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SAFETY STUDIES OF THE SHEAR-LEACH PROCESSING OF ZIRCALOY-2-CLAD SPENT NUCLEAR FUELS

G. A. West
C. D. Watson

OAK RIDGE NATIONAL LABORATORY
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OCTOBER 1967

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FOREWORD

This study of the safety of the disassembly, shearing, and leaching of Zircaloy-2-clad nuclear fuels is one in a series of six reports relating to the development of a shear-leach head-end process for spent nuclear power reactor fuel reprocessing plants. The other reports are:

1. C. D. Watson, B. C. Finney, G. A. West, and B. A. Hannaford, The Shear-Leach Process for Nuclear Fuels, ORNL-3625.
2. B. C. Finney, B. A. Hannaford, G. A. West, and C. D. Watson, Shear-Leach Process: Semicontinuous and Batch Leaching of Sheared Unirradiated Stainless Steel and Zircaloy-2 Clad UO_2 and UO_2-ThO_2, ORNL-3984.
3. C. H. Odom, C. D. Watson, and B. C. Finney, Shear-Leach Process: Design and Development of Mechanical Components, ORNL-4062.
4. E. D. Arnold, Calculated Induced Activities in Nuclear Reactor Fuel Cladding Materials, ORNL-4065.
5. J. E. Strain, W. J. Ross, G. A. West, and J. W. Landry, The Design and Evaluation of a Delayed-Neutron Leached-Hull Monitor, ORNL-4135.

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SAFETY STUDIES OF THE SHEAR-LEACH PROCESSING OF ZIRCALOY-2-CLAD SPENT NUCLEAR FUELS

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ABSTRACT

A safety study of the handling of Zircaloy particles produced by the Shear-Leach processing of Zircaloy-clad nuclear fuels was conducted by means of a literature search and some abrasive disc sawing, shearing, and ignition tests. It was concluded that Shear-Leach processing can be accomplished safely, providing the appropriate precautions are followed.

The literature survey showed: (1) Dry zirconium powder is ignitable at room temperature by static electricity or simple friction and explodes spontaneously when dispersed into the air as a cloud.[1] With a moisture content of 16% or less, it has on a few occasions exploded spontaneously while under vacuum.[2] (2) Violent explosions, which have occurred and produced fatal injuries, involved powders finer than 62 μ, or finer than 230 mesh size.[3] When the surface area per gram approaches 0.1 m^2, caution must be exercised in how the powder is handled.[4] Particles finer than 20 mesh (800 μ) and coarser than 120 mesh (125 μ) should be collected wet (under water) and mixed with an equal volume of sand or grinding wheel grit.[3]

Tests showed that sawing can be done safely, provided large quantities (more than 1 to 2 kg) of the particles are not allowed to accumulate in the system. The Zircaloy-2 particles diluted with abrasive

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[1]National Safety Council data sheet No. 382 (Rev.) (1957).

[2]Zirconium Fire and Explosion Hazard Evaluation — Interim Report, TID-5365 (Aug. 7, 1956).

[3]W. W. Allison, Zirconium, Zircaloy, and Hafnium Safe Practice Guide for Shipping, Storing, Handling, Processing, and Scrap Disposal, WAPD-TM-17 (Rev.) (December 1960).

[4]W. R. DeHollander, An Evaluation of the Zirconium Hazard, HW-44989 (Aug. 15, 1956).

particles from wet sawing (the preferred method) at a minimum of three parts by volume is considered safe to handle and store.

Particles produced by shearing are not converted to the oxide. Those less than 149 μ can be ignited by a Tesla coil. Particles resulting from shearing should be settled out and collected, following the leaching step, and chemically dissolved.

1. INTRODUCTION

The use of zirconium (and its alloys) for reactor fuel element fabrication is popular in the nuclear industry because of its very low thermal neutron capture cross section (0.18 barn), excellent mechanical and fabrication properties, exceptional corrosion resistance, and strength at high temperatures. Zirconium metal is pyrophoric, and its use presents a safety hazard in the scrap (small particles) produced in the fabrication and reprocessing of nuclear fuels. These particles are usually easily ignited by sparks or small flames, and the fine powder can react with explosive violence by autoignition due to static charges.

The safety aspect of handling Zircaloy-2 particles resulting from the mechanical head-end process in remote reprocessing of spent reactor fuels in a heavily shielded cell was studied by sawing and shearing tests and a literature survey. The head-end process consists of abrasive sawing to remove the surplus inert end fittings of an assembly, followed by shearing of the chopped tubular array of fuel rods into short (~1 in.) lengths for acid leaching of the core material.

The abrasive sawing tests were conducted by sawing 3/8-in.-OD reactor-grade Zircaloy-2 rods with and without a water spray on the material and abrasive blade, followed by sizing, chemical analysis, and spark testing. The Zircaloy-2 particles (from shearing) were produced by shearing a stacked 8 × 8 array of unsecured Zircaloy-2-clad reactor-grade fuel tubes (0.4395 in. OD × 0.032 in. wall with a 0.025-in. annular UO_2 pellet clearance) into 1-in. lengths in the ORNL 250-ton shear.

2. ABRASIVE SAWING TESTS

The safety of wet and dry abrasive sawing of zirconium metal was investigated by sawing annealed, 3/8-in.-diam Zircaloy-2 rods. The dry

abrasive sawing created a voluminous amount of sparks and dense smoke, but this was virtually eliminated in the wet sawing operation. The Zircaloy-2 particles diluted with the abrasive particles from either the wet or dry sawing could not be ignited by a 20,000-v spark. See Sect. 4.

It was concluded that the wet abrasive sawing of Zircaloy-2 would be less dangerous than the dry sawing operation, since the extensive sparking in the latter constitutes a potential fire hazard.

A Ty-Sa-Man Machine Company abrasive saw used in the tests was built to accommodate a 16-in.-OD abrasive disc blade which is belt driven by a 7.5-hp, 1750-rpm motor at a spindle ratio of 1:1.47 (Fig. 1). The Zircaloy-2 rods met ASTM B-353-60T specifications, with analyses of: 1.53% Sn, 15.5 ppm H, 68 ppm Hf, 1155 ppm O, and a hardness of 100 Rockwell B. The abrasive blade used was a medium-hard aluminum oxide abrasive (Manhattan No. 225, 1/8 in. thick × 16 in. OD) rotated at 10,450 SFPM, average, and feed rates of 3 and 6 in./min for wet and dry cutting respectively, A fire-resistant plastic shroud around the abrasive saw contained the sawed particles, which were collected by a vacuum system.

2.1 Dry Sawing

Abrasive sawing of the Zircaloy-2 rods produced a stream of high-brilliance sparks and dense smoke while removing 2.0 cm^3 of Zircaloy-2 metal per minute. This sparking could be hazardous if low-ignition-temperature materials are present. Neither the parent metal nor the uranium dioxide pellets could be ignited by the sparking. Tests show that sparking is advantageous because most of the ignitable Zircaloy-2 fines are converted to zirconium dioxide, which is much safer to handle and store. In addition to conversion to ZrO_2, the zirconium particles are diluted with the abrasive particles at a ratio of 1 to 7.3 by volume. There are indications that this dilution increases the safety factor in preventing spontaneous or static spark reactions of the zirconium particles.[3] It has also been demonstrated that Zircaloy-2 particles diluted with sand, 1 to 1, would not explode.[3]

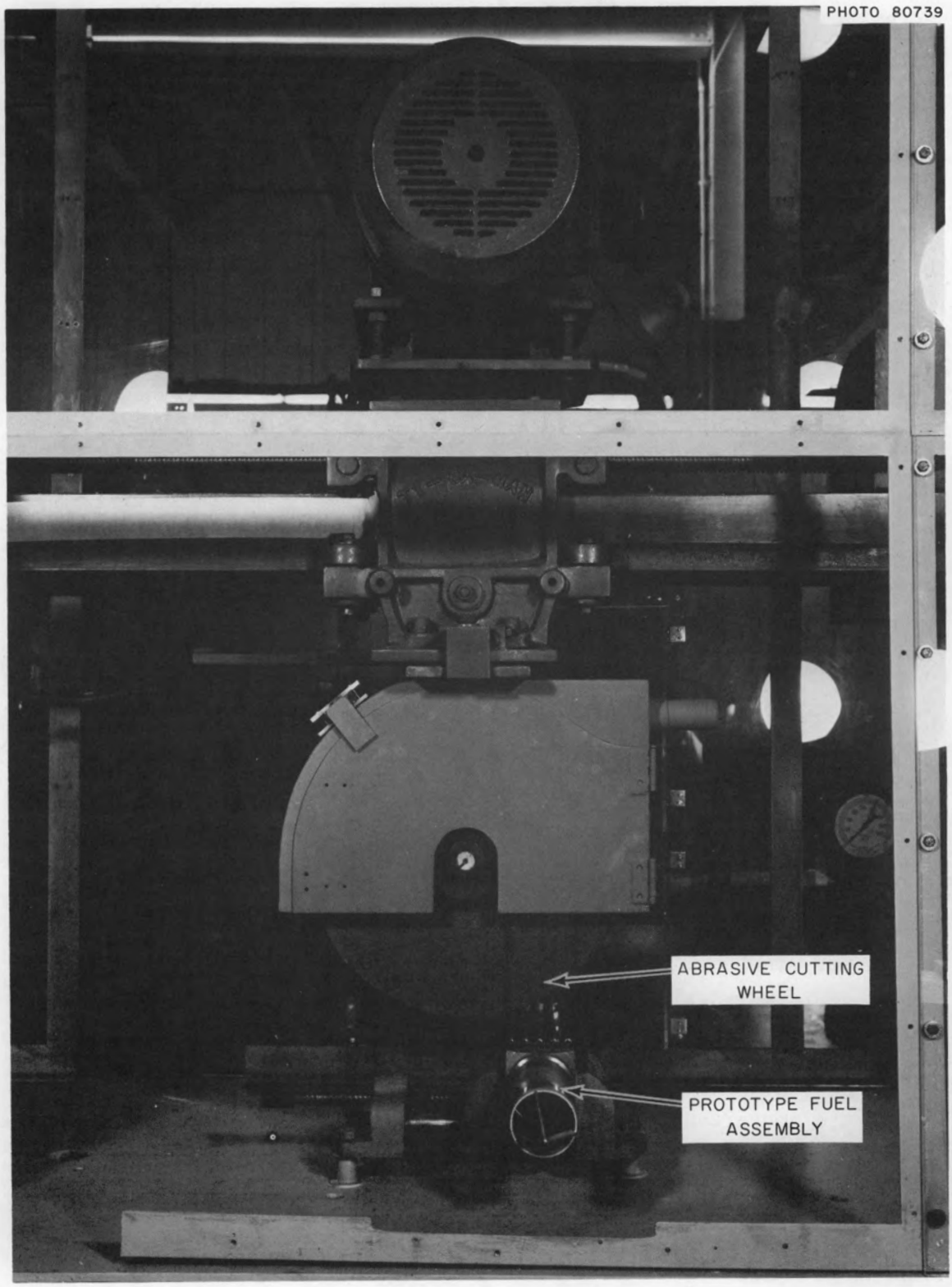

Fig. 1. Abrasive Disc Saw.

The zirconium and abrasive particles were collected and then classified by U.S. Standard shaker sieves. Metallurgical examination[5] of these particles revealed that the most dangerous (pyrophoric) size range (<149 μ, 64% of the Zircaloy-2 particles collected) was eliminated during the dry sawing operation. Observation through a stereomicroscope at 20 to 120× revealed that most of the metal particles that could be identified as Zircaloy-2 were coated with a white-to-gray oxide scale, and the proportion increased with decreasing screen size. The results of analyses to determine the amount of each material in the various mesh sizes collected after the dry sawing test are as follows:

Screen Opening		Particles Less than Size Indicated (wt %)[a]	
Microns	Mesh No.	Zircaloy-2	Abrasive
−2000	−10	91.1	99.1
−840	−20	87.1	98.2
−420	−40	78.8	54.0
−250	−60	71.4	31.5
−177	−80	67.2	25.3
−149	−100	63.7	21.8
−74	−200	49.1	14.1
−44	−325	30.1	9.4

[a]Weight percent based on total weight of Zircaloy-2 or abrasive particles collected.

2.2 Wet Sawing

The wet sawing test was conducted by directing two water spray nozzles on either side of the 16-in.-OD abrasive blade and a third nozzle on the cutting edge. The water spray virtually eliminated any fire hazard that could result from the sparks created by the abrasive cutting; however, the cutting rate was reduced about 50% because of cooling effects on the metal and the drag of the water spray on the blade. The maximum speed at which the blade cut the Zircaloy-2 rods without stalling the 7.5-hp motor was 3 in./minute.

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[5]M. L. Picklesimer, Metals and Ceramics Division, Oak Ridge National Laboratory. Now Director of Metallurgy, Southern Research Institute, Birmingham, Ala.

The wet Zircaloy-2 and abrasive particles were collected, dried, and sized through U.S. Standard vibratory screens with openings from 2000 to 44 μ. Analyses of the particles collected show that 100% more Zircaloy-2 particles are retained from the wet sawing than dry sawing (because of the burning during the dry sawing) and that 78% of these are less than 150 μ in size. The dilution of the Zircaloy-2 particles with abrasive was 1 to 6.6 by volume, making them less likely to ignite. Chemical analysis had indicated that 7% of the total Zircaloy-2 particles were from 250 to 2000 μ. However, observations at 120× magnification showed that most of the Zircaloy-2 in this range was in the form of small particles clinging to the larger ones; only an occasional particle was observed in the 2000-μ range. The results of the chemical analysis of each fraction measured are as follows:

Screen Opening		Particles Less than Size Indicated (wt %)[a]	
Microns	Mesh No.	Zircaloy-2	Abrasive
−2000	−10	99.5	94.3
−840	−20	98.7	84.3
−420	−40	96.2	74.6
−250	−60	92.2	68.4
−177	−80	85.5	61.7
−149	−100	75.4	52.2
−88	−170	64.0	47.6
−74	−200	58.6	44.7
−44	−325	37.5	27.6

[a]Weight percent based on total weight of Zircaloy-2 or abrasive collected.

3. SHEARING OF ZIRCALOY-2-CLAD UO_2 UNIRRADIATED PROTOTYPE FUEL

A Shear-Leach head-end method* for reprocessing spent power reactor fuels involves disassembly of fuel assemblies by sawing, followed by shearing of the disassembled fuel rods into short lengths. The fuel is leached from the sheared rods and reclaimed by solvent extraction.

*See 1. of Foreword.

Since contemporary power reactors will use Zircaloy-4-clad fuels which will require reprocessing, a study was made to determine the amount and size of the Zircaloy-2 particles (used as a substitute for Zircaloy-4) produced in shearing the fuel rods into 1-in. lengths, and the probable hazard of handling the particles. The ORNL 250-ton shear (Fig. 2) was used to shear a stacked 8 × 8 array of unsecured Zircaloy-2-clad fuel tubes, 0.4395 in. OD × 0.032 in. wall with a 0.025-in. annular UO_2 pellet clearance.

The shearing created one to three sparks per shear cut through the 64 stacked Zircaloy-2-clad UO_2 fuel tubes. This sparking appeared to be of low energy and was not considered a hazard in igniting the parent cladding or UO_2 fuel. The sheared pieces were collected and sized by vibrating them through U.S. Standard type sieves of screen sizes from 4760 to 44 μ. Chemical analysis of these various screened fractions showed a negligible amount (~0.1%) of Zircaloy-2 in the powdery fines, less than 400 μ (Fig. 3). The total amount of Zircaloy-2 dislodged was only 2.1% of the total cladding that was sheared. Analysis of the Zircaloy-2 particles by a Phillips electron probe analyzer did not reveal any oxidation resulting from the shearing operation. The presence of oxide on the particles would be indicative of burning during shearing, which would probably necessitate measures to prevent ignition of the parent cladding or any uranium metal present. The analytical results show that (1) shearing of Zircaloy-2-clad UO_2 fuel tubes is safe and (2) the Zircaloy-2

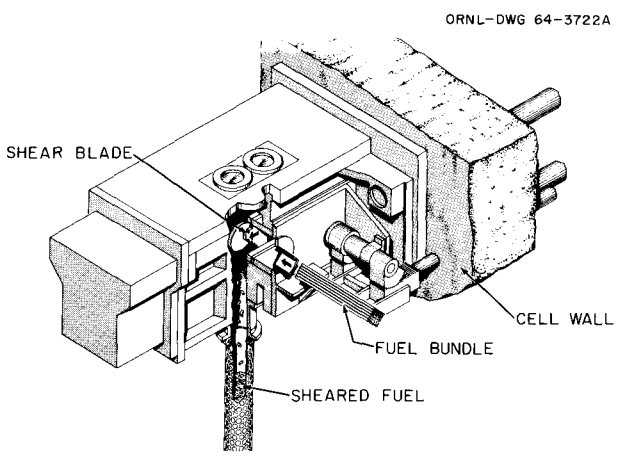

Fig. 2. ORNL 250-Ton Shear.

particles must be periodically collected and destroyed by either burning or dissolving in an HNO_3-HF solution to prevent the accumulation of a mass of particles capable of combustion under the proper conditions.

Calculations based on the particle sizing data (Fig. 3) indicate that the amount of Zircaloy-2 fines that would be accumulated in process-

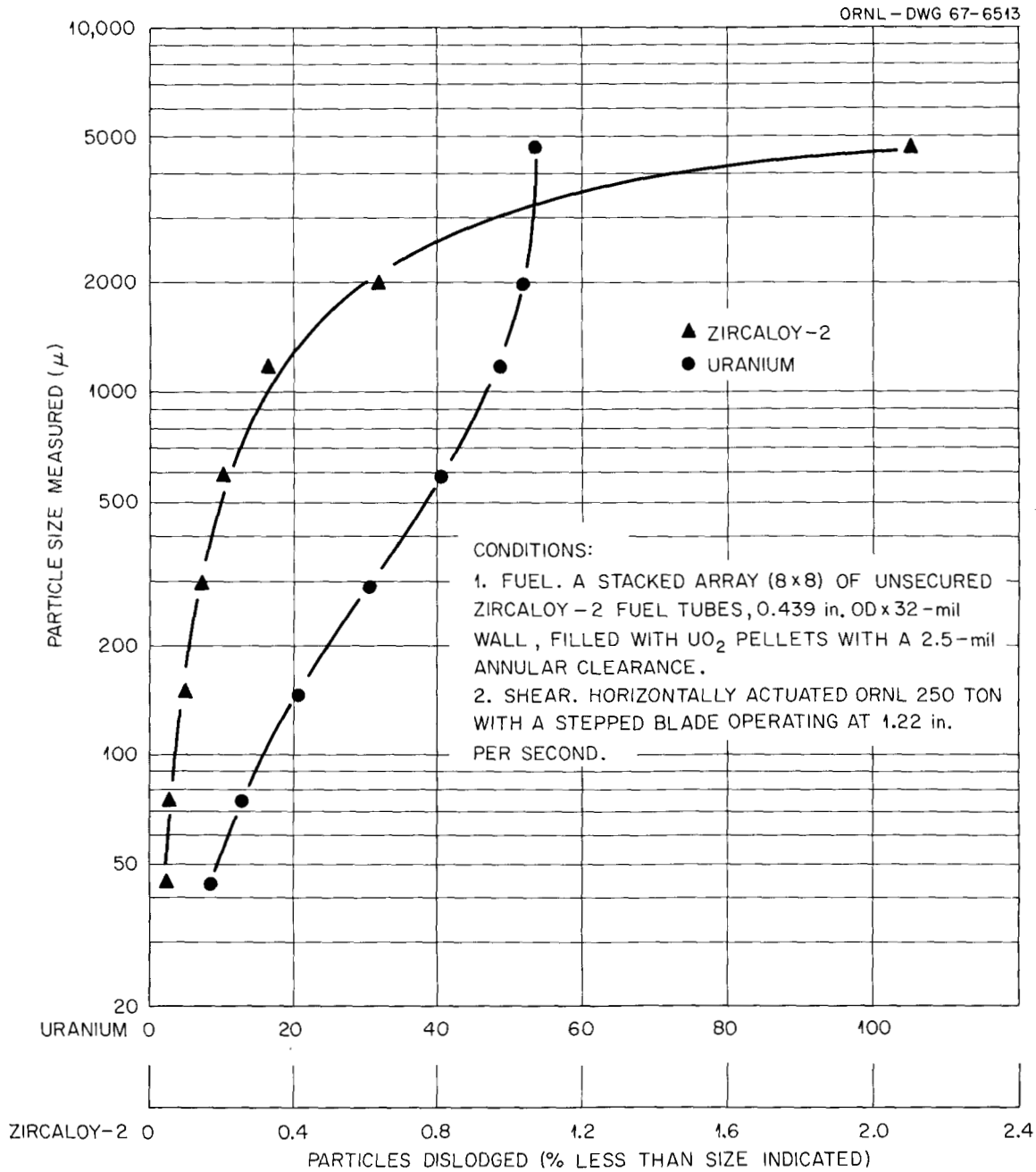

Fig. 3. Size Distribution of Zircaloy-2 and Uranium Particles Dislodged from Shearing Zircaloy-2-Clad UO_2 Prototype Fuel Rods into 1-in. Lengths.

ing 1 metric ton of uranium (1400 kg of sheared Zircaloy-2-clad UO_2, 19% cladding) would be as follows:

Particle Size (μ)	Zircaloy-2 Particles	
	Kilograms	Liters
−4000	3.990	0.623
−3000	1.530	0.239
−2000	1.410	0.220
−1000	0.745	0.116
−590	0.532	0.083
−297	0.372	0.058
−149	0.266	0.042
−44	0.106	0.017

In a typical leaching process, perforated baskets (1/8-in.-diam holes, about 5% free area) contain the sheared pieces of spent fuel. An analysis of the particle data indicates that the amount of Zircaloy-2 fines (< 3175 μ) which could possibly escape from the perforated basket and settle to the bottom of the dissolver would be 2.39 kg when processing 1 metric ton of uranium. These particles remaining in the dissolver after the product is decanted should be destroyed in a dissolution step using a boiling nitric acid–fluoride solution. This technique prevents an accumulation of fines and the possibility of an explosion.

4. SPARK IGNITION TESTS

The ignition tests of the particles from shearing and sawing Zircaloy-2 were conducted by subjecting the various size particles to an electrical spark. The electrical spark was generated by a high-frequency Tesla coil probe (model BD No. 10, Electro Technic Products) operated at 0.35 amp and 20,000 v, maximum.

A small portion of each fraction of Zircaloy-2 collected from the shearing of Zircaloy-2-clad UO_2 fuel tubes was placed in a 1.5-in.-diam flask; the material was then subjected to the spark. The UO_2 was dissolved away from the Zircaloy-2 fines with boiling 6 M HNO_3. The particles were aged in air for 276 days before dissolving away the UO_2, and an additional 45 days before the spark test. The 20,000-v electrical spark did not ignite particles that were >590 μ in size; the finer

screen fractions (149, 74, and less than 44 μ) were readily ignited by the sparks from the test probe. This spark test shows that the Zircaloy-2 fines can be hazardous and should be collected after each leaching run and destroyed by controlled burning or dissolution in an HNO_3-fluoride solution.

The Zircaloy-2 particles diluted with the abrasive grit at a ratio of 1 part to 6.6 and 7.3 parts of abrasive grit from the wet and dry abrasive sawing, respectively, were not ignited by the electrical spark. Tests were not made with the abrasive grit removed.

The results of the electrical spark test of the various screened fractions were as follows:

Screen Size		Results of Zircaloy-2 Produced from:	
Mesh No.	Microns	Sawing[a]	Shearing
10/16	2000/1190	No reaction	No reaction
16/30	1190/590	No reaction	No reaction
30/50	590/297	No reaction	One flash[b]
50/100	297/149	No reaction	One flash[b]
100/200	149/74	No reaction	Brilliant flash
200/325	74/44	No reaction	Brilliant flash (more pronounced)
−325	−44	No reaction	Brilliant flash (instantaneous and more pronounced)

[a]Zircaloy-2 particles diluted with the abrasive grit.

[b]Apparently, ignition involved a smaller particle clinging to larger particles of this fraction.

5. CONCLUSIONS

As a result of the study of Zircaloy-2, it is concluded that the sawing and shearing of this metal can be done safely, provided large quantities (more than 1 to 2 kg) of the particles are not allowed to accumulate in the system. Periodic removal of the fines is necessary

to prevent a mass that would be capable of combustion under the proper conditions.

Wet abrasive sawing of the Zircaloy-2 is more desirable for controlling sparking that could result in an immediate or potential fire hazard.

The Zircaloy-2 particles diluted with abrasive particles from the sawing operation at a minimum of one to three parts by volume are considered safe to handle and store for short periods until proper disposal.

Sheared particles less than 149 μ can be ignited, as was demonstrated with electrical sparks from a Tesla coil. The particles diluted with abrasive from both wet and dry sawing could not be ignited. This demonstrates the additional safety feature that results from mixing these particles with inerts such as sand or grit torn from a cutting wheel.

Some additional conclusions and ovservations from published reports, of related studies of zirconium, are as follows:

1. The dry powder is ignitable at room temperature by static electricity or simple friction. When dispersed into the air as a cloud, it explodes spontaneously.[1]
2. Powders containing 16% or less moisture are considered particularly hazardous and have, on a few occasions, exploded spontaneously while under vacuum.[2]
3. The surface area of powders in square meters per gram is within 10% of the reciprocal of the average particle size in microns. When the surface area per gram approaches 0.1 m^2, caution must be exercised in how the powder is handled.[4]
4. The chance of igniting zirconium stored under water is less than for dry storage. But in the event that wet scrap is involved in an external fire of small level, an extremely violent explosion approaching a low-order detonation with shock fronts moving at 1000 to 2000 m/sec is a distinct and real possibility.[4]
5. Massive irradiated zirconium may be disposed of, as are other radioactive wastes, without special treatment. Irradiated zirconium, in other than its massive form, should be converted as soon as practicable to a chemical compound not capable of burning or exploding, for disposal as radioactive waste.[6] It may be converted to the oxide by burning, or to a salt such as the fluoride or chloride, or other innocuous chemical forms.

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[6]G. H. Bulmer and A. Quinton, Recommendation on the Safe Handling of Zirconium Metal and Zirconium Alloys, AHSB(s) R-59, United Kingdom AEA (1963).

6. The addition of a fluoride salt, such as ammonium fluoride, to processing solutions will decrease the danger of explosion.[3]

7. The violent explosions which have occurred and resulted in fatal injuries have all involved powders finer than 62 μ, or finer than 230 mesh size.[3]

8. Scrap material finer than 20 mesh (800 μ) and coarser than 120 mesh (125 μ) should be collected wet (under water) and mixed half and half with sand or grinding wheel grit.[3]

9. Zirconium residues which have been stored for a fairly long period are the most hazardous to handle, particularly if they are in a moist state (3 to 16% water content). Prompt disposal of residues is, therefore, advisable.[7]

10. Test of crystal bar Zircaloy-2 irradiated for 540 Mwd showed that there was no appreciable amount of hydrogen pickup due to irradiation. Fast-neutron bombardment (8×10^{19} nvt) increased the electrical resistivity from 49.3 to 55.2 microhm centimeters. The temperature coefficient of resistivity was unaffected by irradiation. The microhardness of Zircaloy-2 irradiated to 15.2×10^{19} nvt increased from 135 to 173 DPH as measured at a 50-kg load.[8]

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We wish also to acknowledge and thank Martha Stewart, R. P. Metcalf, and J. L. Rich for their suggestions and editorial assistance.

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[7]F. B. Holt and S. G. Wilson, A Code of Practice for the Handling of Zirconium and Its Alloys, No. IGR-TD/S-578, United Kingdom AEA (July 1957).

[8]M. L. Bleiberg and L. S. Castleman, Effects of Neutron Bombardment Upon the Properties of Zirconium and of a Zirconium-Tin Alloy, WAPD-78 (Mar. 17, 1953).

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