POWDER METALLURGY DEVELOPMENT AT SRL

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INTRODUCTION

The Savannah River Laboratory (SRL) is developing a powder metallury (P/M) process for manufacturing reactor-grade fuel tubes containing high wt % U_3O_8-Al cores clad with 8001 aluminum. The P/M cores are made by isostatic compaction. They are assembled in billets, outgassed, and hot-extruded using conventional coextrusion techniques.[1] Cores have been compacted with up to 100% U_3O_8 and tubes extruded with 80 wt % oxide cores.

Irradiation tests have been made using P/M core tubes in the Savannah River reactors. These tubes contained U_3O_8 concentrations up to 59 wt % and no significant swelling or blistering occurred. The tubes were irradiated to $\sim$40% burnup or 1.6×10^{21} fissions/cc of core.

This report discusses both small-scale and production tests for high-density P/M fuel development. The purpose of the P/M development program at SRL is to:

- determine the maximum U_3O_8 content that can be fabricated into thin wall tubes,
- irradiate high-density tubes to high burnup and assess irradiation and dimensional stability,
- continue metal forming studies for extrusion and drawing, and
- evaluate hydrostatic extrusion and hydrostatically assisted drawing of P/M core tubes.

TUBE FABRICATION PROCESS

Production fuel assemblies have a 12.5-ft-long core and generally consist of 3 concentric tubes as shown in Figure 1. The ID and OD of the assembled tubes range from about 1.75 to 3.75 in., respectively. SRP fuel tubes operate at low temperature and use aluminum to facilitate separations. However, the basic technology may be applicable for other fuel tubes and materials, i.e., stainless steel and Zircaloy.

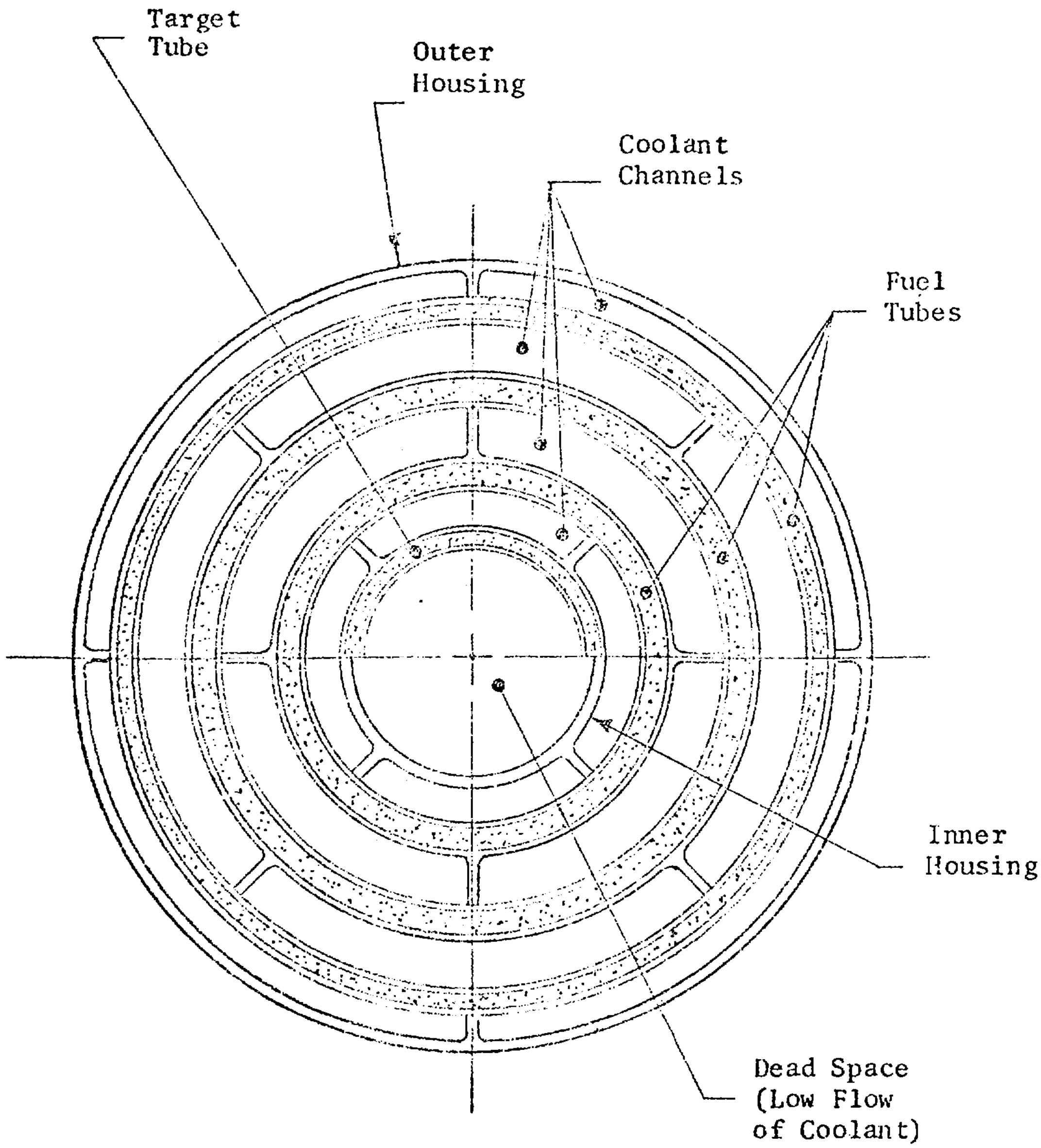

Fig. 1. Cross Section of a Fuel Assembly

Process steps are similar to those used commercially for aluminum tube manufacture. The basic process is divided into fabrication and quality control (QC) steps. Each tube is carefully inspected to insure top quality fuel to prevent activity released to the moderator during irradiation. Determination of the core characteristics led to the development of the Fuel Distribution Analyzer (FDA) and the Nuclear Test Gage (NTG). The FDA is an instrument which uses x-rays to dermine the relative concentration of uranium or U_3O_8 along the length of the tube. High fuel concentrations can cause local heating and burnout of the cladding. The NTG is a small subcritical reactor that measures the reactivity of the tube in 1-ft increments.

PROCESS CONDITIONS FOR FABRICATING P/M FUEL BILLET CORES

These steps are used to make P/M billet cores for reactor fuel tubes at SRP and summarized in the following order:

1. Oxide firing and grinding
2. Blending of U_3O_8 and aluminum powders
3. Isostatic compaction of P/M billet cores
4. Extrusion of fuel tubes
5. In-line recycle of imperfect cores and tubes

Figure 2 shows the flow of material through the overall operation, integrated with the in-line recycle.

1. Oxide Firing and Grinding

UO_3 is the feed material for the U_3O_8-Al compact core process. It is calcined to U_3O_8 in resistance-heated box furnaces at 800°C for 6 hours in nitrogen and air cooled to ambient temperature. Oxide is fired in "Inconel"* 600 pans of critically safe configuration to prevent nuclear criticality incidents. The as-fired U_3O_8 is ground and sieved to provide powder of the desired particle size distribution. Oversize material from grinding is reground while excess undersize material is compacted and reground.

Oxide particle size must be controlled for three reasons:

- Maximum particle size must be limited to avoid large hard particles that can penetrate the tube cladding and/or cause hot spots sufficient to produce melting during irradiation. The maximum particle size is 100 US standard mesh (149 μ).
- The quantity of -325 mesh (<44 μ) particles is restricted for radiation stability.
- The particle size distribution <100 mesh is controlled to match the size distribution of the aluminum powder to obtain a blend with acceptable homogeneity.

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*Trademark of International Nickel

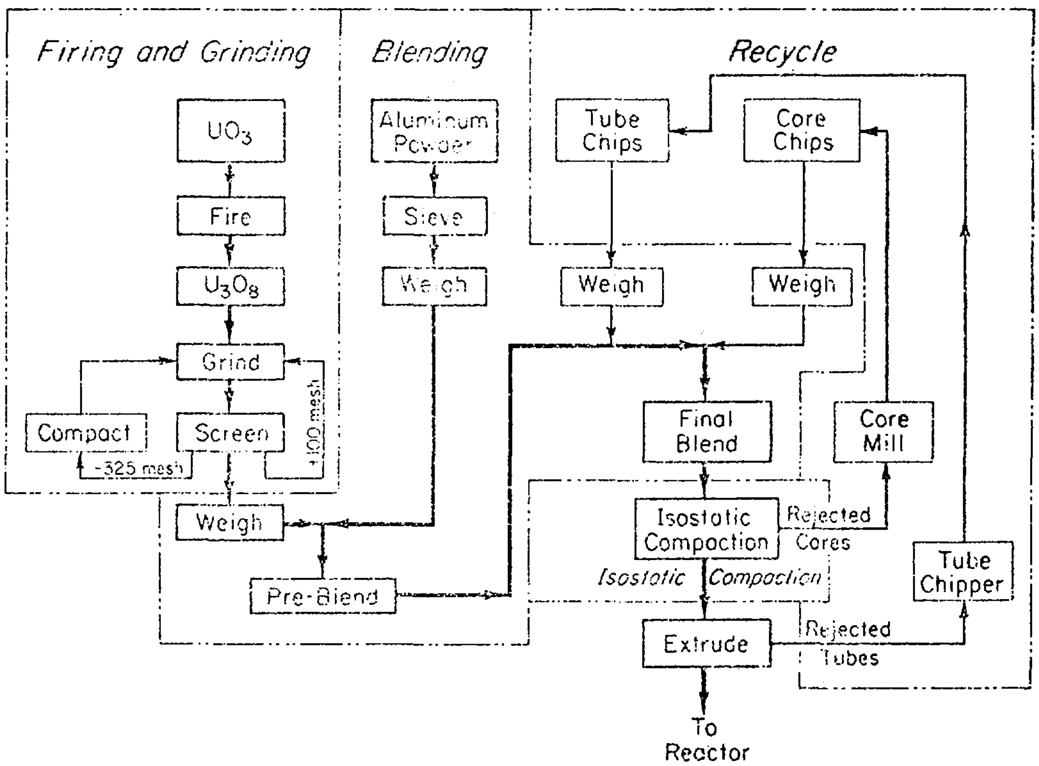

Fig. 2. Block Diagram for P/M Core Fabrication.

U_3O_8 is ground in a commercial roll grinder modified to provide precise roll gap adjustment, variable speed, minimal holdup of powder in the housing, and to minimize dust release. U_3O_8 is fed into the grinder by a separate vibratory feeder at a uniform rate and distributed across the entire width of the rolls. Ground U_3O_8 is discharged directly into a commercial vibrating-screen separator. Powder is discharged from the separator into critically safe containers. Oversize material is recycled directly through the grinder. If excess -325 mesh material is produced from one batch, it is blended into batches that have low -325 mesh contents. If all excess -325 mesh material cannot be utilized in this manner, it is compacted and reground.

The particle size distribution depends on the properties of the oxide, the grinding conditions, such as roll gap and pressure, roll speed, and feed rate. Grinding conditions were developed to produce a maximum of -100 mesh powder per pass through the grinder and at the same time maintain a maximum of 40% -325 mesh material. The particle size distribution of the -100/+325 mesh material produced using these conditions does not exactly match that of the aluminum powder (Figure 3); however, the matchup is close enough that it blends adequately with the aluminum.

2. Blending of U_3O_8 and Aluminum Powders

The U_3O_8 is uniformly blended with aluminum powder to produce material for the billet fabrication process. To overcome the tendency of fine U_3O_8 powder to agglomerate and segregate, preblending and sieving steps were incorporated. After a 5-minute preblend in a horizontal drum blender, the powder in the drum, which has an 80-mesh screen at its outlet, is attached to a modified drum blender or tumbling blender and transferred through the vibrating screen (Fig. 4) into the final blend container. This final screening breaks down the agglomerated U_3O_8 in the feed, and ensures that no large particles get into the final blend. Thereafter, the aluminum powder in the mixture hinders any reagglomerated U_3O_8. The drum and screen are then removed, the blender is closed and adjusted to horizontal blending attitude, and the feed is tumbled to obtain a uniform mixture. Then the compaction mold assembly is attached to the open blender and the rotating blending is inverted to transfer the feed into the mold. The blend in the mold is then vibrated to uniform prepressing density (~55% of solid density), the mold is sealed, and the powder is subjected to isostatic compaction to ~80% of solid density.

The U_3O_8 and aluminum powder tend to segregate after blending because of the differences in density (8.4 g/cm^3 for U_3O_8 and 2.7 g/cm^3 for Al) and in particle size distribution of the two powders. Segregating mechanisms such as pouring and vibrating are controlled closely. Blended powder is transferred from the mixer to the mold (which is connected directly to the mixer) by tilting the mixer as it rotates and tumbles the powder directly into the mold. Mold vibration, which is necessary to produce uniform mold-to-mold powder density, is limited to ~20 seconds to reduce potential segregation.

Tests with Type 101 aluminum powder and ^{235}U-depleted U_3O_8 powder compared the efficiency of the tumbling blender with that of a standard horizontal drum blender. The basic difference is that the tumbling blender has 1-in.-diameter pins which increase shear mixing. The particle size distribution of the U_3O_8 was varied from a typcial as-ground distribution to all -325 mesh.

Twelve samples, each equivalent in blend volume to a 0.25 by 0.25 in. section of extruded core, were taken at specified intervals from the blend.

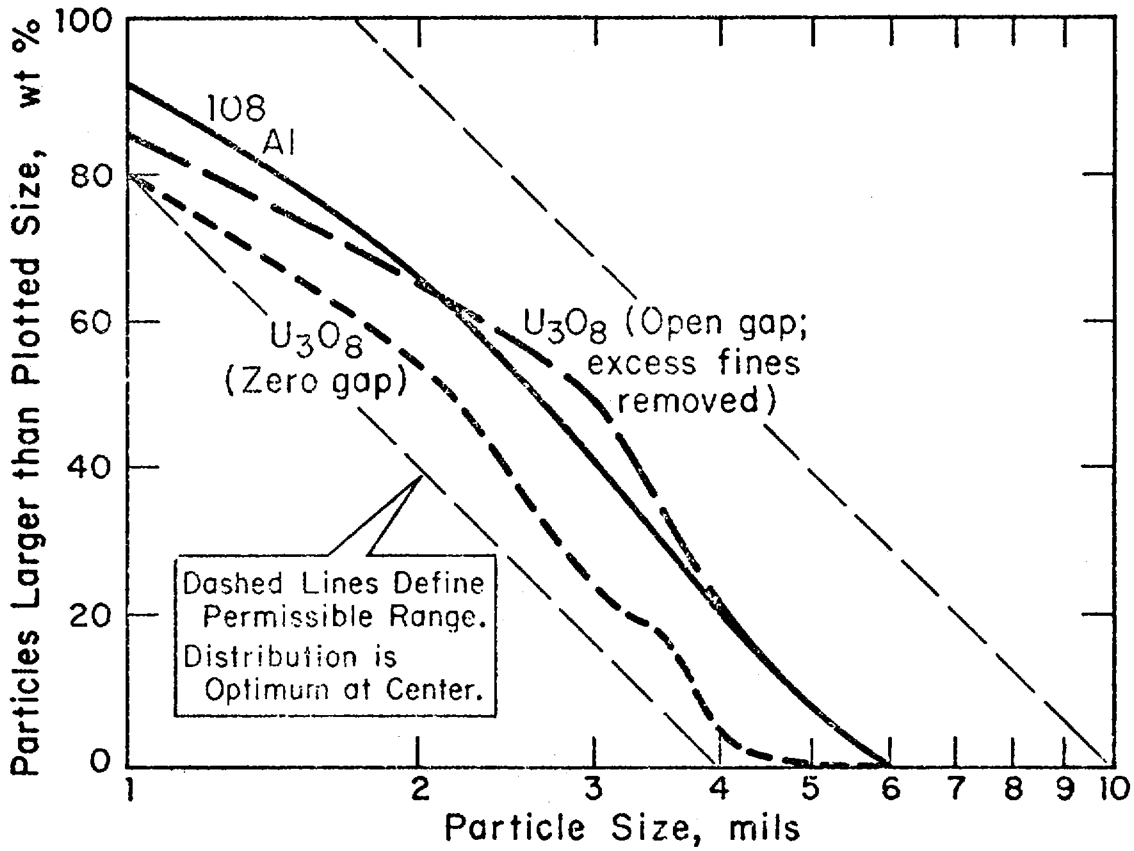

Fig. 3. Particle Size Distribution for Roll Ground U_3O_8 and 108 Aluminum Powder.

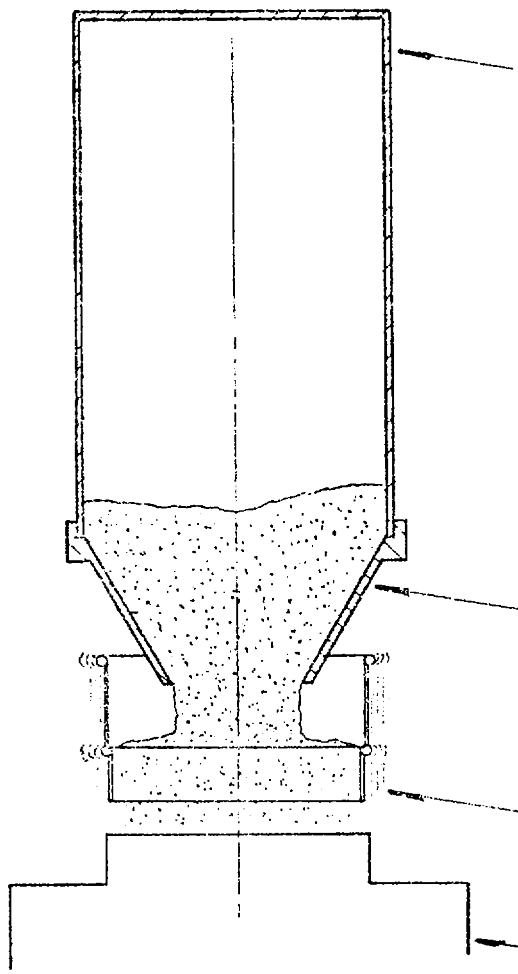

Fig. 4. Powder Pre-Blend and Sieving Apparatus

A thiefing procedure was developed to minimize errors. U_3O_8 content was determined by neutron activation with estimated accuracy of ±0.5 wt % U_3O_8.

Results for the tumbling blender and a standard horizontal blender showed the superiority of the tubmling blender for blending fine (-325 mesh) powders. The analysis of the data was carried out by expressing the coefficient of variation (CV) as a function of the number of blender revolutions. The coefficient of variation is defined as the standard deviation of the theifed samples expressed as a percentage of the mean oxide content. For a 26 wt % mixture, CV for the tumbling blender was 50% less than the value for the standard blender remained less with an average value of ~2.5% after 200 revolutions (10 minutes). The CV for a 40 wt % U_3O_8-Al blend with the specified oxide particle size distribution is shown in Fig. 5 for the tumbling blender. After 100 revolusions, CV average about 5%, which corresponds to 10% variation in uranium content per 0.25 × 0.25 in. section of the core at 95% confidence.

3. Isostatic Compaction of P/M Billet Cores

Isotatic compaction, in which powder is sealed in a 0.5-in.-thick elastomeric urethane mold and compacted under high fluid pressure, is used to form the P/M cores. Urethane bag life has not been determined, but over 150 cores have been made from a single bag with no indications of wear. Cores made by this process slide easily from the depressurized mold; they have very uniform density and the total uranium content can be controlled precisely. Although the OD of the core does not have the dimensional uniformity of cast or machined cores because of the elasticity of the mold, the compact is fully suitable for direct insertion in the billet assembly. The core ID, formed by a steel mandrel, is always the same. Variations in core wall thickness of ±0.03 in. (average wall thickness 1.4 in.) and variations in OD of ± about 0.05 in. (average OD 7.4 in.) have been measured. These variations correspond to less than 3% variation in thickness of the extruded core.

Core made in a mold with a cylindrical ID tend to have an hourglass shape because of an end restraint by the mold. To compensate for this effect, a barrel-shaped mold has been designed and refined to produce tight-cylindrical unmachined cores.

4. Extrusion of Fuel Tubes

As-compact P/M cores are extruded using either a 520-ton laboratory or 3000-ton production press. The extrusion billet is made by capsulating as-compacted cores in aluminum components. The assembly is evacuated at an elevated temperature to remove gases. This high temperature outgassing operation also sinters the compact, but no reaction between U_3O_8 and aluminum has been observed. Before extrusion, the billet is preheated and coated with a graphite-lead-oil lubricant. The tooling temperatures range from 175°C to 400°C depending on the type of tube being extruded.

5. In-Line Recycle of Imperfect Cores and Tubes

A method of in-line recycle of the ^{235}U from imperfect tubes and cores is essential. The P/M compacts or tubes cannot be recycled by melting (as used for alloy) and chemical processing would be time consuming and would increase liquid waste, so mechanical recycle of rejected cores and tubes was developed.

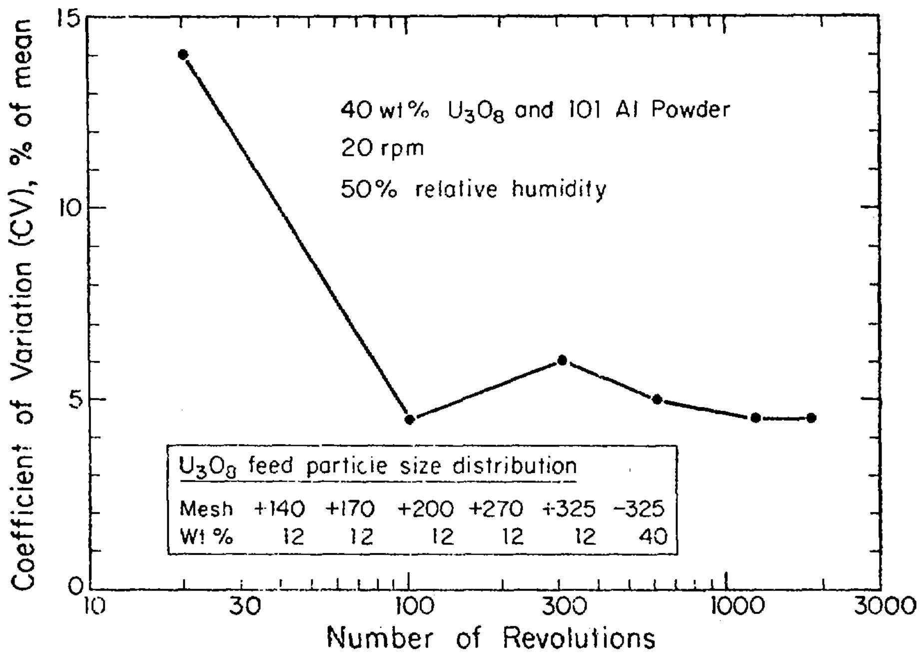

Fig. 5. Test of Modified Tumbling Mixer.

Laboratory tests have demonstrated that P/M cores can be pulverized and recompacted. The pulverized material is ~10% more dense than a powder blend of the same U_3O_8/Al ratio. Commercial pulverizing equipment can reduced compacts to -80 mesh powder which is satisfactory for recylcing. The pulverizer is installed in a special glove box to contain any dust that may be formed during operation.

Four 2-flute, spiral, 0.5-in.-diameter solid carbide cutters are used to chip the core of an oxide tube from which 2/3 of the cladding has been removed. Radiography of a compact containing 18% chips showed good uniformity. For decladding, a machine has been designed to remove ribs from ribbed tubes (2/3 of the cladding is then removed by alkaline etch). The four cutters reduce a full-length tube core to chips in <2 hr. The chips are uniform in size (about 3 by 80 by 120 mils) and only slightly curled.

The final blend for isotatic compaction can tolerate as much as 20 wt % of the chips. This is an acceptable addition rate because more than 90% of the products meet specifications.

CHARGE CALCULATIONS

All charge calculations for a billet core are made using an IBM 360 computer. The computer program determines the amount of U_3O_8 and aluminum needed for each tube type and given an inventory for ^{235}U accountability.

The oxide is received in 10-kg batches each of which contains a different isotopic composition. The program determines the average composition and the amount of high and low enriched material for each core needed to obtain a final blend with the average isotopic composition.

FABRICATION OF SMALL-SCALE U_3O_8-Al FUEL TUBES

Small-scale tubes are used to demonstrate the process capability for powder compaction and extrusion. The model is compared with production-scale tubes in Fig. 6. Thin wall (0.35-in.) billet cores of 100% uranium oxide were successfully compacted in the SRL isostatic press. Scale model cores were initially made in bags with 1-in.-thick walls, but thinner (0.25-in.) bags had to be used for the small compacts to prevent core breakage when compaction pressure is released. Cores from the thin urethane bags were dimensionally uniform.

For initial extrusion development, small-scale parametric tests are determining the effect of extrusion variables such as die cone angle, tooling temperature, extrusion ratio and ram speed, core composition, and end plug alloy. Two different tube configurations are used: thick wall (with outer cladding/core/inner cladding thickness of 30/70/30 mils) and thin wall (10/20/10). Both tube sizes with cores of 80 wt % U_3O_8 were successfully extruded in the laboratory extrusion press.

The relationships among die cone angle and tooling temperature, core composition, and extrudability are shown in Fig. 7. These variables represent measurable quantities and are parameters for the fabrication process. Tubes

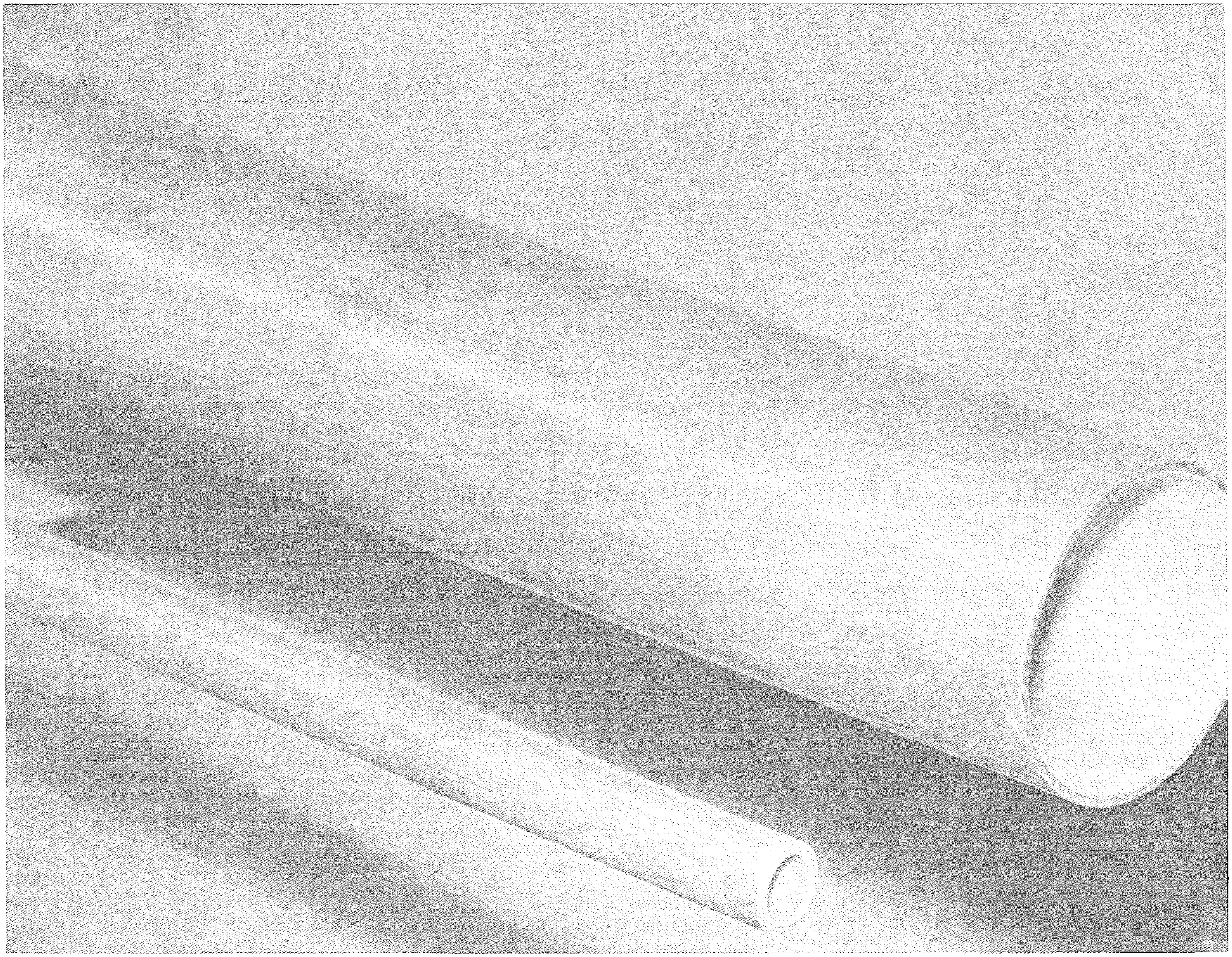

FIGURE 6. Small-Scale and Full-Size Outer Fuel Tubes

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The relationships among die cone angle and tooling temperature, core composition, and extrudability are shown in Figure 7. These variables represent measurable quantities and are parameters for the fabrication process. Tubes are more likely to be free from surface or internal defects at half die cone angles larger than the 45° and at relatively low extrusion

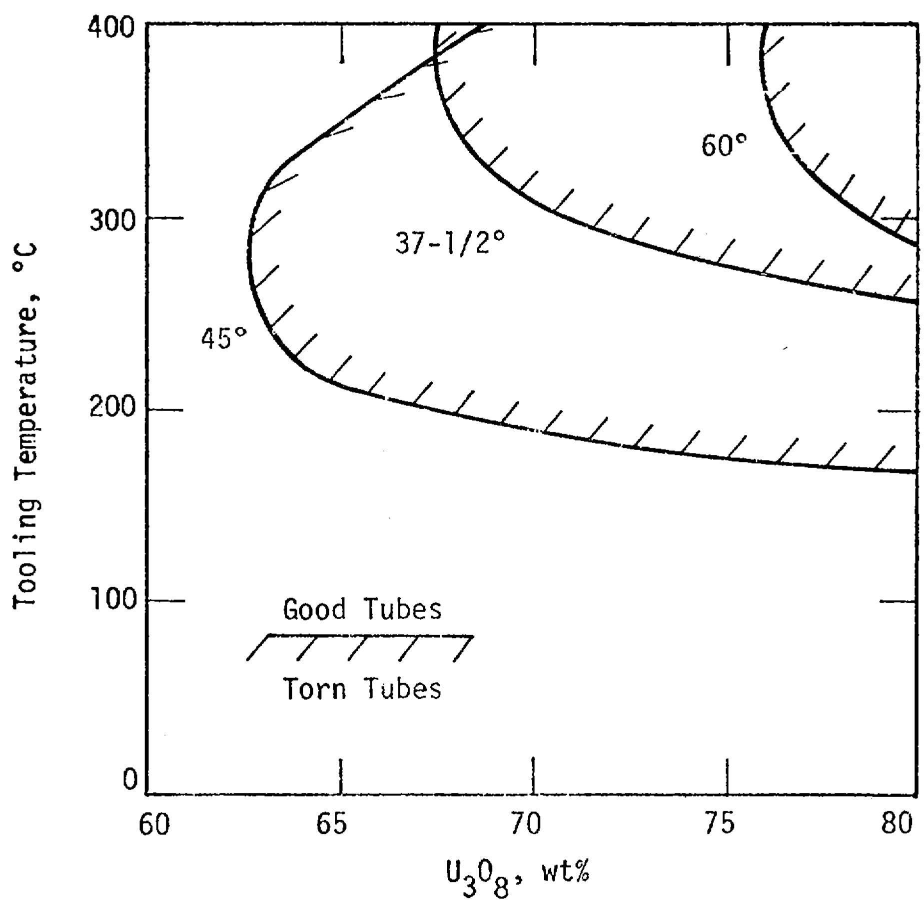

Fig. 7. Effect of U_3O_8 Content, Extrusion Tooling Temperature, and Half the Die Cone Angle on Tube Quality.

tooling temperature (175°C). Tests are in progress at even larger die cone angles and with cores containing more U_3O_8.

MEASUREMENTS OF EXTRUSION FORCES

Extrusion forces are monitored by a transducer on the extrusion press hydraulic system and with a load cell equipped with a strain gauge which measures the force applied to the extrusion die. Results show that the billet-to-container friction is small (approximately 10%). Thus, the present lubricants (graphite, lead, and oil) for the billet and container are effective at both low and high extrusion temperatures. As the billet becomes shorter and the billet-to-container friction decreases, the difference in the forces on the stem and the die does not decrease. Therefore, force difference represents primarily internal work in deforming the billet rather than friction between the billet and container.

The force required to extrude small SRL tubes with high U_3O_8 concentrations increases at low tooling temperature and with increasing core concentration as shown in Figure 8.

CLADDING THICKNESS DETERMINATION

The thickness of the cladding over the core in fuel tubes is determined using x-ray fluorescence. The instrument uses silver x-rays from a 100 mCi ^{109}Cd source. The ratio of the α to β peak heights allows determination of the cladding thickness. For a 30-mil aluminum clad tube, the accuracy is ±2 mils. Thicknesses up to 40 mils have been measured but with less accuracy.

P/M IRRADIATION TESTS AT SRP

In 1971-72, nine 20-mil clad fuel tubes and 4 complete or partial 3-tube assemblies with U_3O_8-Al cores were irradiated in three different reactors. These tubes had 18-59 wt % U_3O_8-Al cores. Irradiation conditions are given in Table 1. All tubes irradiated well except one tube each of 29.5 and 59 wt % which developed a small hole in the 20-mil cladding during the latter part of the irradiation. The holes formed in areas of thin cladding which were caused by large or agglomerated U_3O_8 particles in the as-fabricated core. Subsequent work led to a better understanding of process variables and resulted in development of satisfactory techniques for the P/M core fabrication process.

The most recent irradiation test of P/M fuel was in 1976-77 when six 3-tube assemblies with 30 mils of cladding were irradiated to ~40% burnup or 1.6×10^{21} fissions/cc of core. The cores contained 46-57 wt % U_3O_8. The irradiation was completed successfully and final inspection and testing showed no defects associated with irradiation of high wt % P/M fuel.

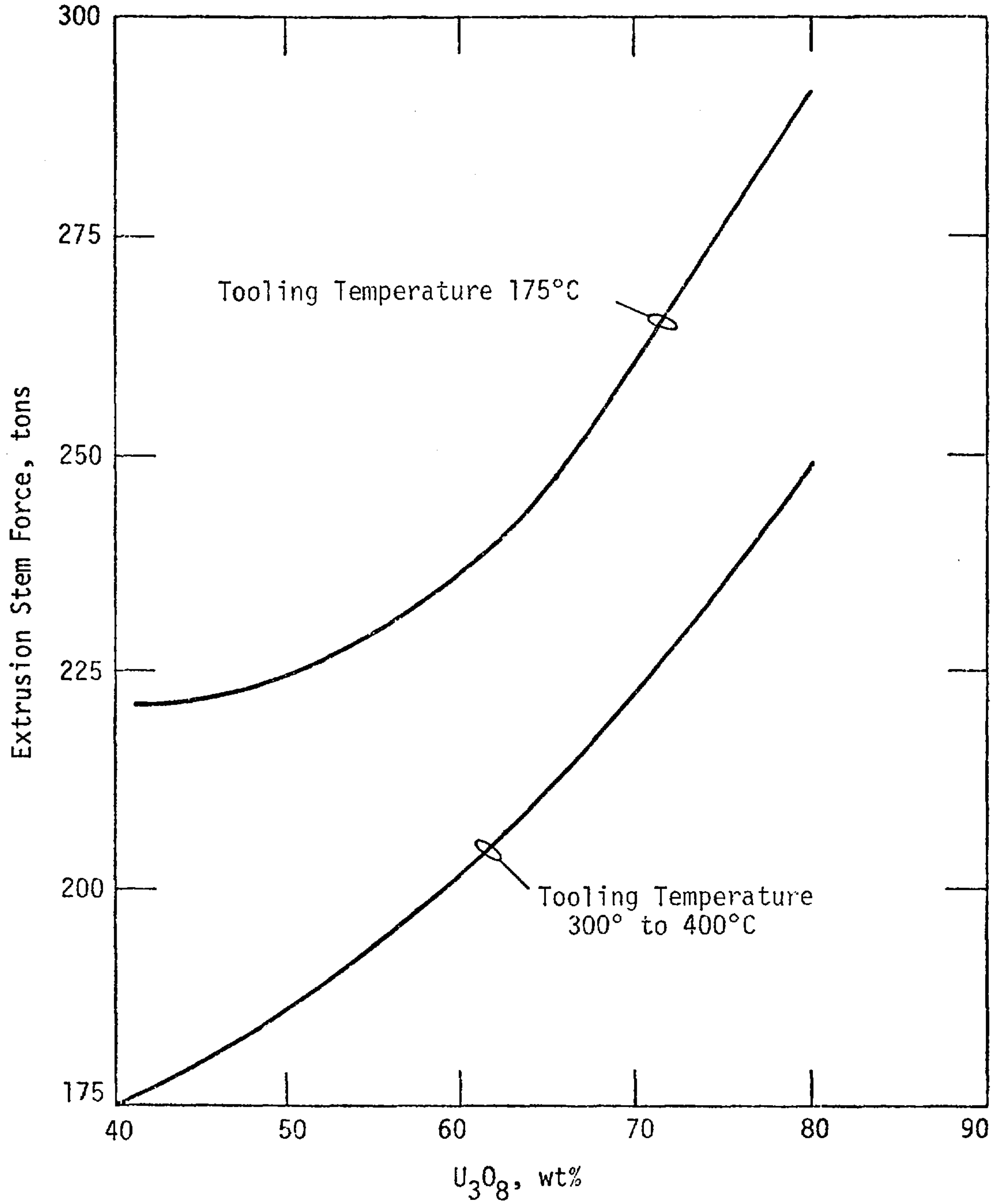

Fig. 8. Effect of Tooling Temperature and U_3O_8 Concentration on Extrusion Force Required.

TABLE I. SRP Irradiation of P/M Fuel Tubes.

Number Irradiated	*Wt %*			*Average Fission Density (10^{21}/cc Core)*	*Max Core Temp,[a] °C*	*Irradiation, days*
	U	*U_3O_8*	*Fines (<325 Mesh)*			
5	12	17.6	68	0.5	195	105
4	25	29.5	68	0.7	200	122
15	33-50	38-59	40	1.0-1.6	220	140-208

a. Calculated from reactor operating data using predicted aluminum oxide thickness and measured hot spot factor.

EVALUATION OF IRRADIATED P/M FUEL

Maintaining the original dimensions of U_3O_8-Al powder fuel cores during irradiation is vital to successful reactor operation. Fission gas blistering of cladding and high-temperature reactions in the core are being studied to develop fabrication specifications. Both irradiated and unirradiated tubes from previous tests have been evaluated to characterize process variables that may affect the safety of operating with P/M fuel cores.

Fission Gas Blistering No blistering or dimensional instability has occurred in plant reactor irradiations of experimental U_3O_8-Al P/M fuel tubes. These tubes, which contained about 18, 30, and 59 wt % U_3O_8, were irradiated from $0.5\text{-}1.6 \times 10^{21}$ fissions/cc of core.

Heating sections from the irradiated tubes showed that the threshold temperature for blistering depends on exposure and uranium content in the fuel tube as shown in Figure 9. No blistering occurred in any tube except at temperatures at least 175°C above the maximum operating temperature for U_3O_8-Al P/M fuel (~225°C). Blister thresholds for the P/M tubes ranged up to 600°C, within 60°C of the melting temperature of the cladding.

CONCLUSIONS

Experimental results thus far indicate that:

- cores containing fine (-325 mesh) U_3O_8 and aluminum powders can be made practically free of high-density areas using the outlined P/M preblending and sieving techniques.
- U_3O_8-Al cores can be isostatically compacted with up to 100 wt % U_3O_8 and tubes successfully extruded with up to 80 wt % oxide.
- fission gas blistering of U_3O_8-Al P/M tubes as indicated by the blister tests is a function of fissions/cc of U_3O_8 in the core. Decreasing the fission density of oxide increases the threshold temperature for blister formation.
- U_3O_8-Al P/M fuel tubes with up to 59 wt % U_3O_8 have been successfully irradiated in SRP reactor to 1.6×10^{21} fissions/cc of core or $\sim 7 \times 10^{20}$ fissions/cc of U_3O_8.
- small-scale metal forming tests sufficiently mock up production operations so that variables can be initially tested in the laboratory.
- parametric metal forming studies show a relationship between measurable variables of tooling temperature, half die angle and core composition. Lower temperatures and larger die angles (50-60°) favor extrusion of high-density fuel tubes.

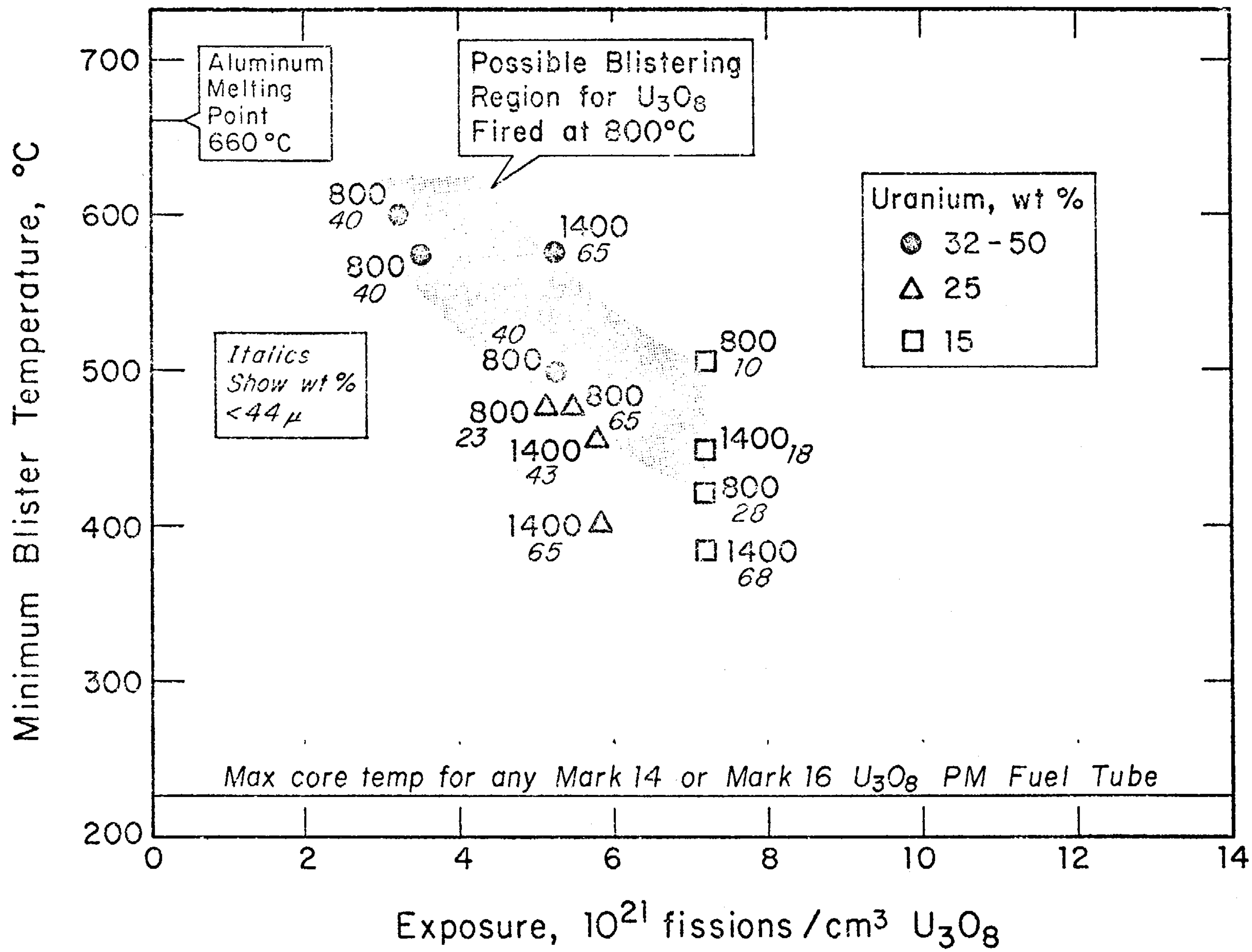

Fig. 9. Temperatures Required for Blister Formation on Irradiated Fuel Tubes with U_3O_8 P/M Cores ~40% Burnup.

DISCUSSION

BINFORD (ORNL): Have you done any thinking about the thermite reaction with those high loadings?

PEACOCK: Yes, we have. We have done considerable work with thermite reactions. We have looked at up to about 80 wt% oxide, and we have found that the reaction in the billet core is not a violent reaction. It is very similar to what the results found by Ivins and Tesla here at the Argonne Lab. They looked at it and said that it apparently was not very violent. Fleming and Johnson at Georgia Tech did some work where they said it was real violent. Our experience with the thermite reaction is that it will occur somewhere in the neighborhood of 930°C, and the most that we've seen happen is that it melts the billet down.

THRESH (ANL): This material of the higher oxide looks as if it's free from porosity. Would you make some comment on that?

PEACOCK: Our oxide is the low fired oxide, and it is not free from porosity. There is considerable porosity in the oxide itself and the low temperature does not sinter it to eliminate this. The density of our fuel tubes that we have measured in the extruded tube range from about 8 to 10% of porosity inside the tube. We also get some porosity from the drawing operation. Oxides will not withstand any low-temperature forces and, therefore, during the cold drawing operation we do have some additional fabrication voids that are introduced.

THRESH (ANL): How much cold work are you putting into these tubes? Is this just a sizing operation or a strengthening operation?

PEACOCK: Primarily a sizing operation. The drawing operation is about 10% and stretch strengthening is somewhere around 2-5%.

HICKEY (U.K. Atomic Energy): Have you tried any cold extrusions?

PEACOCK: No, we have not.

HICKEY (U.K. Atomic Energy): Do you preheat both the tubes and the billet?

PEACOCK: It is a hot extrusion operation where the billet is preheated and the tubing is preheated. (The results that I showed you on that slide; all of the billets were preheated to the same temperature.) Just a matter of difference in tubing temperature which is greatly affected by the heat transfer between those two and changes of flow stress and forces required to extrude the tube.

THRESH (ANL): What sort of structures do you see in your cast primarily UAl material. I assume at the higher uranium content you fight this problem of massive UAl_4 particles, presumably.

PEACOCK: Yes, we see the large particles, although there's been some work, although I cannot explain it, where they have established that given a certain critical cooling rate, they are able to get the peritectoid reaction in uranium/aluminum high weight percent system. Going through this they get bursting of the particles. They claim there is a refinement in the particle size within this range. I have not seen or done this, but that is the reported result, and they have, for the high weight percent castings, been able to get what you would expect. You would expect to see larger particle aluminides, but they are somewhat smaller than what I would expect. They still don't extrude very well because of the high volume percent of hard base.

THRESH (ANL): I was interested in this because we recently made fuel here at ANL by basically your process; the old NMI process. We basically have been vacuum melting this material, and we found that after the first recycle through the vacuum furnace, we were able to undercool that structure and primarily UAl_4 particles were completely supressed, you have a much softer type of material.

TRAVELLI (ANL): You mentioned there determining the limits of the highest loading of U_3O_8 that you can achieve. And also for irradiation tests, do you have any time scale for this program?

PEACOCK: The only time scale that we have right now is for the irradiation for 70 wt%. As far as to complete the thing, we would hope that we would be able to do significant inroads to this problem within 2-3 years.

TRAVELLI (ANL): When do you expect the irradiation tests may be completed?

PEACOCK: 70 wt% will go into the reactor. If everything goes smoothly and we're not bumped out by other programs, which happened sometimes, it's scheduled to go in the last of this year around November of 1979. It will be in there for a couple hundred days, and then it will come out and we will inspect it. We will do some blister testing. We have irradiated up to 60 wt% oxide, 59-60% oxide. What we're going to do is one increment higher at 70%. Someone asked the other day about bonding of the cladding to the core. As you increase the oxide content, you just don't get the bonding. The aluminum won't bond to oxide. In fact, we have problems whenever we do metallography. We have to use the vacuum impregnation because if you cut the tube and you're not careful, the loosely fitted oxide on the surface will be pulled out during the polishing operation. It bonds because of geometry until it's in the reactor. Once it's in the reactor, it does react with the aluminum and irradiation greatly accelerates this reaction. So if you have U_3O_8 in the aluminum and your fuel tubes, you put this in the reactor. We have seen no reaction prior to putting tubes in the reactor. But once they are in the reactor, they should form a reactive zone around those particles relatively fast. I don't know how long it will take, but once that happens, then everything is controlled by diffusion.

HICKEY (U.K. Atomic Energy): Following on from that point, have you in the post irradiation examination you have done any evidence of diffusion of the oxide through the cladding, or getting anywhere near the surface.

PEACOCK: No. Up to 60 wt% we have seen no reaction with the cladding, no degredation of the cladding in those areas. We are concerned about that. We've got some tests that are going on right now with extremely high wt%, up to about 80 wt%, but we are going to react for a period of time and outside the reactor you have to go to high temperatures for a long time to get a reaction between U_3O_8 and aluminum. It's probably something like 3,000 hrs at 400-500°C. We're going to accelerate this by going to high temperature for a long time.

MARTIN (ORNL): Concerning the bonding question. Does the meat portion of the extruded tube contribute needed strength for mechanical stability of your assemblies? Do you assume the meat portion contributes no strength and you want to rely on the cladding?

PEACOCK: The higher the weight percent, the less is contributed to the strength of the tube by the meat of the core. We have some data on the tensile strength of the tube because we are concerned about that, and as it approaches about 50%, the strength of the tube is greatly decreased. At the higher weight percent you're going to be very much dependent only on the aluminum cladding for your strength.

MARTIN (ORNL): The question concerns the design. In your calculations, do you consider you need any strength in the meat-bearing portion of the tubing?

PEACOCK: We think we have adequate strength with just the cladding for our purposes. One thing about these particular tubes -- we've looked at them under post irradiation examination and we've found that the oxide particles themselves retain the fission gases and basically act like little small chambers if you have aluminum surrounding the particles. Because if you look at these things you can see the voids, nice spherical voids, which could only be generated through gases under high pressures. As you decrease the amount of aluminum then you're going to need more voids, therefore, places for the fission gases.

CUNNINGHAM (ORNL): When you are into the component testing phase, do you have in your program the thermal cycling of the element that is normally done in the fuel program?

PEACOCK: The final components that go in the reactor are cycled. They do go up and down in the reactor.

CUNNINGHAM: (ORNL): It's in the reactor you cycle. You don't simulate it outside.

PEACOCK: No, we don't.