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DENSIFICATION OF AN OXIDE POWDER MIXTURE

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GENERAL DYNAMICS | FORT WORTH

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Materials Science

DENSIFICATION OF AN OXIDE POWDER MIXTURE

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RESEARCH & ENGINEERING DEPARTMENTS

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ABSTRACT

An oxide powder mixture containing iron, lithium, nickel and zinc was studied as to densification techniques by dynamic and static pressure means. Densification by impact has been unsuccessful in a 150 ft. per sec. - 500 gram impact device but a good increase in density has been obtained by compacting and pressing to 100 kilobars pressure in the UniRam tetrahedral device.

The purpose of this work is to study the effect of dynamic and static pressures upon materials. The intent is to form irreversible phase transformations and metastable equilibrium for improved physical properties in materials.

One material studied in this program was an oxide powder mixture composed of 64.8% iron, 1.8% lithium, 0.15% nickel, and 0.15% zinc and with a particle size of minus 200 mesh. In the powder form, this material is a dull reddish brown or hematite color, whereas, the densified phase has a jet black appearance.

For the dynamic compaction studies, a modified Bendix high velocity impact device was used. This device consisted of an impact piston propelled by a blank .410 gauge shotgun shell. The impact piston carried a cylindrical rod shaped impact head which impacted into a die cavity in which the sample under study was inserted. The total weight of the impact head was 500 grams and velocities of impact were in the range of 120 to 150 feet per second.*

Samples were prepacked into the cylindrical die and, prior to impact, measured about 3/8 inch in diameter and varied from 1/8 to 5/8 inches in length. A variety of auxiliary die inserts were used initially to control the impact such as silver chloride, teflon and paper. An interesting sidelight in regard to using silver chloride was the plating of the die and impact rod with

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* Reference: ERR "Explosive Driven Anvil for Operation in the 10-15 Kilobar Regime." - J. A. Regalbuto

metallic silver as a result of the silver chloride breaking down under impact.

Very little evidence of any densification effect was obtained in these degraded shots although some oxide powder cylinders showed a trace of blackening on the outside. Most cylinders pulverized to powder upon ejection from the die.

Some impact shots were made without auxiliary die inserts in an attempt to obtain full impact effects upon the sample. Most of these shots resulted in fractured metal parts or upset impact rods and some experimenting was done to find a suitable steel to use for die and impact rod material. Table I shows the types of steel tried in these tests. LaBelle HT type steel has shown the best response so far although there are some other types being recommended for trial.

None of these full impact runs produced black oxide cylinders. Most of these cylinders did show a black skin effect and some showed a slight internal darkening with fractures becoming more dish-like or conchoidal.

An additional attempt to densify the powder was made using the UniRam tetrahedral ultra high pressure device; see Figure 1. Although the UniRam is not basically designed for use with fine powders, it is possible by careful gasketing to pack enough powder in the sample cavity in several preliminary pressings at low pressure. The final results of maximum pressure runs on these preliminary pressings indicated that the final density was directly related to the amount of powder capable of being packed into the cavity. This was also directly related to the amount of fracturing of the sample during the final maximum pressure pressing. Such sample fracturing proved highly disastrous to the carbide anvils of the UniRam and the tests were abandoned. Table II gives the results of the UniRam densification runs of about 100 kilobars pressure.

The results indicate that this oxide powder can be densified by high pressure application; however, the final tetrahedrons are nearly always cracked during the final pressing stages. Figure 2 is a picture of the No. 9 tetrahedron; the color on the surface and in cross section is a jet black and the outside surfaces are quite reflective of light. Figures 3 through 7 show tetrahedrons 3, 11, 1 and 9 in cross section. Tetrahedron No. 11 was the result of sintering the No. 2 tetrahedron to see if this would result in further densification; the increase in density was about 3.5% and final tetrahedron was extremely hard. Attempts to obtain a more direct comparison of the sintering effect upon a maximized UniRam product have not been successful, primarily due to the cracking effects in the powder oxide tetrahedron during final pressing.

Table I

TYPES OF STEELS USED IN IMPACT TESTS

RDS - Diamond 3

Composition	Treatment
0.70 C 0.35 Mn 0.25 Si 1.00 Cr 1.75 Ni	Oil Quench from 1525° F Tempered one hour 300°F R_c 60-61

Hampden - Oval 2

Composition	Treatment
2.10 C 0.25 Mn 0.25 Si 12.50 Cr 0.50 Ni	Oil Quench from 1775° F Tempered one hour 400°F R_c 62-63

LaBelle Ht

Composition	Treatment
0.43 C 1.35 Mn 2.25 Si 1.35 Cr 0.30 V 0.40 Mo	Oil Quench from 1675° F Tempered two hours 575°F R_c 56-58

Table II

DENSITIES OF COMPRESSED OXIDE POWDER TETRAHEDRONS

Sample No.	Volume* cc	Weight grams.	Density gr/cc
1	.3423	1.4860	4.341
2	.3330	1.1811	3.547
3	.3787	1.3340	3.522
4	.3044	1.2663	4.160
5	.3087	1.2871	4.169
6	.3731	1.4481	3.881
7	.3141	1.2383	3.943
8	.5047	1.9172	3.798
9	.3065	1.3645	4.451
10	(.2545)	1.1811	(4.635)
11	(.2443)	1.1729	(4.801)
12	.3146	1.4150	4.498

All tetrahedrons were pressed to 100 kilobars. #10 was sample #2 measured on a different basis, before sintering; #11 is #10 after sintering; values in () are comparable only within each other and not to the other values in the table. Final calculated density of #11 was 3.67 based upon an indicated increase of 3.5% over the #2 value.

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*Volumes were calculated and based upon thickness of extruded webs and corrected for corner losses resulting from web trimming.

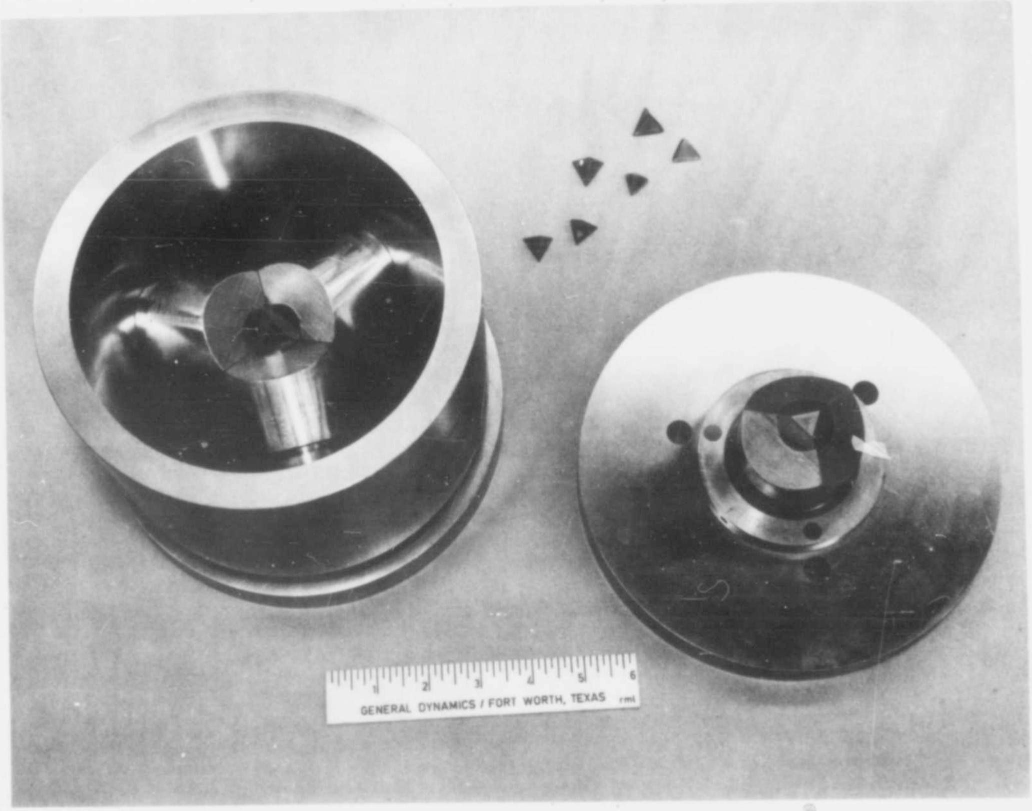

Figure 1. Photograph of the UniRam

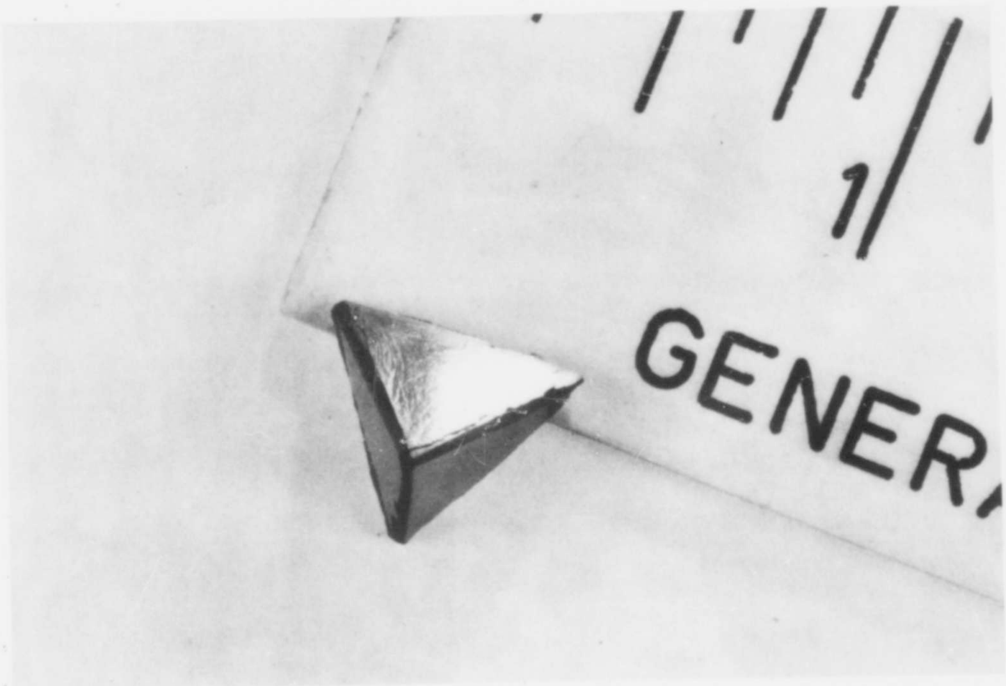

Figure 2. Photograph of the #9 pressed oxide powder tetrahedron.

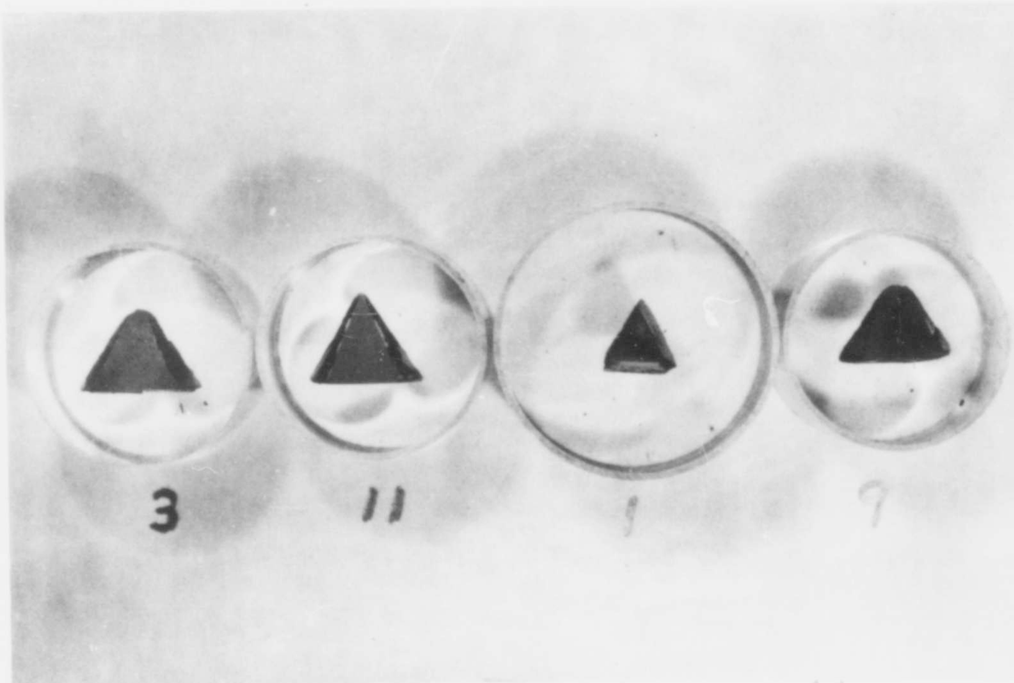

Figure 3. Oxide powder tetrahedrons nos. 3, 11, 1 and 9, sectioned and polished; see following pictures for surface details. Maximum edge length of tetrahedrons is half an inch; original magnification of picture was 1-1/2 x

Figure 4. Photograph of a cross section of the #3 pressed oxide powder tetrahedron. Corners of cross section show some darkening and general texture is somewhat rough; density 3.52. Original magnification 8 x

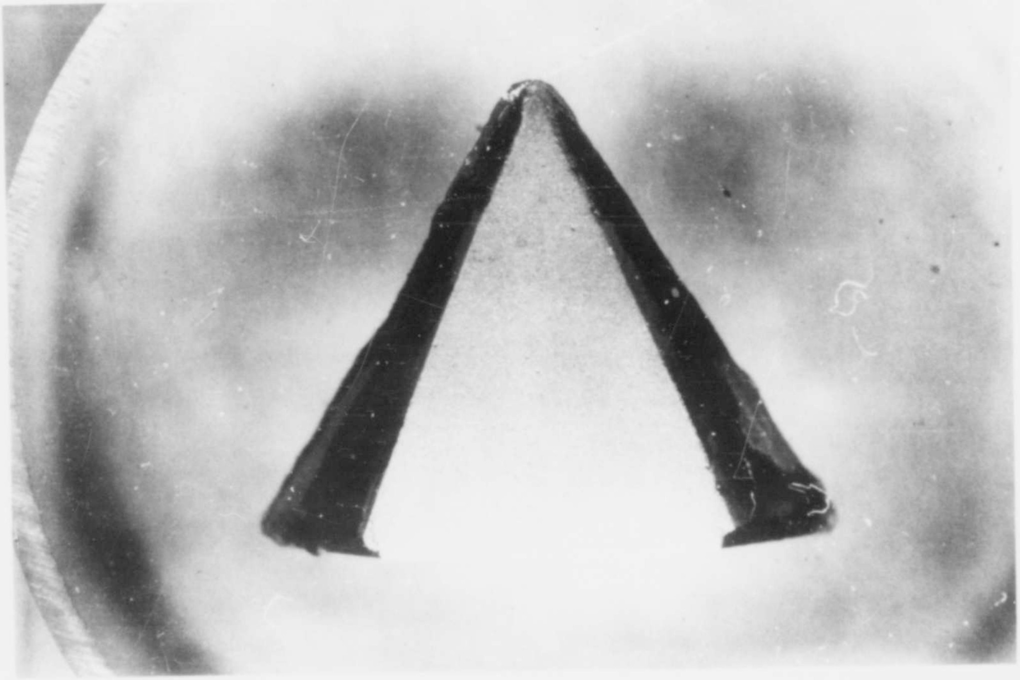

Figure 5. Photograph of a cross section of the #11 pressed and sintered oxide powder tetrahedron. Surfaces are black and material was too hard to cut with file. General texture of cross section was smooth; density before sintering was 3.55, increase in density due to sintering was 3.5% (final calculated density 3.67). Original magnification 8 x

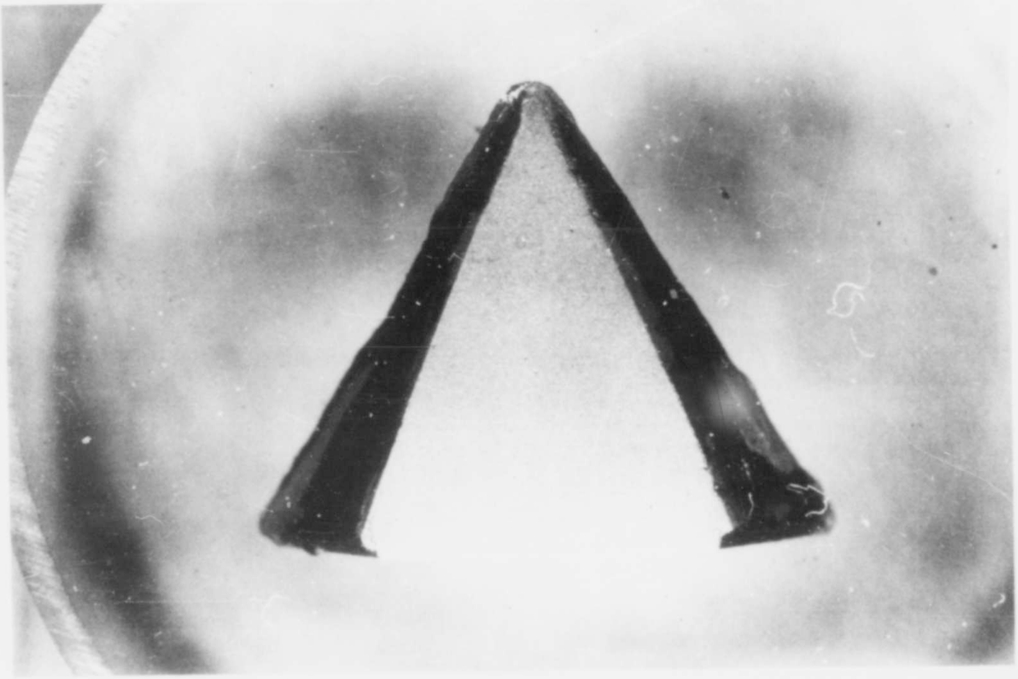

Figure 5. Photograph of a cross section of the #11 pressed and sintered oxide powder tetrahedron. Surfaces are black and material was too hard to cut with file. General texture of cross section was smooth; density before sintering was 3.55, increase in density due to sintering was 3.5% (final calculated density 3.67). Original magnification 8 x

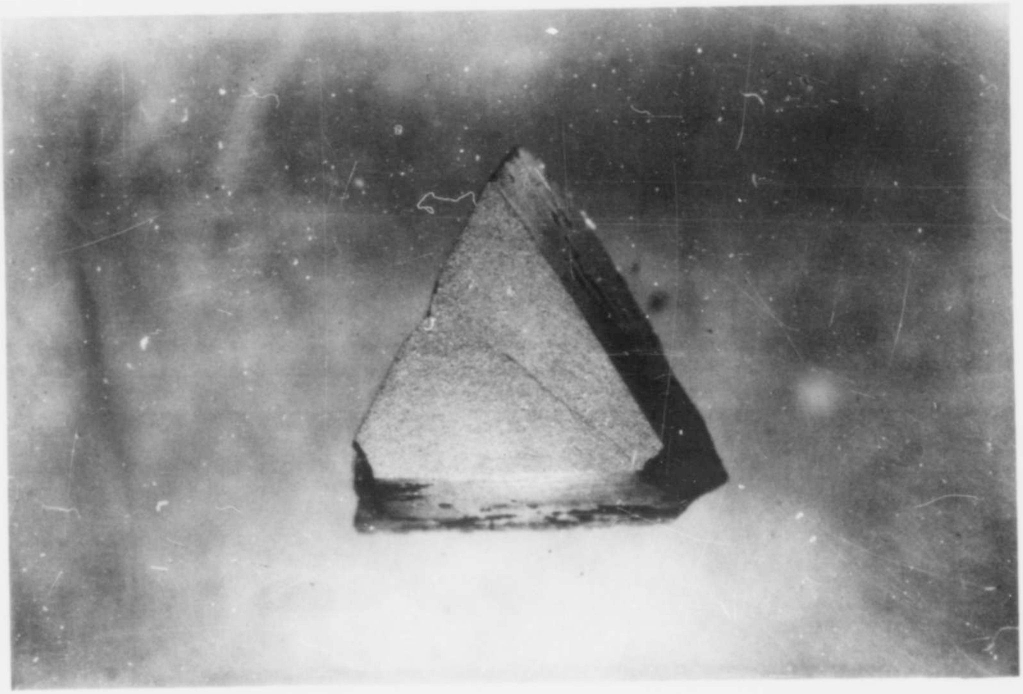

Figure 6. Photograph of a cross section of the #1 pressed oxide powder tetrahedron. General texture was smoother than #3 (Figure 4) but not as smooth or as hard as #11 (Figure 5); density 4.34. Original magnification 8 x

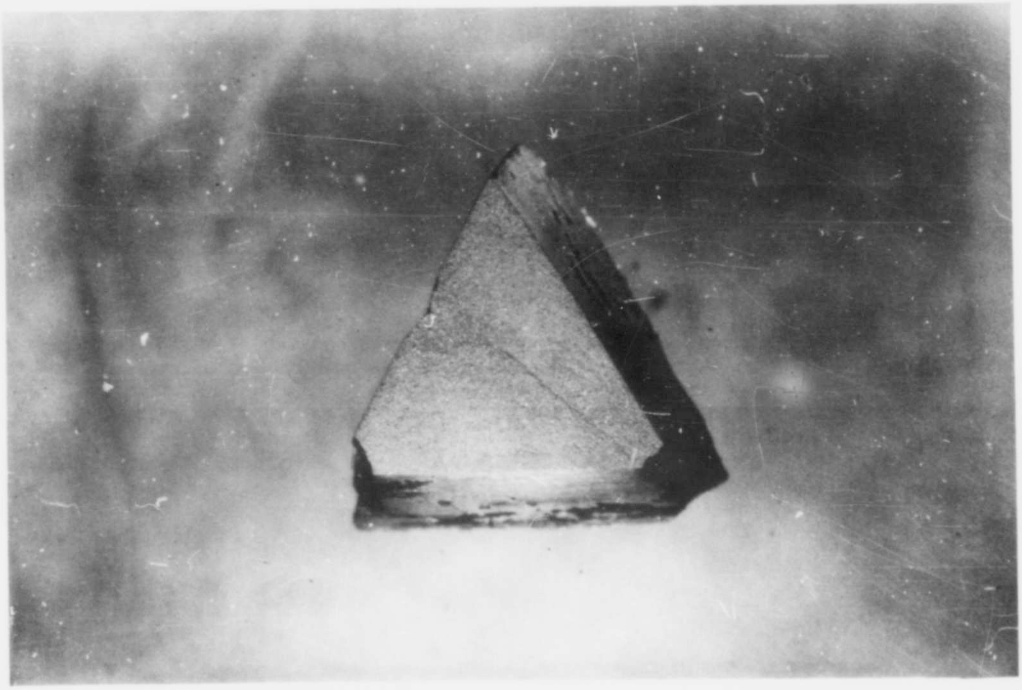

Figure 6. Photograph of a cross section of the #1 pressed oxide powder tetrahedron. General texture was smoother than #3 (Figure 4) but not as smooth or as hard as #11 (Figure 5); density 4.34. Original magnification 8 x

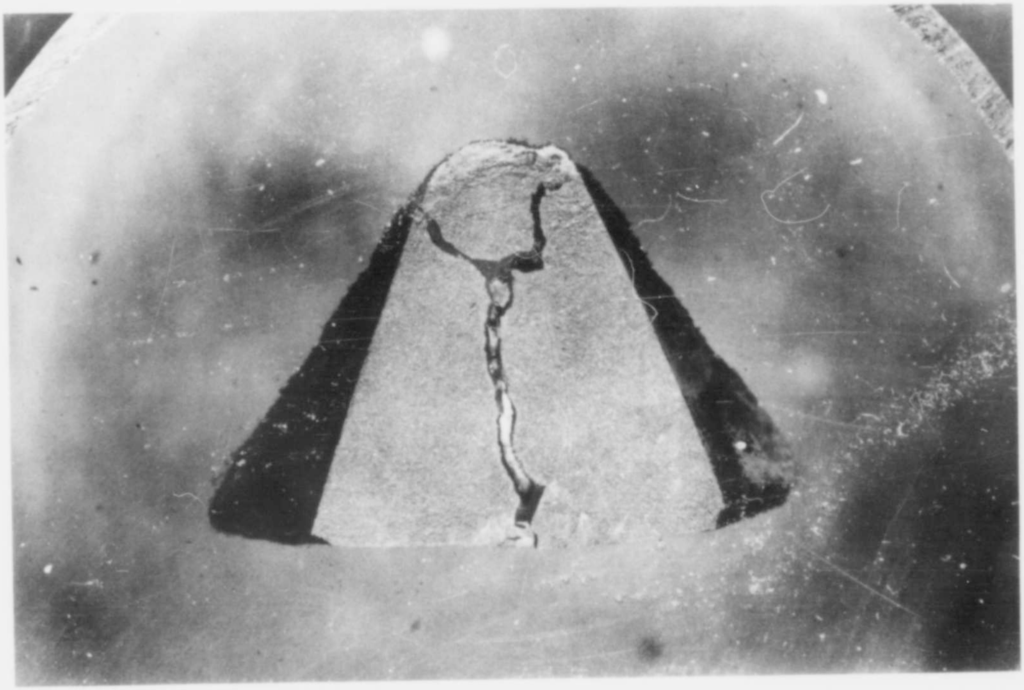

Figure 7. Photograph of a cross section of the #9 pressed oxide powder tetrahedron. General texture is smooth but not as smooth and hard as #11 (Figure 5); density 4.45. Crack has been opened up by mounting process. Original magnification 8 x