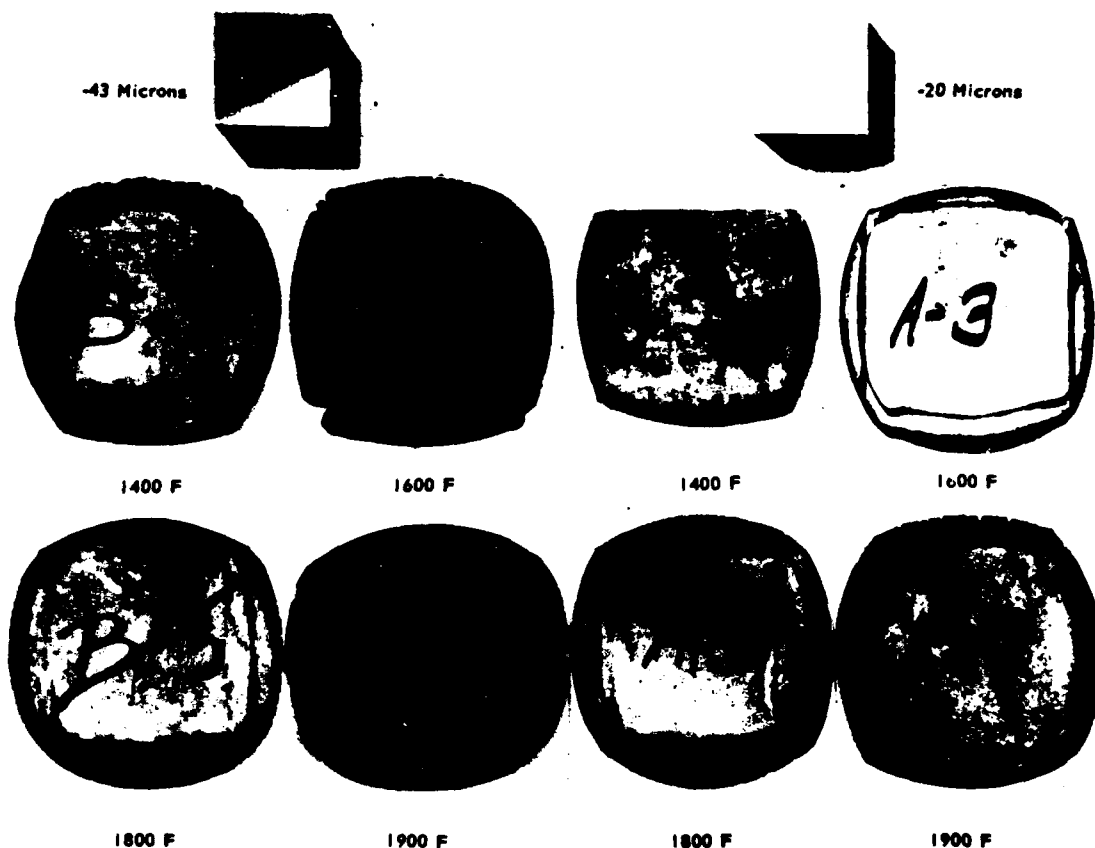


Figure 1. PRESS-FORGED BERYLLIUM, SHOWING STARTING BLOCK AND PLATE FORGED AT VARIOUS TEMPERATURES

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The possible influence of the above-described variations on uniaxial tensile properties, impact behavior, and hardness then was examined. Tensile samples of the size indicated were tested with an extensometer attached, duration of the test being of the order of minutes. Impact test bars, in smooth simple-beam configuration, were struck by a swinging pendulum and the total energy for fracture was recorded. Samples were of 0.394-inch square cross section (about 1 cm) and 1-5/8-inch span (about 4 cm), and the estimated rise time to fracture was of the order of milliseconds. Finally, hardness indentations of Rockwell B nomenclature were taken on both tensile and impact test samples.

## RESULTS

Impact strength was found to be influenced by powder particle size as indicated in Figure 2. The -20 micron material exhibited considerably greater resistance to impact than the -43 micron material, but this behavior was confined to the lower end of the processing temperature scale employed. Heating

Table II. PHYSICAL PROPERTIES OF FORGED ELECTROLYTIC BERYLLIUM POWDER BLOCK

Forging Temperature		Additional Thermal Treatment	Impact Energy* (ft-lb)		Strength Properties†						Rockwell B Hardness‡		Resistivity** (microhm cm)	
					-20 μ			-43 μ						
					deg F	deg C	-20 μ	-43 μ	Y.S. (ksi)	T.S. (ksi)	Elong. (%)	Y.S. (ksi)	T.S. (ksi)	Elong. (%)
1400	762	As forged	30.8	22.5	54.1	85.8	18	34.9	65.2	6.0	91.2	90	4.28	4.48
		Aged††	4.2	5.4	42.1	64.2	2	32.9	64.2	5.0	88.0	87	4.27	4.25
1600	871	As forged	31.0	14.5	41.6	71.9	7	44.9	70.6	8.0	89.2	88.5	4.52	4.08
		Solutionized††	4.0	8.6	35.5	66.9	5	39.8	57.1	1.0	87.5	86.5	4.58	3.91
1800	982	As forged	16.8	9.5	35.1	69.4	5	36.2	70.8	12.0	87.0	86.0	4.42	4.09
		Solutionized	7.0	7.3	33.8	62.4	4	36.2	64.5	3.0	86.2	86.5	4.46	3.90
1900	1038	As forged	13.0	12.1	36.7	70.6	10	31.4	68.2	3.7	85.5	85.5	4.61	4.06
		Solutionized	9.5	8.9	34.8	73.0	11	28.5	60.7	4.6	86.0	85.0	4.32	4.18
												4.46	4.07	
												4.48	4.25	

Heat treatment: Homogenized, 1900 F (1038 C), in vacuum, 6 hours, cooled in flowing argon, before forging.

\*Smooth test bars. Average of at least two observations.

†Average of two observations.

‡Average of at least four observations.

\*\*Average of at least six observations.

††1400 F (762 C) in vacuum, 8 hours, furnace cooled.

†††1900 F (1038 C) in vacuum, 6 hours, cooled in flowing argon.

at higher temperatures, whether for forging or other thermal treatment, is seen generally to have nullified the effect. Tensile strength, given in Figure 3 shows trends of the same kind, but to far lesser extent on the respective ordinate scales employed. This behavior is reflected also for hardness, given in Figure 4. It is seen that the -20 micron material was always the harder by a small but measurable increment, and that the general loss in strength associated with thermal history was accompanied by loss in hardness.

In general, the higher values of impact strength were coincident with a greater degree of plastic deformation on impact. The limits experienced are illustrated in Figure 5 with samples of about 30 and 3 foot-pound impact strength level. While the difference in plastic deformation appears small in the figure, the numerical difference in absorbed impact energy

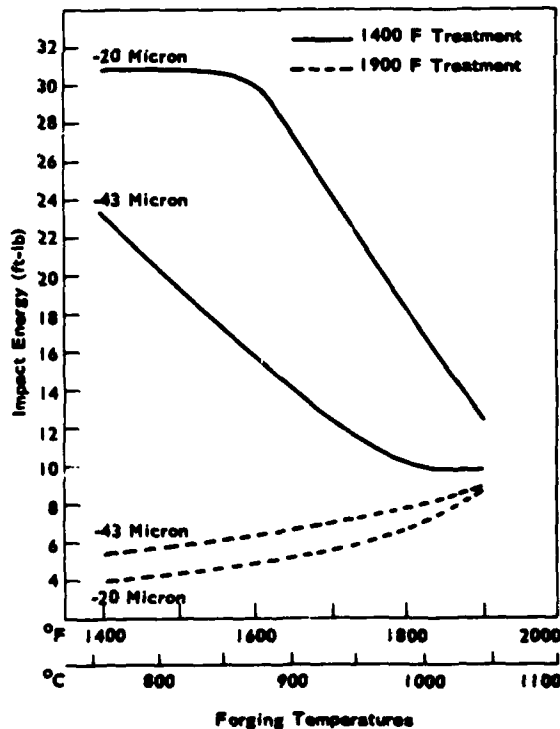


Figure 2. IMPACT RESISTANCE OF FORGED BERYLLIUM POWDER BLOCK  
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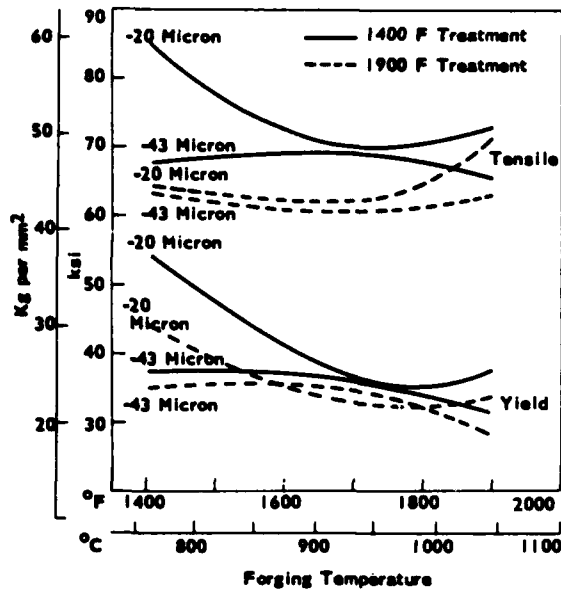


Figure 3. YIELD AND TENSILE STRENGTHS OF FORGED BERYLLIUM POWDER BLOCK

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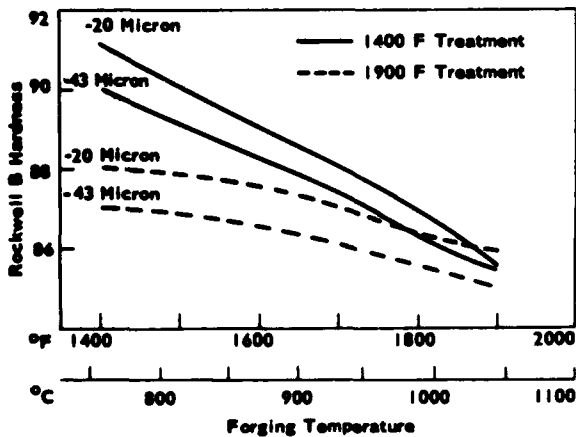


Figure 4. HARDNESS OF FORGED BERYLLIUM POWDER BLOCK

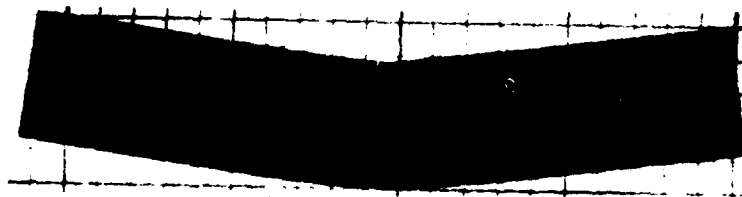
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is seen to have been relatively great. Thus, it seems reasonable that further marginal increases in ductility could be of magnified significance to impact behavior of this kind. Fracture markings in all samples were indicative generally of brittle fracture as shown by a typical macrofractograph in Figure 6. Yet, some change toward a more "ductile" appearance, as well as the appearance of some crack arrest, is observed on the compression side of the test bar. For beryllium, it is known that suppression of fracture can result in increased slip activity, and the preceding probably is such an example.

Photomicrographs showing dispersoid (in bright light) and grain structure (in polarized light) are presented in Figure 7. This metallography shows little distinction between the two powder materials with respect to general dispersoid configuration, but it is known that very small oxide particles must be more profuse in the -20 micron material. Also, grain sizes appear nearly equivalent, and it is significant that the relatively large differences in impact strength were not accounted for by this factor.

Precision electrical resistivity data given in Table II show that the metallic impurity present (Table I), did not undergo solutionizing and aging reactions that resulted in measurable changes in the electrical resistivity. This is concluded from the general lack of distinction between aged and solutionized conditions, as well as between the processing temperatures shown.

Considerable variability between some groups is seen, but without specific relation to mechanical behavior. This might be an indication that the distribution of impurity in the parent block material was not uniform, although the extent involved is insensitive to the present mechanical testing.



30 ft-lb Impact Energy



3 ft-lb Impact Energy

Figure 5. DUCTILITY IN BERYLLIUM IMPACT TEST BARS  
19-066-321/AMC-68



-Tension

-Neutral

-Compression

Figure 6. TYPICAL MACROFRACTOGRAPH.  
BERYLLIUM IMPACT TEST BAR  
19-066-320/AMC-68

With metallic impurity seen to have been of negligible consequence, within the present scope, mechanical behavior must have been influenced principally by the oxide dispersoid. Results are as though they were influenced by a condition of continuity or adhesion between particle and matrix that was subject to disruption by the temperature experienced, the effect increasing with temperature. Loss of this adhesion should not affect resistivity measurably, but should affect strength, which is exactly in accordance with the data that have evolved. Thus, the possibility of some form of continuity between the beryllia particle and the beryllium matrix, though apparently unusual, is indicated.





30 ft-lb Impact Energy



Bright Light

3 ft-lb Impact Energy

Polarized Light

Figure 7. MICROSTRUCTURES OF BERYLLIUM IMPACT TEST BARS.  
Left shows dispersoid, right shows grain structure. Mag. 500X.

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## SUMMARY AND REMARKS

The oxide dispersoid in powder metallurgy beryllium, which is known to be an important constituent with respect to mechanical behavior, was related further with strength, hardness, and process history. The dispersoid was seen to have been influential beyond its effect in grain refinement strengthening. Further understanding should lead to more effective use of the parameters involved. The possibility of some form of adhesion between particle and matrix has been indicated, though this concept of a nondeformable particle, particularly of the beryllia-beryllium system, appears unusual. Unquestionably, the particle must play a fundamental role in the fracture process, in crack origin as well as crack propagation. Particles are not in uniform dispersion, but are concentrated in network-like zones, predominantly in the vicinity of grain boundaries, as indicated in Figure 7. These zones are in greater resemblance to the aggregate structure of this class of materials than the disperse structure, which in itself is seen to be a strengthening factor.<sup>8,9</sup> If crack origin is at grain boundaries, as has been indicated in some bicrystal studies,<sup>10</sup> then the role of the dispersoid is emphasized. However, electron micrography in other work has shown very small oxide particles, in considerable numbers, within grains also.<sup>11</sup> Apart from these observations that indicate the need for understanding of particle-matrix relationships, additional empirical data, based on methods generally employed for this class of dispersion-strengthened system, may lead to significant development of powder metallurgy beryllium.

## ACKNOWLEDGMENT

The author is grateful to Dr. K. J. Tauer and Mr. L. R. Aronin of AMRC for helpful discussions regarding electrical resistivity measurement and interpretation of data.

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3. REPORT TITLE SOME EFFECTS OF POWDER PARTICLE SIZE ON THE PHYSICAL BEHAVIOR OF PRESS-FORGED BERYLLIUM		
4. DESCRIPTIVE NOTES (Type of report and inclusive dates)		
5. AUTHOR(S) (Last name, first name, initial) Greenspan, Jacob		
6. REPORT DATE May 1968	7a. TOTAL NO. OF PAGES 8	7b. NO. OF REFS 11
8a. CONTRACT OR GRANT NO.		9a. ORIGINATOR'S REPORT NUMBER(S) AMMRC TR 68-08
a. PROJECT NO. IC024401A328		9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report)
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