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SWAGING OF URANIUM-MOLYBDENUM ALLOY POWDERS
CONTAINING 10 TO 15 WT % MOLYBDENUM

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Metals and Ceramics Division

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S. A. Rabin, A. L. Lotts, and J. P. Hammond

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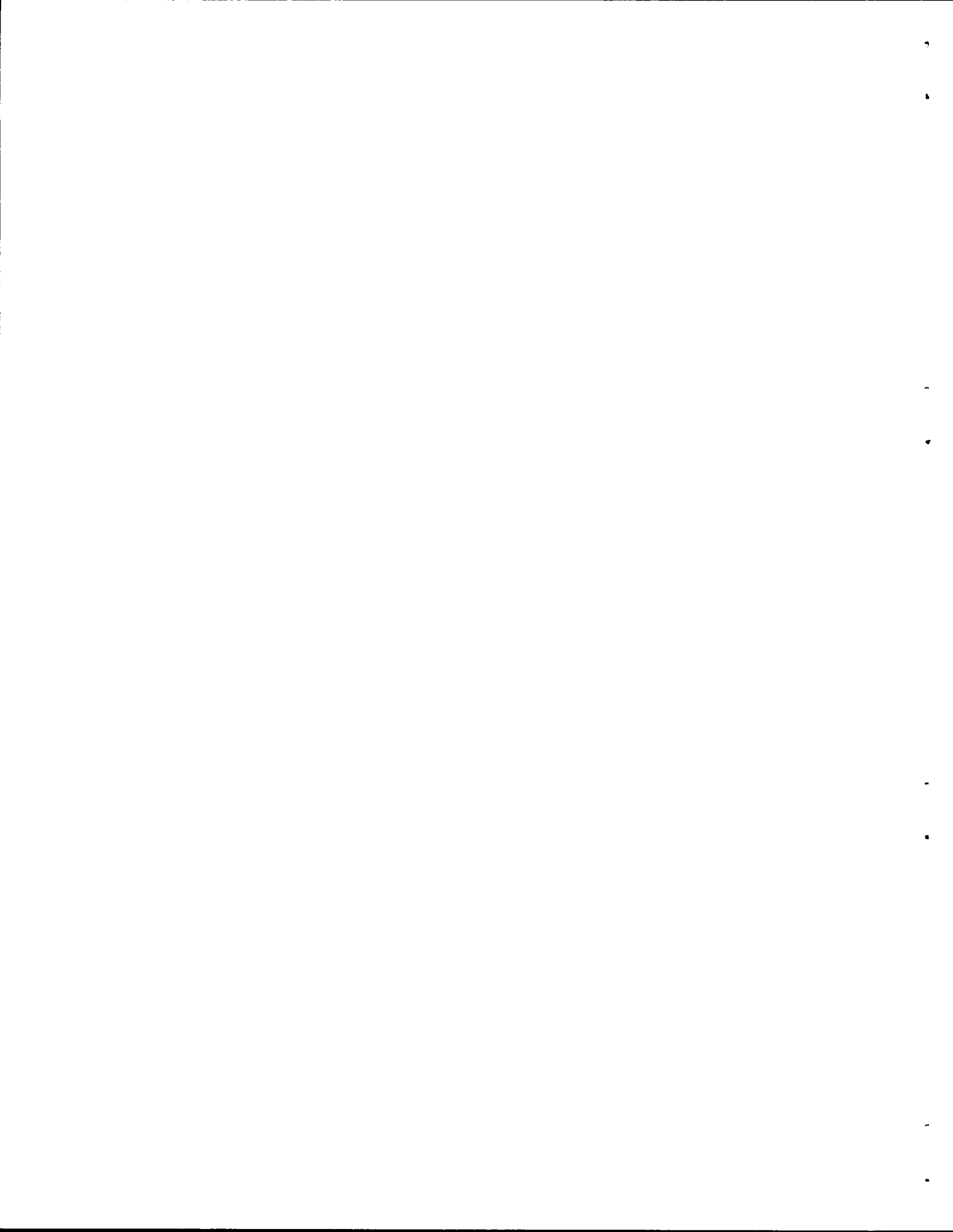


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ABSTRACT

Uranium-molybdenum alloy rods containing from 10 to 15 wt % Mo and 3/16-in. in diameter were successfully fabricated by hot rotary swaging, followed by machining to remove the protective sheathing (Inconel with molybdenum barrier). Structurally strong rods with densities greater than 95% of theoretical were produced from both calcium-reduced uranium mixed with hydrogen-reduced molybdenum and acid-cleaned, prealloyed shot when reduced in area about 55% at 1050 or 1100°C. Alloy homogeneity was good with prealloyed powders; however, traces of molybdenum-rich, gamma phase persisted in the elemental uranium-molybdenum material after swaging at 1100°C. Swagings embodying hydride uranium or oxide-contaminated prealloyed shot were unsatisfactory because of insufficient consolidation or poor interparticle bonding.

INTRODUCTION

One of the advanced fuel element concepts initially considered for the Enrico Fermi Fast Breeder Reactor utilized a dispersion of enriched UO_2 or UC in an isotopically depleted gamma-phase uranium-molybdenum alloy matrix.¹ This fuel system is attractive because it capitalizes upon a high burnup and good thermal performance while concomitantly offering a relative high conversion ratio by virtue of the fertile matrix.

As an adjunct to the fabrication development of this dispersion fuel element, a program was initiated to investigate procedures for compacting

¹S. A. Rabin et al., Fabrication Development of U-Mo- UO_2 and U-Mo-UC Dispersion Fuels for the Enrico Fermi Fast Breeder Reactor, ORNL-3437 (June 10, 1963).

gamma-phase uranium-molybdenum powders to high densities by rotary swaging. The objectives of this study were twofold: (1) to assess this fabrication procedure for powders selected as the matrix for the UO_2 , and (2) to provide samples for associated tests.

Since the program was prematurely terminated, the experimental work could not be completed and the results and conclusions should, therefore, be regarded as preliminary in nature.

MATERIALS

Pertinent physical and chemical properties of the powders used are listed in Table 1. Photomicrographs of the prealloyed uranium-molybdenum shot and elemental uranium powders are shown in Figs. 1 and 2, respectively. Details of the powder preparation processes, which are in some instances unique, have been described elsewhere¹ and are summarized in Appendix A.

Sheathing materials were of a commercial grade. Molybdenum tubing, 5/8-in. OD \times 0.098-in. wall, served as the inner clad. Molybdenum end plugs for closure of the inner sheath were machined from 1/2-in.-diam rod. Cold drawn and annealed seamless Inconel tubing, 1-in. OD \times 0.035-in. wall, was used for the outer jacket. This tubing was swage-reduced to 0.750-in. OD. Inconel end plugs to seal the swaging capsule were made from hot-rolled, annealed, and pickled 1-in.-diam bar stock.

EXPERIMENTAL PROCEDURE

The effects of type of powder, temperature, total reduction in area, and postswaging heat treatment on the density and structure of swaged uranium-molybdenum alloy powders were investigated. The powders were encapsulated in the containment materials and consolidated by rotary swaging. In general, effects on the sheathing materials were not evaluated.

Prior to loading, the elemental powders were dry blended for 2 hr in an oblique blender. The prealloyed powders were not blended.

When it became apparent that the oxide coating on the prealloyed shot caused poor results, the following chemical procedure was developed to remove the film. The powder was cleaned in a solution containing 194 ml of 3% H_2O_2 -6g $Na_2C_2O_4$, buffered to a pH of 4.5 with oxalic acid, and heated to 90°C.

Table 1. Information on Powders for Swaging

Nominal Composition	Type	Grade and Size	Chemical Analysis										X-ray Diffraction Analyses ^a		
			U (%)	Mo (%)	O ₂ (ppm)	H ₂ (ppm)	Inert +N ₂ (ppm)	C (ppm)	Fe (ppm)	Mg (ppm)	Ca (ppm)	Si (ppm)	Phases	Intensity	
U-10 wt % Mo	Prealloyed shot	Spherical -100 and -70 +100 mesh	89.85	10.35	580	---	---	---	175	535	90	---	70		
U-15 wt % Mo	Prealloyed shot	Spherical -100 mesh	84.91	13.7 ^b	---	---	---	---	158	464	3	---	35	γU(Mo) Mo UO	S M S
Uranium	Hydride	Irregular -325 mesh (av ~5μ)	97.82	---	42,000	2500	2000	---	446	150	2	40	135	αU βUH ₃ UO ₂	MS MF MS
Uranium	Calcium reduced Batch 94	Spheroidal -325 mesh (50% < 16μ)	99.26	---	1,430	---	---	---	136	460	>190	125	---	αU UO ₂	S M
Uranium	Calcium reduced Batch 95	Spheroidal -325 mesh (50% < 15.5μ)	99.44	---	4,000	180	42	---	120	80	103	75	---		
Molybdenum	Hydrogen reduced	Good-comm. grade -325 mesh	---	99.85	1900	---	---	---	200	---	---	---	---		
Molybdenum	Hydrogen reduced	Standard grade -325 mesh (av ~4μ)	---	99.9	400	---	---	---	140	---	---	---	---		

^aReported on the basis of the following ratings: where M is medium, S is strong, and F is faint. Diffractograms were taken on Debye-Scherrer camera.

^bNational Lead Value: Mo = 14.9%.

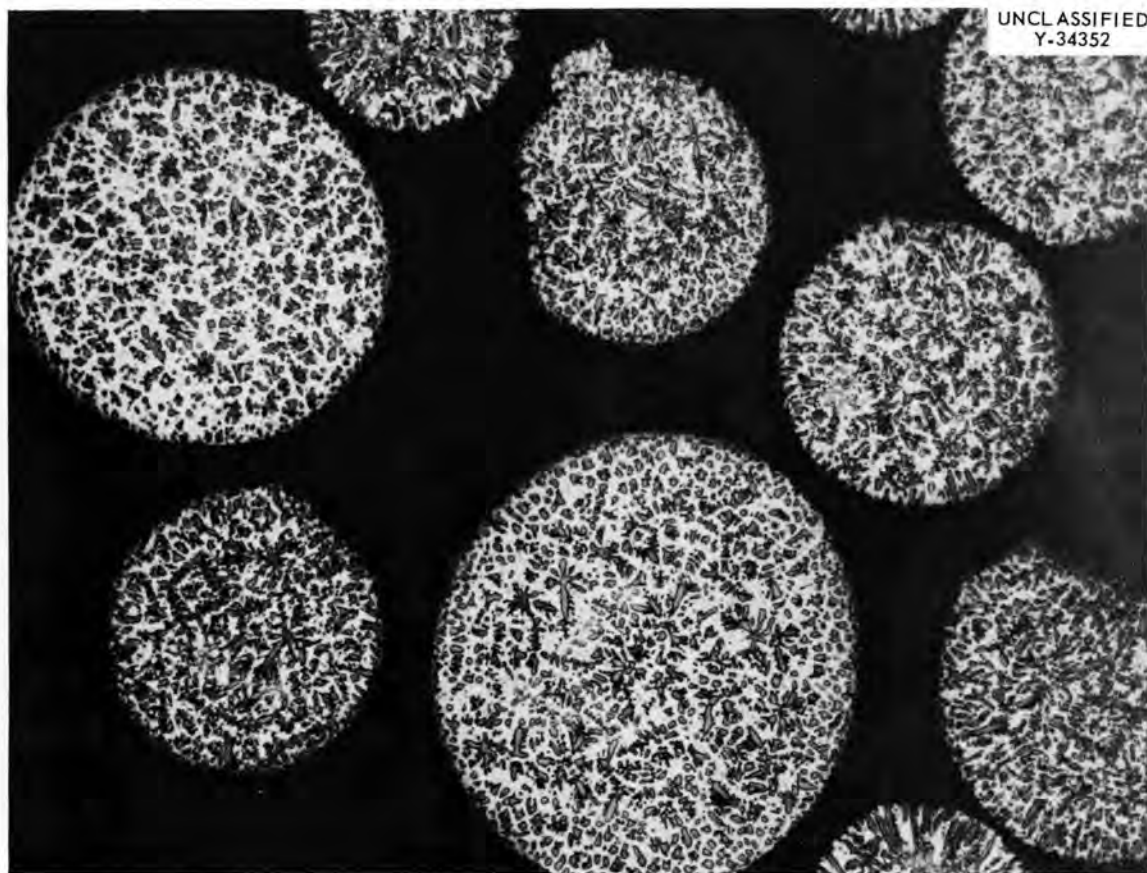


Fig. 1. Polished Cross Section of Prealloyed U-13.7 wt % Mo Shot Showing Cored Structure and Spheroidicity of Particles -100 Mesh. Etchant: 50% HNO_3 -50% $\text{HC}_2\text{H}_3\text{O}_2$. 500X.

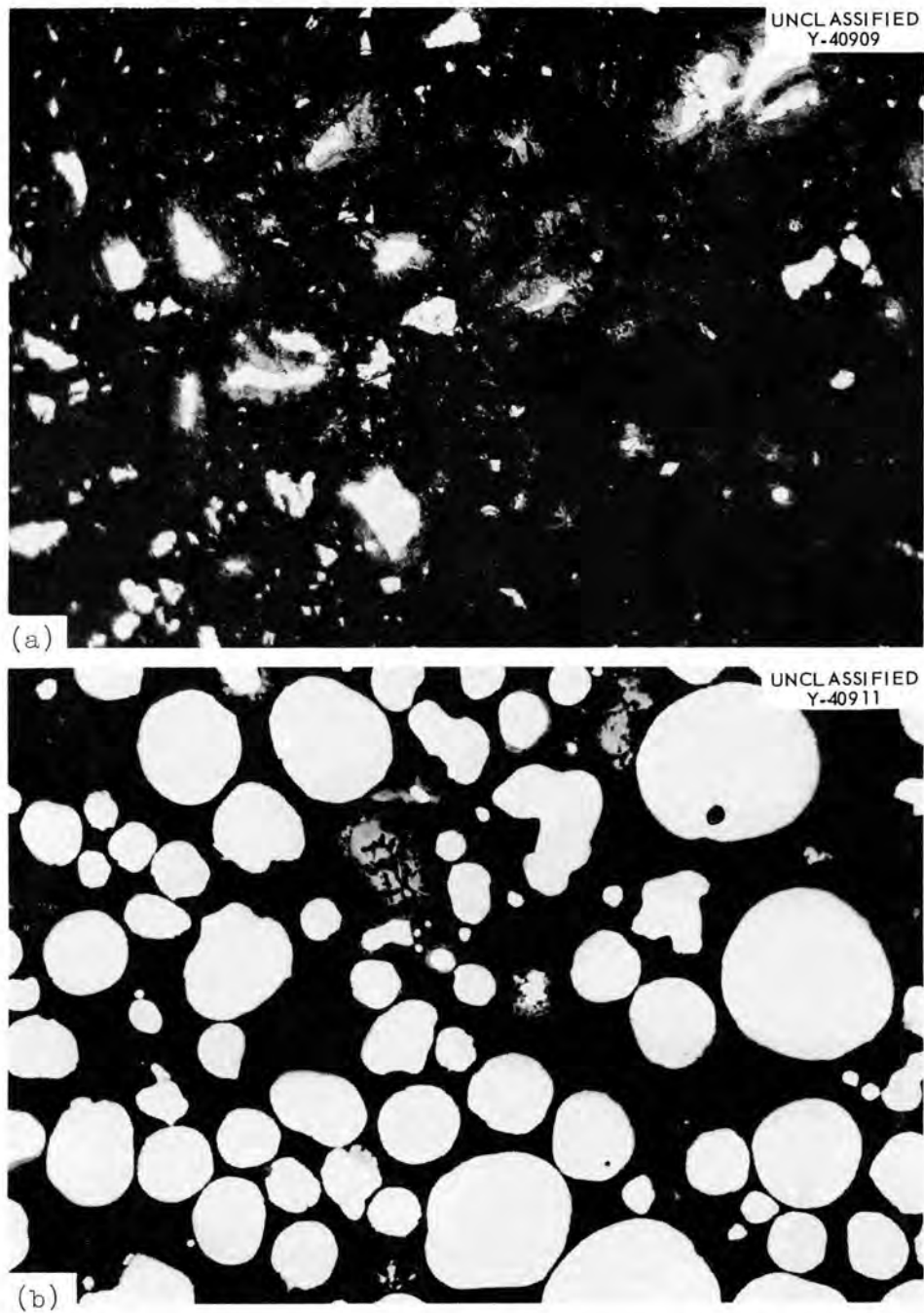


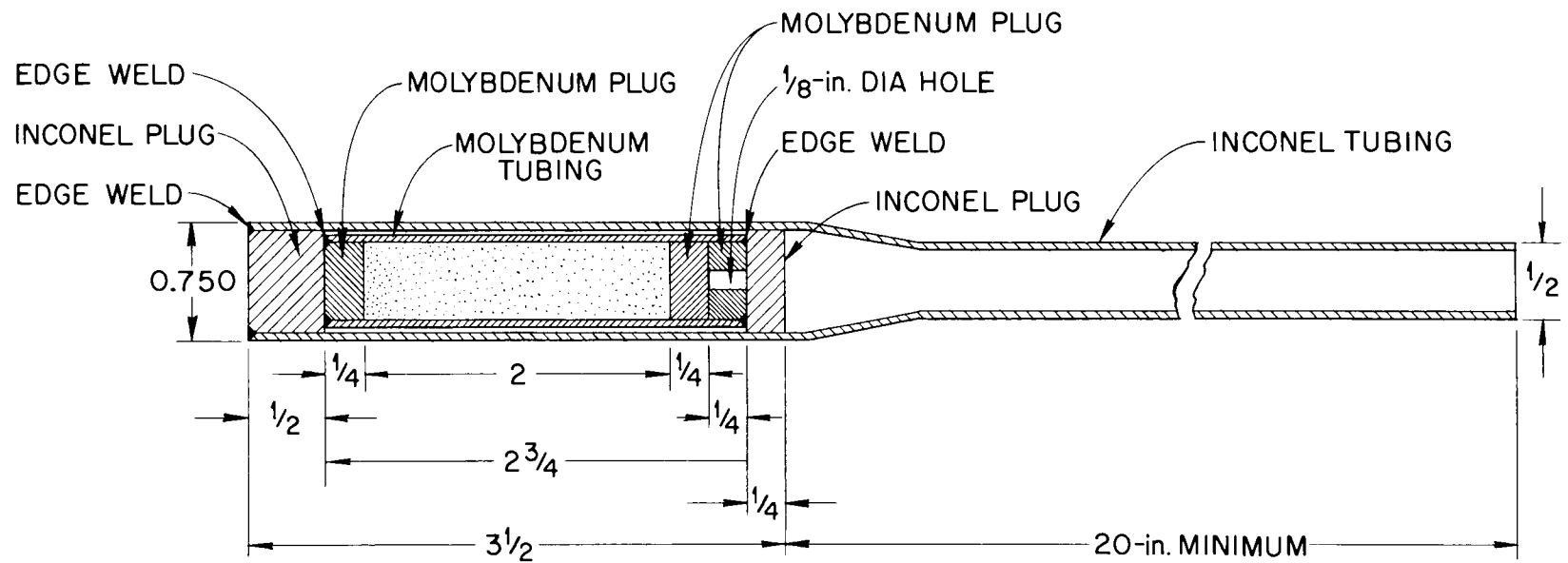
Fig. 2. Polished Cross Section of Two Types of -325 Mesh Uranium Powder. Observe that particles produced by hydride route are extremely fine, irregularly shaped, and grossly contaminated (note surfaces at arrows). Calcium-reduced powder is coarser and has less surface contamination. As-polished. (a) Hydride process powder, 1000X. (b) Calcium-reduced powder, 500X.

The design of the capsules used for elevated-temperature swaging (1050 or 1100°C) is illustrated schematically in Fig. 3. To minimize welding operations, a continuous section of Inconel tubing was swaged to the configuration shown (Fig. 3) and used as the outer sheath. All components were cleaned in hydrogen before loading the powder. The molybdenum inner sleeve was tamp packed with 50 g of uranium-molybdenum powder, seal welded with end plugs, and inserted into the capped Inconel tube. A second end plug was then welded to the Inconel sleeve. All operations including welding by the TIG method were performed in an argon-filled dry box. The molybdenum functioned as a barrier to prevent formation of a eutectic between uranium and the Inconel. (Uranium binary eutectics form with nickel, iron, and chromium at 740, 725, and 860°C, respectively.) The assembled billet was evacuated to a pressure of less than 1×10^{-3} torr, heated with a torch to assure outgassing, and sealed along the stem by hot forging.

Compaction was effected with a 2-die Fenn Model 2F rotary swaging machine and a progressive reduction in die size. A typical swaging schedule is shown in Table 2. The capsules were preheated in air for 1 hr before swaging at the prescribed temperature. As the swaging dies were at room temperature, it was necessary to reheat the capsules in the furnace for 15 min after each swaging pass.

Postswaging heat treatments were performed at temperatures in the vicinity of 1100°C on transverse sections of material in the clad state, both under "dynamic" and "static" vacuum conditions. Because much brighter specimens were obtained by "static sintering" (vacuum encapsulation in quartz containment), all heat treatments were done in the static vacuum in the latter part of the program.

Geometric density determinations of the alloy cores were made from weight and dimensional measurements obtained on 1-1/2-in. lengths of the swaged rod. Thickness of the sheathing materials was determined metallographically and considered in the density determinations. The procedure used in making these calculations is given in Appendix B. Where it was feasible to machine the clad from the core without the uranium-molybdenum alloy spalling, more accurate density measurements were made directly on



DIMENSIONS ARE IN INCHES

Fig. 3. Swaging Capsule for Uranium-Molybdenum Powders.

Table 2. Typical Swaging Schedule

Pass Number	Die Size (in.)	Measurement Outside Diameter (in.)	Pass Reduction Area (%)
1	0.687	0.700	13.0
2	0.593	0.597	27.3
3	0.531	0.541	17.8
4	0.480	0.500	14.8
5	0.445	0.453	17.9
6	0.405	0.410	18.0
7	0.397	0.400	4.9

the core material, both by geometric and CCl_4 -immersion methods. X-ray diffraction and chemical analyses were also made and correlated with the metallographic examination.

DISCUSSION OF RESULTS

The results of the swaging experiments, which were conducted with both prealloyed uranium-molybdenum powder and mixed elemental uranium and molybdenum powders are discussed in this section.

Prealloyed Uranium-Molybdenum Powder

Table 3 shows the swaged density of prealloyed U-10 wt % Mo shot as a function of swaging temperature, reduction in area, and powder condition.

Densification obtained with room-temperature swaging generally was inferior, while swaging at 650 and 1050°C gave reasonably high densities. For cold swaging, the density increased with increasing reductions in area. At elevated temperature, however, reduction used did not appear to markedly affect the density, except perhaps for the acid-cleaned shot. The best interparticle bonding for as-received shot was exhibited by specimens swaged at 1050°C, but even here bonding was largely of a mechanical nature and the materials were not strong.

Table 3. Effect of Temperature and Reduction in Area on the Swaged Density of Prealloyed U-10 wt % Mo and U-13.7 wt % Mo Shot

Composition	Swaging Temperature (°C)	Reduction in Area (%)	Theoretical Density ^a (%)	
U-10 wt % Mo	Tapped Bulk Density		61.3	
	Room Temperature		49.9	85.9
			61.6	86.7
			79.5	89.4
			88.4	90.8
	650		46.9	Powder spalled
			56.0	92.4
			66.7	92.5
			76.3	94.5
			83.2	93.2
	1050		48.4	92.4
			54.3	96.2
			56.8	92.2
			58.3	92.8
		67.1	97.8	
		78.0	91.7	
U-13.7 wt % Mo	1050 ^b	55.4	94.6	
	Tapped Bulk Density		99.2	
	650		62.4	
			56.6	Powder spalled
			66.4	96.2
			74.8	93.0
	1050		84.2	98.7
			46.0	92.6
			55.3	95.7
			59.8	93.8
	68.6	98.9		
	76.5	93.5		

^aU-10 wt % Mo: Theoretical density = 17.3 g/cc. U-13.7 wt % Mo: Theoretical density = 16.7 g/cc.

^bShot was acid cleaned before swaging.

Figure 4 shows the effect of swaging temperature on microstructures of the as-received prealloyed shot. In agreement with the density determinations, the lowest porosity was displayed by the specimen swaged at 1050°C. The lack of interparticle bonding was attributed to an oxide film still observed on the powder particles after swaging (Fig. 4e). Poor adhesion was confirmed when the U-Mo cores spalled upon machining.

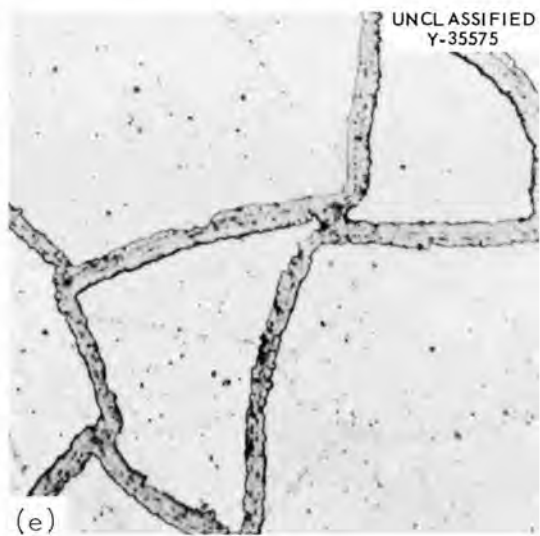
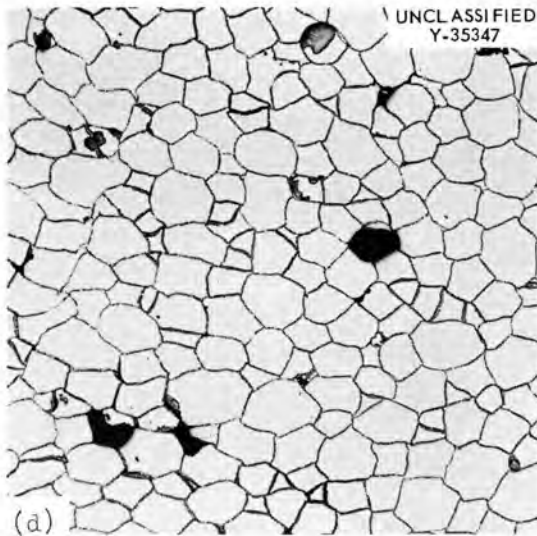
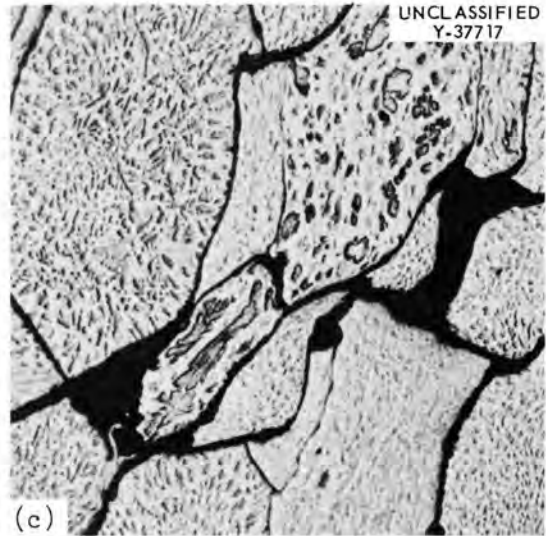
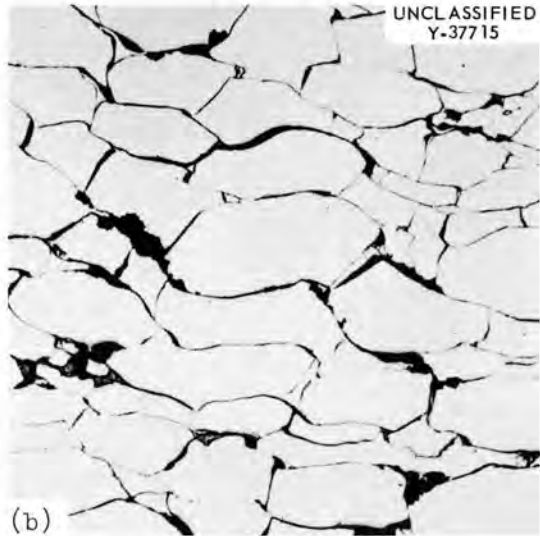
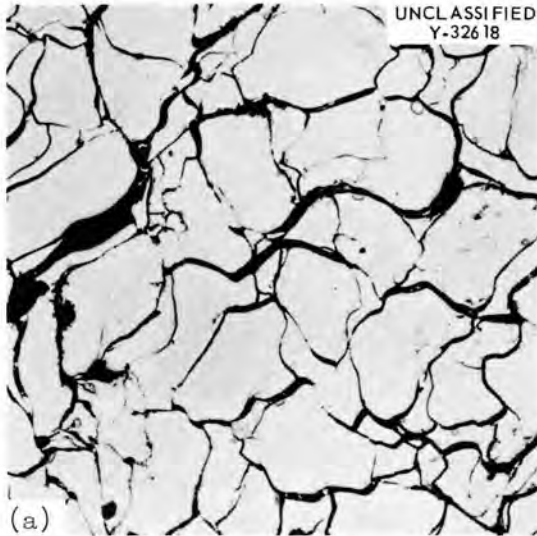


Fig. 4. Effect of Swaging Temperature on Microstructure of Swaged Prealloyed (As-Received) U-10 wt % Mo Shot. Transverse cross section. Reduced 6%. (a) Room temperature, as-polished, 200X. (b) 650°C, as-polished, 200X. (c) 650°C, etchant: 1% HNO₃-1% HC₂H₃O₂, 500X. (d) 1050°C, as-polished, 75X. (e) 1050°C, etchant: 1% HNO₃-1% HC₂H₃O₂, 750X.

To achieve good cohesion in swagings prepared from prealloyed shot, two approaches were investigated. The first method used a postswaging heat treatment at 1100°C while the second and more successful approach consisted of cleaning the uranium-molybdenum shot as previously described preparatory to swaging.

In general, the postswage annealing of samples fabricated at 1050°C did not significantly improve density or bonding and cores still spalled with machining. Annealing caused some coalescence of the oxide film, and it appeared that some additional oxidation may have occurred during the heat treatment. Other investigators also have observed that under conditions of high-sintering temperatures for long times, surface tension forces can cause spheroidization of films or impurities.²

Capsules containing acid-cleaned -70 +100 mesh shot were swaged at 1050°C to 50 and 70% reductions in area (Table 3). While one sample reduced 50% showed inferior bonding, another swaged to a 70% reduction could be machined without spalling. After a 120-hr heat treatment in static vacuum (24 hr at 1150°C followed by 96 hr at 1100°C), both specimens were machinable.

Metallographic examination revealed that an oxide film remained after swaging the cleaned powder; however, it was substantially thinner than for the as-received powder. This effect is illustrated by a comparison of Fig. 4 with Fig. 5 showing the oxide film after swaging for the powder in the as-received and cleaned conditions, respectively. Figure 5 shows the cleaned and swaged uranium-molybdenum alloy powder after the postswaging anneal. Note that the continuous oxide structure which formerly separated the metal particles has essentially coalesced into intermittent oxide inclusions at the grain boundaries. Such a structural change would be expected to significantly improve the strength of the body.

²H. A. Saller and F. A. Rough, "Alloying by Powder Metallurgy," p 126 in Powder Metallurgy in Nuclear Engineering, Proceedings of the Conference on Powder Metallurgy in Atomic Energy, Philadelphia, Oct. 20, 1955, American Society for Metals, Cleveland, 1958.

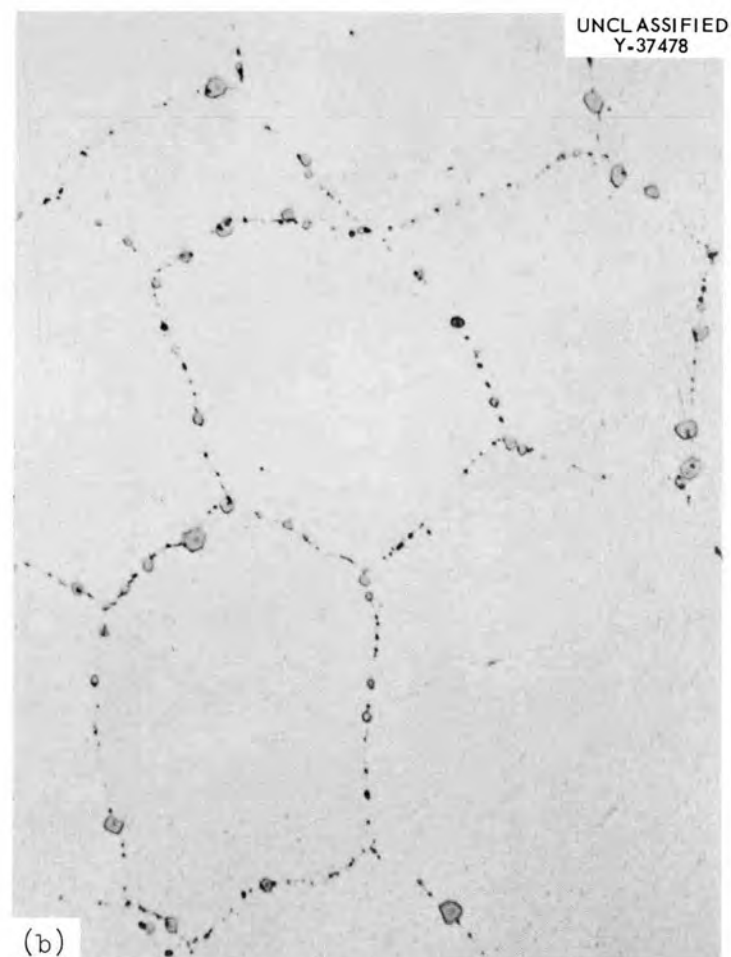


Fig. 5. Effect of Postswage Annealing on Acid-Cleaned Prealloyed U-10 wt % Mo Shot Swaged at 1050°C to 70.6% Reduction in Area. (a) As-swaged. Note oxide particles at grain boundaries. (b) Same specimen after annealing in "static" vacuum for 24 hr at 1150°C + 96 hr at 1100°C. Note spheroidization of oxide. Etchant: 1% H₂O₂-1% NH₂OH. 500X.

The effects of powder condition, swaging temperature, reduction in area, and postswaging heat treatment just discussed for the U-10 wt % Mo shot were found to apply generally for the U-13.7 wt % Mo shot. Typical densities are included in Table 3. The higher molybdenum content did not appear to adversely affect the ease with which swaging was accomplished.

Elemental Uranium-Molybdenum Powders

Concomitant with the work on prealloyed powders, studies were performed using mixtures of elemental powders. Elemental powders afford an advantage in that they are generally softer and more compactible than the alloy powders. On the other hand, to form a homogeneous alloy, the mixed elemental powders must interdiffuse as well as sinter when heated to achieve desired results. As will be shown subsequently, homogeneity proved to be a problem.

Preliminary swaging studies using blended hydride uranium and -325 mesh molybdenum gave densities less than 55% of theoretical. The poor swageability probably resulted from the low tapped density and excessive contamination of this powder. The tapped density of U-15 wt % Mo with hydride uranium was only 27.1% of theoretical, whereas with calcium-reduced uranium it was twice as high. Work with this material was therefore discontinued.

Swaging with calcium-reduced uranium, on the other hand, resulted in structurally strong alloys of reasonably high density. Photographs showing machined sections of swaged rods are shown in Fig. 6. The effect of swaging temperature on density for the calcium-reduced U-10 wt % Mo is illustrated in Table 4. To achieve high density along with good structural strength, high-swaging temperatures again were required. Powders swaged at 650°C were fairly dense (Table 4), but some spalling occurred when the rods were machined. Swaging at 1050 and 1100°C gave good interparticle bonding as well as high density, provided a suitable reduction was used.

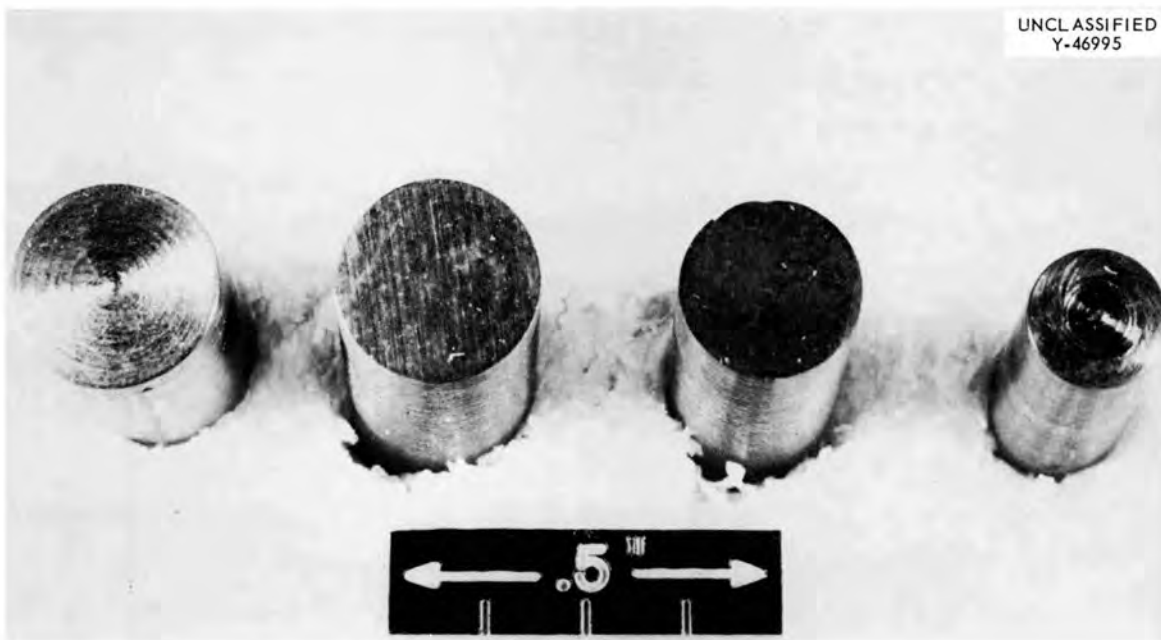


Fig. 6. Machined Sections from 1050°C Swaged Rods Showing Bare Core.

Table 4. Effect of Fabrication Temperature on the Swaged Density of Calcium-Reduced Uranium-10 wt % Molybdenum Powders

Swaging Temperature (°C)	Reduction in Area (%)	Density (% of Theoretical) ^a
650	57.9	88.7
1050	55.5	96.3
1100	56.9	95.0

^aTheoretical Density = 17.3 g/cc.

Increasing the reduction in area in the range examined (50 to 70%) did not consistently increase density (Table 5) although there was a general improvement at high reductions. This was most evident with swagings incorporating the finer molybdenum powder. Additionally, interparticle bonding was inferior at lower reductions, and at high reductions crack formation in the core and clad was a problem. The amount of reduction did not appear to affect the degree of alloying (only 15 min reheats were given between passes). A reduction in area of about 60% appeared to give the most favorable overall results.

X-ray diffraction and metallographic examination revealed that the specimens swaged at 1050°C contained undissolved particles of molybdenum along with some UO₂ as a contaminant (Fig. 7a). X-ray diffraction showed the rods swaged at 1100°C to be solely the gamma-phase alloy; however, metallographic examination showed slight evidence of inhomogeneity. A constituent in trace amounts, probably patches of the gamma phase rich in molybdenum content, remained in the structure (Fig. 7b).

In addition to increasing swaging temperature from 1050 to 1100°C, two other procedures were explored for overcoming segregation. In the first, a finer size molybdenum powder was substituted for the standard -325 mesh powder. The second approach consisted of a 24-hr postswaging anneal in "static" vacuum at 1150°C. Neither of these changes proved effective in improving homogeneity nor did they seem to improve other properties such as density (Table 4).

Table 5. The Effect of Reduction in Area on the Density of Elemental Uranium (Ca-Reduced) + Molybdenum Powders Swaged at 1050°C

Composition (wt %)	Uranium	Molybdenum	Reduction in Area (%)	Density ^a (% of Theoretical)
U-10 Mo	Calcium reduced No. 94	MCA	53.2	95.3
			55.5	95.2
			60.8	97.6
			68.8	96.6
			71.9	97.6
U-10 Mo	Calcium reduced No. 94	WC	53.5	92.0
			62.6	95.9
			68.6	97.4
U-15 Mo	Calcium reduced No. 94	MCA	50.9	96.8
			53.6	97.6
			60.6	97.3
			67.3	97.4
			71.4	97.0

^aU-10 wt % Mo: Theoretical Density = 17.3 g/cc.

U-15 wt % Mo: Theoretical Density = 16.7 g/cc.

The same parameters studied in developing swaging for the elemental U-10 wt % Mo alloy were examined with the elemental U-15 wt % Mo composition. The results differed little from those for the former composition. The geometric densities were somewhat higher for the alloy of higher molybdenum content (Table 4), but in other respects the results were similar.

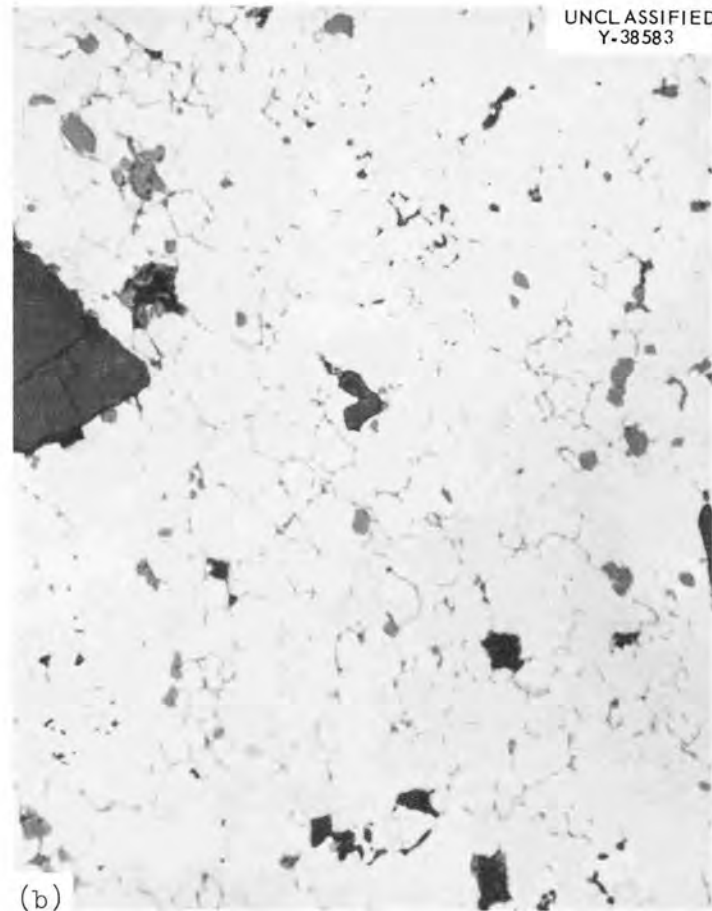
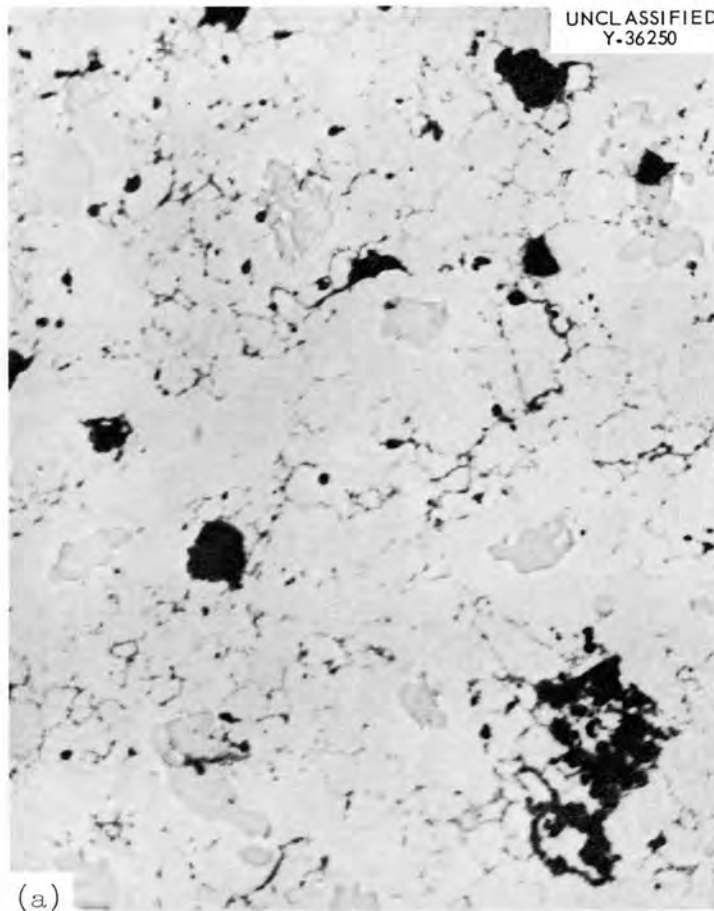


Fig. 7. Microstructures of Swaged Elemental U-10 wt % Mo Alloy Containing Calcium-Reduced Uranium. (a) Swaged 53.2% in reduction of area at 1050°C, giving 95.3% of theoretical density. Uranium-molybdenum, gamma phase (white); molybdenum (light gray indicated by arrows); UO₂ (dark gray); voids (black). (b) Swaged at similar reduction at 1100°C, giving 95.0% of theoretical density. Same as (a) except light gray (at arrow) probably represents molybdenum-rich gamma phase. As-polished. 500X.

SUMMARY AND CONCLUSIONS

1. Uranium-molybdenum alloys containing 10 to 15 wt % Mo can be prepared by hot swaging either (a) mixtures of calcium-reduced uranium powder with elemental molybdenum powder or (b) specially cleaned prealloyed uranium-molybdenum powder. These powders can be compacted to densities in excess of 95% of theoretical by rotary swaging at 1050 to 1100°C. The swaged particles are well bonded to each other and the product appears to be structurally strong.

2. The as-received prealloyed shot can be swaged to densities as high as for the other powders, but acceptable interparticle bonding is obviated as a result of surface oxide on the powder. It is, therefore, necessary to clean this material in order to achieve satisfactory bonding in the swaged product.

3. The alloys made by mixing elemental uranium and molybdenum powders are homogeneous except for slight traces of molybdenum-rich gamma phase which persists after swaging at 1100°C.

4. Factors proving important in achieving good swaging results include: (a) minimizing powder particle surface contamination, (b) high tap density of powders, (c) sufficiently high-swaging temperature, (d) degassing and prolonged annealing before swaging, and (e) maintenance of a good atmosphere in the swaging capsule during processing.

5. In view of the encouraging results realized in swaging the uranium-molybdenum powder fuels, it is suggested that this technique might be suitable for the fabrication of dispersion fuel elements of UO_2 or UC dispersed in a U-Mo matrix. Specific work is needed to optimize interparticle bonding and ameliorate inhomogeneity.

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APPENDIX A

PREPARATION OF POWDERS

Prealloyed Uranium-Molybdenum Shot

Prealloyed shot was procured from an outside vendor. Chunks of uranium and molybdenum powder were vacuum-induction melted and bottom poured onto a rapidly spinning zirconia disk, which dispersed the alloy within a mild steel chamber where the droplets solidified. Spherical particles having a cored dendritic structure were produced. The shot was characterized by a tenacious oxide film.

Hydride Uranium

This material was obtained from the Y-12 plant. Uranium metal chips were hydrided in a fluidized bed at 300 to 350°C under pressure. The brittle hydride was rendered into very fine, irregular particles. The hydride powder was subsequently decomposed in helium between 350 and 550°C. The powder was extremely impure and flocculent.

Calcium-Reduced Uranium

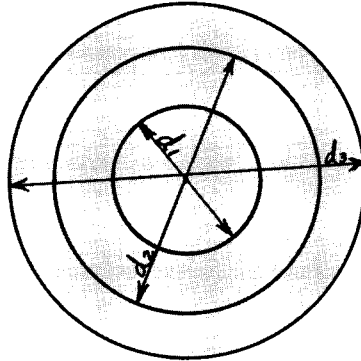
Uranium also was made by the calcium-bomb reduction of UO_2 . The resultant particles were spheroidal and finely sized. Contamination was significantly lower than for the hydride route material.

Molybdenum

The molybdenum powders were produced commercially by two vendors using hydrogen reduction. The powder designated WC was somewhat finer than that identified as MCA (see Table 1).

APPENDIX B

METHOD OF CALCULATING SWAGED URANIUM-MOLYBDENUM DENSITY



Geometric density calculations were made from the dimensions and weight of a 1-1/2-in. section cut from a swaged capsule and machined on the ends to form a right cylinder, by applying the following equation:

$$\rho_{U-Mo} = \frac{W_t - W_{Mo} - W_{Inc}}{V_{U-Mo}}$$

$$= \frac{W_t - \pi \frac{(d_2^2 - d_3^2)}{4} l \rho_{Mo} \left(\frac{16.39 \text{cc}}{\text{in.}^3} \right) - \pi \frac{(d_1^2 - d_2^2)}{4} l \rho_{Inc} \left(\frac{16.39 \text{cc}}{\text{in.}^3} \right)}{\pi \frac{d_3^2}{4} l \left(\frac{16.39 \text{cc}}{\text{in.}^3} \right)}$$

where:

- ρ_{U-Mo} = swaged density of contained uranium-molybdenum (g/cc),
- W_t = total weight of machined section (g),
- W_{Mo} = weight of molybdenum in section (g),
- W_{Inc} = weight of Inconel in section (g),
- V_{U-Mo} = volume of uranium-molybdenum in section (cc),
- d_1 = OD of Inconel (in.),
- d_2 = OD of molybdenum (in.),
- d_3 = diameter of uranium-molybdenum (in.),
- l = length of section, approximately 1-1/2-in. (in.),
- ρ_{Mo} = density of molybdenum (g/cc), and
- ρ_{Inc} = density of Inconel (g/cc).

Using $\rho_{\text{Mo}} = 10.2 \text{ g/cc}$ and $\rho_{\text{Inc}} = 8.51 \text{ g/cc}$, the above equation reduces to:

$$\rho_{\text{U-Mo}} = \frac{W_t/l - 131(d_2^2 - d_3^2) - 109(d_1^2 - d_2^2)}{12.9d_3^2}$$

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