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CONF-8709235--

Beryllium Science: US–UK Agreement on the Use of Atomic Energy for Mutual Defense

JOWOG 22/AVIS 568

Edited by James E. Hanafee

Papers presented at the US–UK Information Exchange,
Livermore, CA, September 22-23, 1987

February 19, 1988

Lawrence
Livermore
National
Laboratory

Contents

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Memorium

John White died on December 19, 1987. His professional ability and thoughtfulness for his fellow man was exemplified by the excellent science and social program he organized for the 1984 Beryllium JOWOG. John's wit, charm, and technical contributions will be long remembered. We all join with his AWRE colleagues in mourning his passing.

BERYLLIUM JOWOG 22B

Agenda

September 22, 1987 (Tuesday)	Bldg. 235 Gold Room	Page No.
8:30 - 9:00	COFFEE/Dinner Registration	
9:00 - 9:05	Welcoming Remarks, Dr. Richard G. Couch, Advanced Projects Office, Military Applications	
9:05 - 10:00	Round Table (DOE, DOD, UK only; Classified)	
10:00 - 10:15	BREAK	
10:15 - 11:30	"DOE Beryllium Supply Program", Richard Jiacoletti, DOE, Albuquerque	3
	"A Strategy for Retooling the Beryllium Industry," Hugh D. Hanes, Vice President, Brush Wellman	16
11:30 - 1:00	LUNCH	
1:00 - 3:00	"Review of AWRE Development Work on the Production of Near Net Shape Beryllium Preforms," D. Roberts, AWRE	27
	"Development of Beryllium Near-Net-Shape Technology at Brush Wellman Inc.," Monty Kuxhaus, BW.	59
	"The Development of a Compaction Diagram for the Hot Consolidation of Beryllium Powder, G. Heald, H. G. Rhodes, AWRE	68
	"Isostatic Pressing of Beryllium Powder into Near Net Shapes," J. L. Krankota, RFP.	77
	"Evaluation of the Strength of Graphite Dies Used in the Hot Pressing of Beryllium Billets," D. Roberts, AWRE.	98
3:00 - 3:15	BREAK	
3:15 - 5:00	"The Kinectics of Void Formation in Annealed Hot Isostatically Pressed Beryllium," J. Ansell, J. S. White, AWRE.	125
	"Beryllium Spin Forming at Rocky Flats," S. T. Abeln, M. P. Riendeau, RFP	137
	"Beryllium Films Formed by Physical Vapor Deposition," C. W. Nordin, R. O. Adams, RFP.	152
	"Application of Beryllium Vacuum Braze Technology to New Geometries," R. E. Priest, P. R. Landon, R. S. Juntz; LLNL (Classified - CRD).	
	"Speeds and Feeds for Carbide on Beryllium," D. Gallagher, RFP.	159
Evening	JOWOG Dinner (No-Host). Restaurant to be announced. Paid Reservations are due by Noon, Tuesday.	

September 23, 1987 (Wednesday) Bldg. 235 Gold Room

JOWOG 22B

Attendees 9/22/87

Abeln, S.	RFP
Borch, N.	LANL
Bowser, (?)	RFP
Brown, C.	RFP
Christiansen, S.	RFP
Cogbill, G.	ROF
Corle, R.	RFP
Gallagher, D.	RFP
Hanes, H.	BW
Jacobson, L.	LANL
Jensen, R.	RFP
Jiacoletti, R.	DOE
Koger, J.	Y-12
Kuxhaus, M.	BW
Lewis, T.	RFP
London, G.	NADC
Loewenstein, P.	NM
Mahin, K.	Sandia
Marder, J.	BW
Markham, T.	BW
Martin, A. J.	AWRE
Nordin, C.	RFP
Priest, R	LLNL
Roberts, D.	AWRE
Sasa, P.	RFP
Smugeresky, J.	Sandia
Stonehouse, J.	BW
Trimby, P.	ROF
White, J.	AWRE
Wilson, L.	RFP
Zenzac, S.	BW
Fulton, F.	LLNL
Juntz, R.	LLNL
Kautz, D.	LLNL
Landon, P.	LLNL
Lingenfelter, A.	LLNL
Musket, R.	LLNL
Nathans, M.	LLNL
Price, C.	LLNL
Syn, C.	LLNL
Wood, D.	LLNL

JOWOG 22B

BERYLLIUM

ABSTRACTS AND PRESENTATIONS

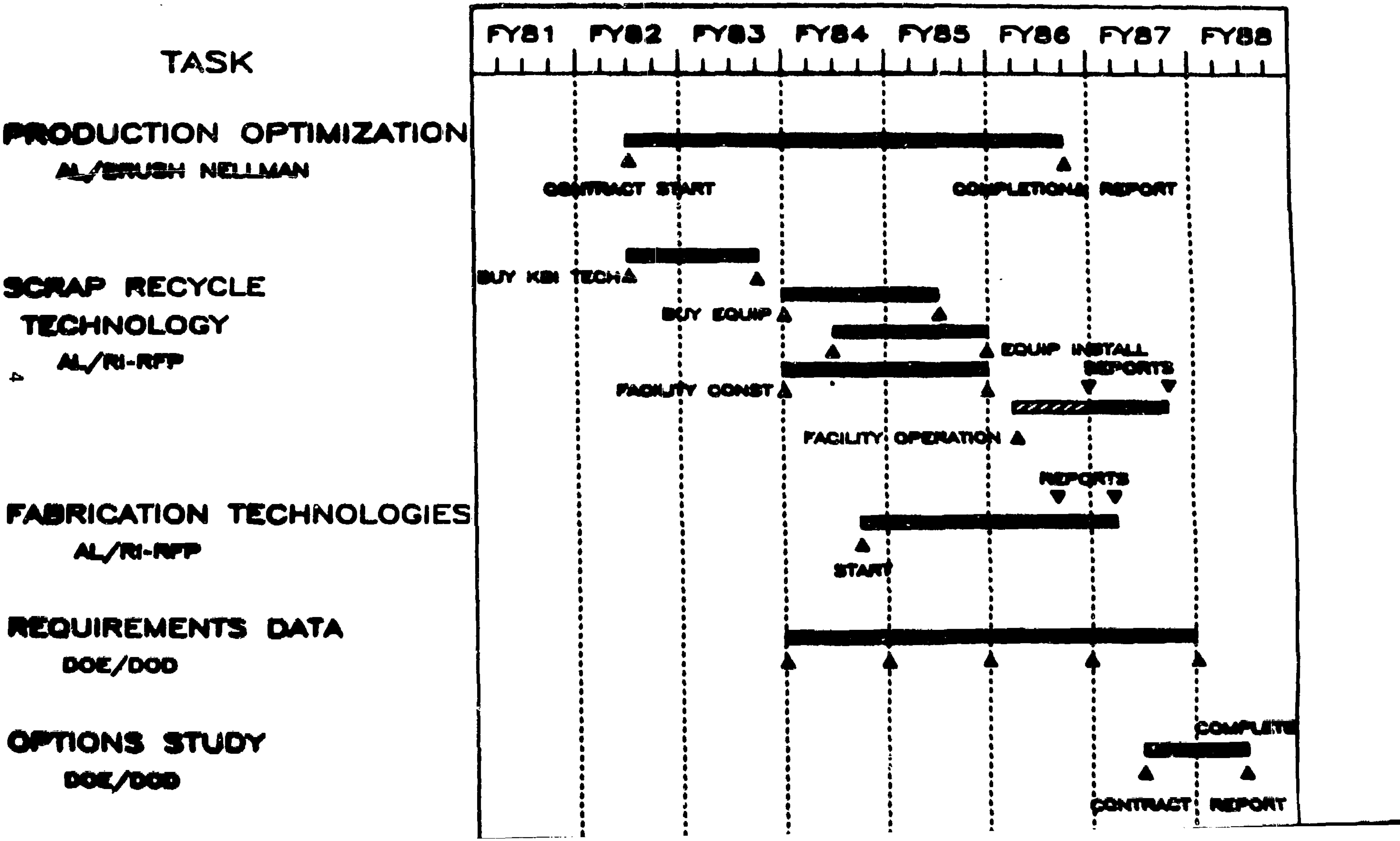
September 22, 1987

DOE Beryllium Supply Program

Richard Jiacoletti

DOE, Albuquerque

BERYLLIUM SUPPLY PROGRAM
MAJOR MILESTONES



BERYLLIUM SUPPLY PROGRAM

BERYLLIUM OPTIONS STUDY TASK

OBJECTIVE

THE EVALUATION OF DEVELOPED PRODUCTION OPTIONS TO ASSURE A CONTINUING SUPPLY OF BERYLLIUM METAL AND OXIDE

TASK AREAS

1. SELECTION OF STUDY CONTRACTOR AND CONTRACT AWARD
2. FINAL STUDY REPORT

AL/POD RJJ 3/87

BERYLLIUM SUPPLY PROGRAM

DOE FORECAST

BERYLLIUM METAL PURCHASES

FISCAL YEAR	1986	1987	1988	1989	1990	1991
COST (MILLION $)	13.7	12.9	14.6	16.6	18.8	16.3
WEIGHT (LBS.)	20449	16980	21018	22615	24732	21783

BASED ON AWLPG DATED OCTOBER 1986

BERYLLIUM SUPPLY PROGRAM

BERYLLIUM MATERIALS PLANNING TASK

OBJECTIVE

THE CREATION AND MAINTENANCE OF A BERYLLIUM DATA BASE FOR DOE/DOD PLANNING

TASK AREAS

1. DOD AND DOE PRODUCTION AND MULTI-YEAR PROJECTED REQUIREMENTS FOR BERYLLIUM METAL AND OXIDE
2. BERYLLIUM PRODUCTION SCRAP INVENTORY
3. NATIONAL STRATEGIC STOCKPILE INVENTORY RECOMMENDATIONS

AL/POD RJJ 3/87

BERYLLIUM SUPPLY PROGRAM

BERYLLIUM RECYCLE TECHNOLOGY DEVELOPMENT TASK

OBJECTIVE

ESTABLISH BERYLLIUM ELECTROREFINING (BER) TECHNOLOGY AT THE ROCKY FLATS PLANT TO EVALUATE THE FEASIBILITY OF PRODUCTION AND PRODUCTION SCRAP RECYCLE

TASK AREAS

1. DESIGN, INSTALLATION AND OPERATION OF A LABORATORY SCALE BER CELL

2. DESIGN, INSTALLATION AND OPERATION OF A HALF-SCALE PRODUCTION BER CELL

BERYLLIUM SUPPLY PROGRAM

ADVANCED PRODUCTION TECHNOLOGY DEVELOPMENT TASK

OBJECTIVE

DEVELOPMENT AND MAINTENANCE OF A BASE OF EXPERTISE IN BERYLLIUM PROCESS TECHNOLOGY AT THE ROCKY FLATS PLANT

TASK AREAS

1. POWDER PRODUCTION TECHNOLOGY
2. NEAR-NET-SHAPE TECHNOLOGY

AL/POD RJJ 3/87

BERYLLIUM SUPPLY PROGRAM

PRODUCTION OPTIMIZATION TASK

OBJECTIVE

COMPLIANCE WITH OSHA STANDARDS FOR AIRBORNE BERYLLIUM IN METAL PRODUCTION

TASK AREAS

1. OPTIMIZATION STUDIES OF CURRENT PRODUCTION PROCESSES & UNIT OPERATIONS
2. IDENTIFICATION AND EVALUATION OF ALTERNATE STATE-OF-THE-ART PROCESSES & UNIT OPERATION CONCEPTS

BERYLLIUM SUPPLY PROGRAM

TASKS

R&D

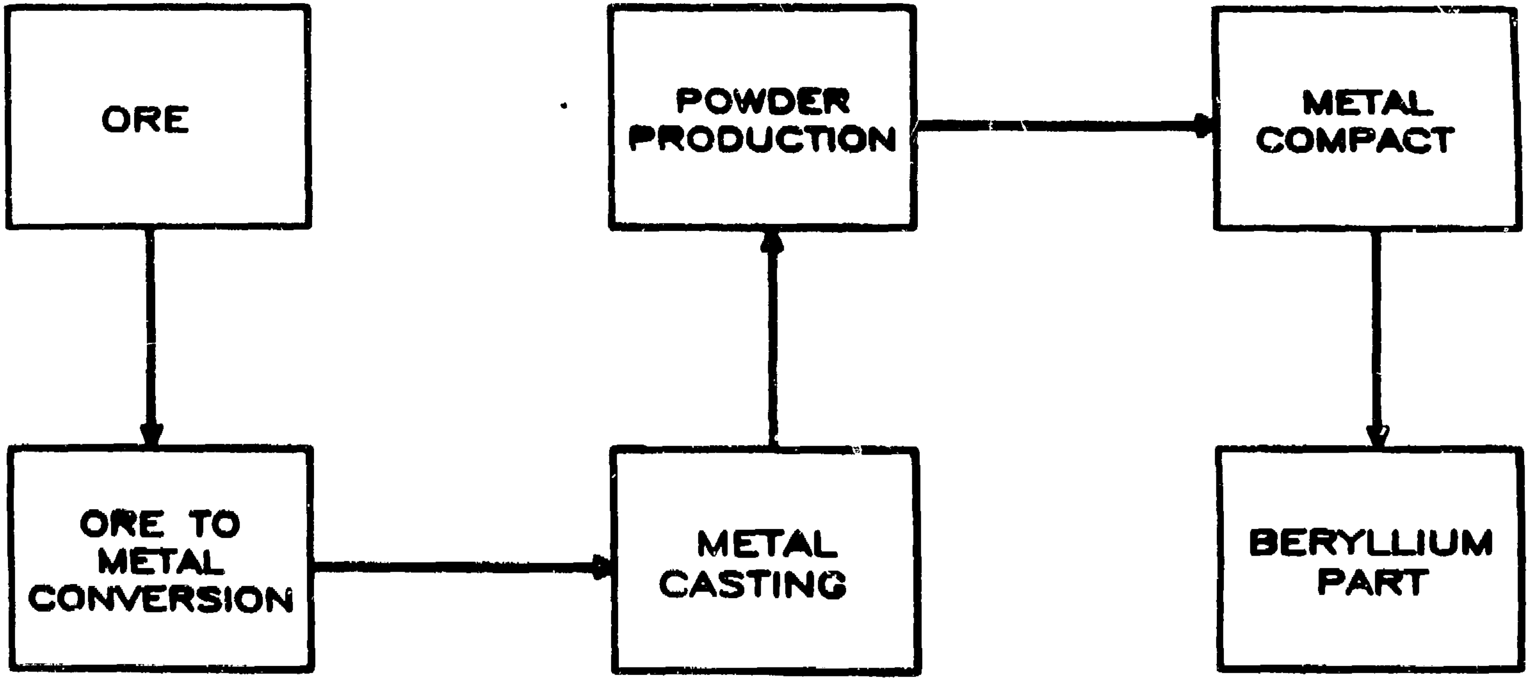
- PRODUCTION OPTIMIZATION
- AVANCED PRODUCTION TECHNOLOGY DEVELOPMENT
- BERYLLIUM RECYCLE TECHNOLOGY DEVELOPMENT
- BERYLLIUM MATERIALS PLANNING

ANALYSIS

- BERYLLIUM OPTIONS STUDY

AL/POD RJJ 3/87

BERYLLIUM SUPPLY PROGRAM
BERYLLIUM PRODUCTION SCHEMATIC



ORE

ORE TO METAL CONVERSION

METAL CASTING

POWDER PRODUCTION

METAL COMPACT

BERYLLIUM PART

BERYLLIUM SUPPLY PROGRAM

OBJECTIVE

TO ASSURE A CONTINUOUS SUPPLY OF BERYLLIUM METAL AND OXIDE TO SUPPORT WEAPONS PRODUCTION REQUIREMENTS

THE BERYLLIUM SUPPLY PROGRAM

IN SEPTEMBER OF 1980, THE DIRECTOR OF THE DIVISION OF OPERATION OF MA REQUESTED THAT AL "ACT AS PROGRAM MANAGER TO ESTABLISH AND COORDINATE ALL PHASES OF AN OVERALL STUDY OF THE WEAPONS PROGRAM REQUIREMENTS FOR BERYLLIUM METAL AND OXIDE AND TO DEVELOP AND EVALUATE PRACTICABLE ALTERNATIVES TO ASSURE AN ADEQAUTE SUPPLY OF BERYLLIUM IN SUPPORT OF THE WEAPONS PROGRAM". AS A RESULT, A PROGRAM TO MEET THESE OBJECTIVES WAS DEVELOPED AND IMPLEMENTED IN 1981.

GENERAL DEFINITION

A CRITICAL MATERIAL IS ANY MATERIAL, OTHER THAN NUCLEAR REQUIRED TO PRODUCE NUCLEAR WEAPON COMPONENTS AND HAS ONE OR MORE OF THE FOLLOWING CHARACTERISTICS:

A. LIMITED OR SOLE SOURCE OF QUALIFIED PRODUCTION MATERIAL

B. LIMITED OR NO DOMESTIC RESOURCES

C. NO IDENTIFIED SUBSTITUTE MATERIALS

D. LONG LEAD-TIME PROCUREMENT OF REQUIRED, QUALIFIED PRODUCTION QUANTITIES

E. EXISTING OR POTENTIAL REGULATORY CONSTRAINTS THAT INHIBIT OR COULD INHIBIT PRODUCTION OR USE OF THE MATERIAL

AL/POD RJJ 10/84

A STRATEGY FOR RETOOLING THE BERYLLIUM INDUSTRY

Hugh D. Hanes
Vice President

Brush Wellman Inc
South River Road
Elmore, Ohio 43416

Results from the DOE Options Study, continuing environmental pressures, as well as competition from alternative engineered materials have resulted in substantial internal investment by Brush Wellman in the beryllium business. The major emphasis is on near-net-shape processing for cost effective fabrication of high-quality beryllium components. However, major investments are also being made in our extraction technology as well as in facilities for recycling beryllium-containing scrap and wastes. This presentation will outline Brush's strategy for retooling the beryllium business.

Editor's Note: The visuals for this presentation are not shown here because of reproduction constraints.

Slide 1

Visual Logo: Brush Wellman.

Narration Our company, Brush Wellman, is the only fully integrated producer of beryllium products in the Free World. We're a strong, well-managed, publicly owned company with annual sales that are currently in the neighborhood of $240 to $250 million per year.

Slide 2

Visual Photograph: Beryllium alloys.

Narration Our products include copper-based and nickel-based beryllium alloys--engineering materials with properties superior to those of their base metals.

Slide 3

Visual Photograph: Beryllia ceramics.

Narration Beryllia ceramics--materials that are excellent electrical insulators yet conduct heat as well as many metals.

Slide 4

Visual Photograph: Metallic beryllium.

Narration And metallic beryllium--an engineering material with unique properties that make it essential for the strategic defense of the United States. Our alloy and ceramic products, too, have strategic applications.

Slide 5

Visual Logo: Beryllium/Mining Division Brush Wellman.

Narration Beryllium/Mining Division, which I head, supplied starting materials for the entire beryllium industry of the free world.

Slide 6

Visual Photograph: Hopper car at mine.

Narration We operate our own mine in the Topaz-Spoor Mountain region of Utah. The product of this mine is bertrandite--an ore that contains one-half to one and one-half percent beryllium oxide. We have proven reserves that assure an ample supply of ore for many decades. And we maintain pipelines to other sources of ore.

Slide 7

Visual Photograph: Delta Mill.

Narration We extract beryllium, in the form of beryllium hydroxide, from bertrandite and other ores at our Delta Mill in Utah. this facility has sufficient design capacity to keep up with the anticipated demand for beryllium through the 1990s. Beryllium hydroxide is the raw material for beryllium alloys, beryllia ceramics and metallic beryllium. We supply this material to competitors as well as to our own plants.

Slide 8

Visual Photograph: Aerial view of Elmore Plant.

Narration At our plant in Elmore, Ohio, we convert beryllium hydroxide concentrate from our Delta Mill into metallic beryllium and into high-purity beryllium oxide--the raw material for beryllia ceramics. Our Alloys Division which also operates a plant on this site, is the largest customer for the Delta Mill.

Slide 9

Visual Photograph: Beryllium part.

Narration The beryllium processing operations at Elmore are DOE's sole source of metallic beryllium--the optimum material for many strategic nuclear applications.

Slide 10

Visual DOE Concerns

- Continuity of supply?
- Is Brush Wellman using state-of-the-art technologies to protect the environment?
- Is Brush Wellman responsive to DOE needs and concerns?

Narration Understandably, DOE is concerned about relying on a single supplier. Can continuity of supply be maintained? Is Brush Wellman using state-of-the-art processing technologies that protect the working environment inside its plants and the public environment outside its plants? And is Brush Wellman responsive to DOE needs and concerns?

Slide 11

Visual 1981 DOE Options Study

- Determine best beryllium processing methods.
- Explore ways to improve the Elmore beryllium processing facility.

Narration Questions like these were addressed in a late, 1981 DOE options study. One objective of the study was to determine the best beryllium processing methods through literature surveys and selective pilot testing. Another objective was to explore ways to improve the existing Elmore beryllium processing facility, particularly from the standpoint of worker health and safety.

Slide 12

Visual Photograph: Worker in plant.

Narration Worker health and safety is, of course, a paramount consideration in the selection of processes for production of beryllium. As you know, some people are sensitive to beryllium dust or fumes and may develop berylliosis, a disabling lung condition if they inhale these materials.

Slide 13

Visual Photograph: Air quality being checked in plant.

Narration Air quality standards require that the level of beryllium in the plant atmosphere not exceed a daily weighted average--DWA--exposure of two micrograms of beryllium per cubic meter of air. We constantly monitor air quality in the plant to make sure conditions are safe. And, as you see here, our industrial hygienists drill our people in the use of respirators so they're ready for emergencies.

Slide 14

Visual Engineering Stud

- Air quality problems in 16 out of 19 plant areas (1981).
- 33 recommendations for improvements.

Narration As part of the DOE options study, we hired an outside engineering firm to study our operations. Initially, there were air quality problems in 16 out of 19 beryllium-processing areas in our plant. The firm made 33 recommendations for improvements.

Slide 15

Visual — Brush Wellman Response

- 26 of 33 changes implemented.
- 7 noncritical changes being considered.

Narration — We responded to these recommendations, of course. Twenty-six of the 33 recommended changes have been or are currently being implemented. Seven noncritical changes have not been implemented but are being considered as part of our long-range plant expansion and modernization program.

Slide 16

Visual — Air Quality Improvement

- Only three deviations from air quality standards in past 24 months.
- Intensified operator training will help prevent future incidents.

Narration — The result: There have been only three DWAs over two micrograms per cubic meter of air in the entire beryllium metal processing facility at Elmore during the past 24 months. At least one of the three deviations from air quality standards was caused by operator error and has resulted in an intensified operator training program to help prevent future incidents.

Slide 17

Visual — Photograph: Recycling area, powder cans visible.

Narration — Some of these changes were relatively easy to implement. One example is providing improved ventilation, particularly in areas where fumes are generated or beryllium powders are handled. This, incidentally, is the area where we clean up beryllium chips and scrap before recycling these materials back into our process stream.

Slide 18

Visual — Vacuum refining furnace.

Narration — Other changes required a considerable amount of engineering and major capital investment. For example, to control airborne contamination, we had to install a new charging system for our vacuum refining furnace. And we made major changes in ventilation covering the reduction furnaces to prevent pollution.

Slide 19

Visual Photograph: Beryllium billet being ultrasonically tested.

Narration As you know, engineering grades of beryllium are produced by compacting blended beryllium powders. This billet, made for the National Strategic Materials Stockpile Program, was produced by vacuum hot pressing.

Slide 20

Visual Photograph: Controls for vacuum hot-pressing operations.

Narration New controls, safer powder handling and die cleaning operations procedures, plus a $500,000 investment in a powder-drying system, have made substantial improvement in our hot-pressing operations.

Slide 21

Visual Photograph: Vacuum-refined ingot.

Narration We currently produce our beryllium powders by converting ingots of vacuum-refined beryllium like this into chips and then milling or grinding the chips into powders.

Slide 22

Visual Photograph: Powder-making facility.

Narration Improving air quality in our powder-making area was one of our greatest challenges. Breaks in the process of transferring powder from one container to another is a major source of air pollution. Producing and classifying I-4N powders can involve as many as 200 process breaks. We are eliminating these breaks by developing and installing a $750,000 closed-loop powder-handling system.

Slide 23

Visual Photograph: Welding operation--plant improvement program.

Narration Time does not allow us to describe all of the changes we've made in our plant in response to the 33 recommendations resulting from the 1981 DOE Options Study. As we developed and installed new equipment, scenes like this have been commonplace in our plant for several years.

Slide 24

Visual Cost of Improvements

- 1981-1986: $8 million
- 1987-1988: $2 million

Total: $10 million

Narration The cost of making these changes--fine-tuning processes and installing new equipment--was high--$8 million from 1981 through 1986. We'll spend an additional $1 million to complete the improvements during 1987 and 1988, for a grand total of $10 million. This capital investment illustrates Brush Wellman's positive response to DOE's concerns about environmental conditions in our metallic beryllium processing facility.

Slide 25

Visual Photograph: Plant stack.

Narration We're equally committed to maintaining high air quality outside our plant. Our stacks are equipped with state-of-the-art scrubbers and other pollution control systems. We're in full compliance with all applicable clean-air standards. Through an air-quality monitoring program, we make sure that public exposure to beryllium dust in the area around our plant is less than one one-hundredth of a microgram per month.

Slide 26

Visual Photograph: Alloy Division pickling tank.

Narration Toxic wastes such as this spent pickling liquor from beryllium-copper alloy processing operations at Elmore present a real environmental threat. Rightly, EPA is tightening regulations through RCRA to control the disposal of such wastes while protecting the environment.

Slide 27

Visual Photograph: Overall view of waste recycling plant.

Narration As part of our program to protect the environment, we recently opened a new $7 million waste recycling plant at Elmore. This plant is designed to recycle wastes for all Brush Wellman operating divisions and--in fact--eventually the entire beryllium industry. The plant currently extracts over 2 million pounds of copper and 30 thousand pounds of beryllium per year from spent pickling liquors. These valuable materials are cycled back into our operations.

Slide 28

Visual Photograph: Gas chromatograph.

Narration In order to control pollution, it's necessary to measure it accurately. During the past several years, we have invested heavily in advanced instruments. this computerized gas chromatograph, for example, detects minute traces of toxic contaminants in wastewaters automatically.

Slide 29

Visual Process Improvement

- Review existing beryllium processing technologies; make improvements.
- Survey literature for new beryllium processing technologies.
- Set up and test demonstration models of new processing technologies.

Narration The DOE options study called for three phases. So far, we've covered just the first phase--reviewing our existing beryllium processing methods and making improvements. The second phase involved searching the literature for new beryllium processing technologies. And the third phase required setting up and testing demonstration models of promising new technologies.

Slide 30

Visual Photograph: Beryllium pebbles.

Narration One area we studied intensively was the production of beryllium pebbles--an intermediate stage in the processing of our beryllium hydroxide concentrate into a high-quality engineering material.

Slide 31

Visual Photograph: Molten beryllium fluoride.

Narration We have always produced beryllium pebbles by the reduction of beryllium fluoride--the molten material you see here--with magnesium. The best alternative method we found in the literature was the electrowinning of beryllium from beryllium chloride. Our studies showed, however, that our present process is less costly and is less apt to cause pollution. So we decided to stay with our present process.

Slide 32

Visual Photograph: Attrition mill.

Narration When we reviewed powder-making processes, however, we decided to make some big changes. Historically, we have produced most of our powder by a process called attritioning. Two of our attritioning mills are seen here. Attritioning is a relatively slow, inefficient process.

Slide 33

Visual Photograph: Impact grinding control station.

Narration On the basis of successful pilot studies, we decided to convert to production of powder by a process called impact grinding. We recently completed this conversion and are supplying 90% of our structural products. The control station for the new process is seen here. Impact grinding is faster and cleaner than attritioning. And it produces blocky-shaped powder particles that give the consolidated material more uniform properties in all directions.

Slide 34

Visual Photograph: Molten vacuum-refined beryllium.

Narration Looking farther ahead, we may produce much of our powder directly from molten vacuum-refined beryllium, seen here, using a process called inert gas atomization. We've successfully demonstrated this process on a recently completed DOE study in our laboratory and allocated $330,000 to modify our laboratory system for further tests. Dr. Marder, our Research Supervisor, will discuss this process with you later.

Slide 35

Visual Photograph: NNS parts being inspected.

Narration We're much father down the road with another new process that was pilot-tested during the period of the DOE Options Study--forming parts to near-net-shape directly from beryllium powders. In fact, we're in the process of completely retooling our plant to use NNS technologies. Several of our NNS parts are seen here. Monty Kuxhaus, Sup't. of Special Products, will cover NNS processing technologies and their benefits to DOE and other customers. For the moment, I'll just note that tooling up for NNS represents a $7.5-$10 million capital investment by Brush Wellman.

Slide 36

Visual Photograph: NNS part.

Narration Someday, a substantial percentage of our beryllium production at Elmore will consist of NNS parts made from atomized powders. This will result in higher quality, better materials utilization, faster processing, and further protection for the environment because of reduced recycling. We'll continue to use conventional powder-making and powder-consolidation processes to meet long-standing requirements and provide reserve capacity for emergencies.

Slide 37

Visual Photograph: Worker taking sample in plant.

Narration So far, we've just talked about retooling our plant. We're retooling our work force, too. We're providing additional safety, technical, and management training for our people. Rewriting 270 SOP's - a 5-year program. We're starting to apply the management philosophies of Dr. Deming, which have worked so well in Japan. And we're filling professional job openings with new talent in areas of extractive metallurgy, powder processing, CIM, materials handling, etc.

Slide 38

Visual Photograph: Someone using an automatic carbon-sulfur analyzer.

Narration We've completely retooled our laboratories with automatic state-of-the -art instruments that help us maintain even higher product quality and give us the information we need to fine-tune our processes. As processes are optimized, we're starting to apply statistical quality control methods. Computer integration of our processing operations is being studied.

Slide 39

Visual Photograph: "Pond" at Delta Mill.

Narration As part of our R&D efforts, we're also developing and testing improved hydrometallurgical methods of extracting beryllium ore at our Delta Mill. We also continuously evaluate alternative beryllium ore sources.

Slide 40

Visual	Photograph: Computerized testing machine.
Narration	Our R&D people aren't neglecting new product development. They're currently developing and testing new grades of beryllium for optical and instrument applications. The tensile strength and elasticity of samples are determined automatically on this new computer-controlled machine.

Slide 41

Visual	None
Narration	You can be sure, too, that we will continue to meet DOE standards for service and quality. Recently, when I visited Rocky Flats and several other customers, program managers told me that they regard Brush Wellman as their best supplier in terms of meeting delivery schedules and maintaining consistently high product quality.

Slide 42

<u>Visual</u> <u>Capital Investment</u>

- Environmental protection (DOE study)--$10 million.
- New process technology (NNS)--$10 million.
- Waste recycling--$7 million.
- 5-year plan--$8-$10 million per year.

<u>Narration</u> The extent of our commitment to being an outstanding DOE supplier is indicated by our heavy capital investment of the past few years--$10 million for new process technology and $7 million for our waste recycling plant. Our most recent 5-year plan calls for additional capital investment at the rate of $8 to $10 million per year, plus R&D expenditures amounting to 3-3.5 percent of annual sales.

Slide 43

Visual	Photograph: Dr. Foos.
Narration	Dr. Raymond A Foos, our president and chief operating officer, promises this group in June, 1986, that Brush Wellman will continue to invest heavily in the beryllium business as long as we can make a reasonable return on that investment. That would be impossible if we had to share the DOE market with other suppliers. Dr. Foos adds that we'll strongly resist all efforts by the United States government to establish a second source with public funds. That's a promise, too. Any questions?

REVIEW OF AWRE DEVELOPMENT WORK ON THE PRODUCTION OF NEAR NET SHAPE BERYLLIUM PREFORMS

by

D.Roberts (AWRE)

Summary

Near net-shape beryllium shell preforms have been vacuum hot pressed at AWRE for a considerable time. However, the process has both its limitations and disadvantages. The work being carried out to improve the hot pressing process together with the development work being done on the isostatic pressing of near net-shape preforms, is described.

Review of current AWE development work in the production of beryllium preforms by powder metallurgy

SLIDE 1

The main component produced at AWRE and ROFC has been a thin hollow hemisphere with a thick equatorial band.

To meet required mechanical properties the original trial preforms were produced by vacuum or die hot pressing and the process became the approved process for production. Since then various process developments have been made to improve die hot pressing.

Since the process was adopted isopressing equipment have become available. For pressing to near net shape this process is ideal. However for use in the present production run the process and the preforms would have to be approved. The savings made by improved net shaping would not justify the cost of process approval.

The isopressing process however has been developed up to a pre-production stage so that in future two trial component will be produced, one by die and one by isopressing, allowing both routes to be assesed and approved and allowing a choice of routes for production

The following is a summary of the development work carried out on both routes.

SLIDE 2

Initially the various possible routes of producing the typical component were looked at and the weight of powder required for the present preform estimated

An attempt has been made at estimating the type of swarf produced by the various routs, and expressing the fraction recoverable as processable to a lower grade powder and as heavily contaminated not processable.

The analysis showed that both machine from solid, and, form from blank, would be heavy on powder usage. The vacuum die pressing would be fairly economic but the best process would be isopressing.

Dealing first with die or vacuum hot pressing

SLIDE 3

The process developed initially from straight billet pressing First

1 The pressing of solid hemispheres with a material saving of about 25%.

2 Next the pressing of hollow hemispheres was developed by pressing a graphite core into the powder during

pressing

3 Then vibrating of a core into the powder bed

4 Finally the present method where the core is pre positioned in the die

The slide shows the type of tooling used and the process steps of

DIE FILLING
REMOVE SPIDER FIT PLUNGER
TRANSFER TO FURNACE
EVACUATE FURNACE AND HOT PRESS
REMOVE ASSEMBLY FROM FURNACE AND EXTRACT PREFORM FROM DIE
MACHINE CORE FROM PREFORM.

The process has been in use for about 15 years . In use the main problems with the process were

SLIDE 4

POOR DIE LIFE
PROCESS OPERATOR DEPENDENT
PRODUCTION LIMITED TO ONE PERFORM PER DAY
POOR NET SHAPING POTENTIAL.

Dealing first with die material The original die material was a soft graphite CS grade . Later tools made from a finer grain high strength graphite were used. Experience showed that the CS graphite gave adequate service and had sufficient strength for the conventional pressing.

The dies made from the stronger graphite were susceptible to scabbing of the top surface during component extraction. By destroying the the top face of the die, the core register, and the lifting points were lost and a new die could be lost in the first pressing operation.

Work to be discussed later showed that the dies were working at a stress level much lower than that predicted by calculation. The need to use the stronger graphites has not occured though it is used exclusively for plungers.

CONSIDERING NEXT POOR NET SHAPE DEVELOPMENT POTENTIAL

SLIDE 5

In the die pressing process the graphite core is trapped in the preform by differential cooling stresses. To avoid these stresses cracking the preform, the preform wall have to be of a minimum thickness. The thickness requirement places a limit on the potential to develop of net shaping

A ready solution to this would be to use a stainless steel mandrel which would contract more than the preform and not allow wall stresses to develop.

The problem with a stainless steel mandrel was that at

1050 C the formation of intermetallic layer would destroy the mandrel. The use of mandrels plasma coated with alumina to avoid the reaction were unsuccessful.

More recent work using ERBIA coated mandrels

SLIDE 6

have been more encouraging with mandrel life of up to three pressing cycles and the pressing of thin wall preforms

SLIDE 7

even though press loads had to be doubled to attain 100% density in the preform

The next problem to be considered is

LIMITED DIE LIFE

SLIDE 8

At present about 20 pressings can be obtained before a die becomes so worn that preform removal becomes difficult and the die has to be scraped. Initially dies were damaged before the bore wear became important but various developments have reduced the instance of early failure.

These were

First improvement in vacuum reducing the rate of formation of crumbly graphite surface and its associated tendency to cause increase of inclusions in the preform. Futher reductions in inclusions are expected from the use of a precompacted CIPed powder fill.

SLIDE 9

SLIDE 10

The other improvements such as damage to bore damage to lifting tabs and scabbing of the top surface have been reduced by die design

SLIDE 11

THE MAJOR PROBLEM WITH THE EXISTING DIE PRESSING METHOD IS THE LIMIT OF ONE PREFORM PRE DAY USING THE DIE ASSEMBLY SHOWN

SLIDE 12

it has been possible to press two preforms in one pressing operation. The process stages are similar to the single process but the die is filled in two operations. The bottom cavity first through the centre. Then after plugging the hole the top cavity is filled as normal. The pressing is then conventional but with the die being double acting.

SLIDE 13

Problems have been encountered in filling the bottom cavity and the method has been chanced to using a pre compacted powder fill in the bottom cavity. The process was successful in pressing two preforms in one operation

SLIDE 14

The use of the precompacted charge is being considered for normal hot pressing hopefully to reduce the operator dependency of the process. The use of a CIPed preform would reduce the problem of die surface cleaning prior to transfering the filled die from from filling box to furnace and also reduce the problem of powder suck out during furnace pumpdown. Using a pre-CIPed charge will also reduce the possibility of graphite based inclusions in the pressing.

SLIDE 15

In producing the green preform the bag contraction was not as expected and the resultant preform did not fit the die contour. As a result during initial pressing movements the preform cracked but rehealed leaving the furrowed surface seen in the slide

SLIDE 16

Examination of sections of the preform show that the furrows are very shallow.

To show that the cracking was due to poor green preform fit the work is to be repeated with the hot pressing die made to fit the CIPed preform. For production use the CIP assembly would be tailored to produce a green preform to fit the die contour.

CHANGING TO ISOPRESSING

SLIDE 17

FOUR possible production routes were available

A	DIRECT HOT ISOPRESSING
B	FLUIDIC ISOPRESSING
C	CIP SINTER HIP
and	CIP HIP

experimental experience showed that the first three processes were not suitable

Direct HIP could give problems in final can welding and can shape optimisation would difficult

FLUIDIC could not be carried out as no suitable powder was available as second fluid

CIP SINTER HIP required excessive temperature to attain closed porosity at the sinter stage

THIS LEFT THE ONLY ROUTE

CIP HIP

Two variations of this route was possible

SLIDE 18

which is CIP HIP using an in-can mandrel

The problem in this route is the long in process time of the mandrel and the limited mandrel life due to intermetallic reaction between mandrel and preform

The process has the advantage that the presence of the mandrel ensures the inner form and protect the preform from distortion in event of can buckling

SLIDE 19

The pressing shown 200mm diameter was pressed using a uncoated stainless steel mandrel
The reaction was such that after three pressings the mandrel was damaged beyond reuse

SLIDE 20

Using erbia coating Mandrel life has been extended to three pressings before recoating becomes necessary.

SLIDE 21

The alternative process worked on has been HOT isopressing of a CIPed preform in a double can with preform fitting on the inner can only

Initially it was thought that the inner can would contract inwards and possibly buckle. To avoid this the

SLIDE 22

CIPed preform was densified to about 86% by CIP ANNEAL PROCESS. THe 200mm diameter preform showed that the inner can acted as a mandrel and no inward movement occurred. Work with 110mm diameter preforms

SLIDE 23

again showed that the inner can acted as a mandrel

The non inwards contraction of the inner form appears to be in contradiction to expected isopressing behaviour but

is IDEAL BEHAVIOUR FOR THE PRODUCTION OF HOLLOW PREFORMS as

it eliminates the need for in can preform and can accommodate the use of loose fitting outer can so eliminating the need for a CIP machining stage.

The reason for the non inward movement of the inner form is being investigated as it may be dependent on can thickness geometry and in other can designs could create problem.

CONCLUDING

SLIDE 24

The slide shows a cross section of a 110mm diameter 6---

8mm wall preform produced by double can CIP HIP process

SLIDE 25

compares a vacuum hotpressed preform in current production with an experimental substitute produced by CIP HIP the weight saving is shown

SLIDE 26

The final slide compares the cross section of 110m

preforms produced by

DIE PRESSING WITH A GRAPHITE CORE

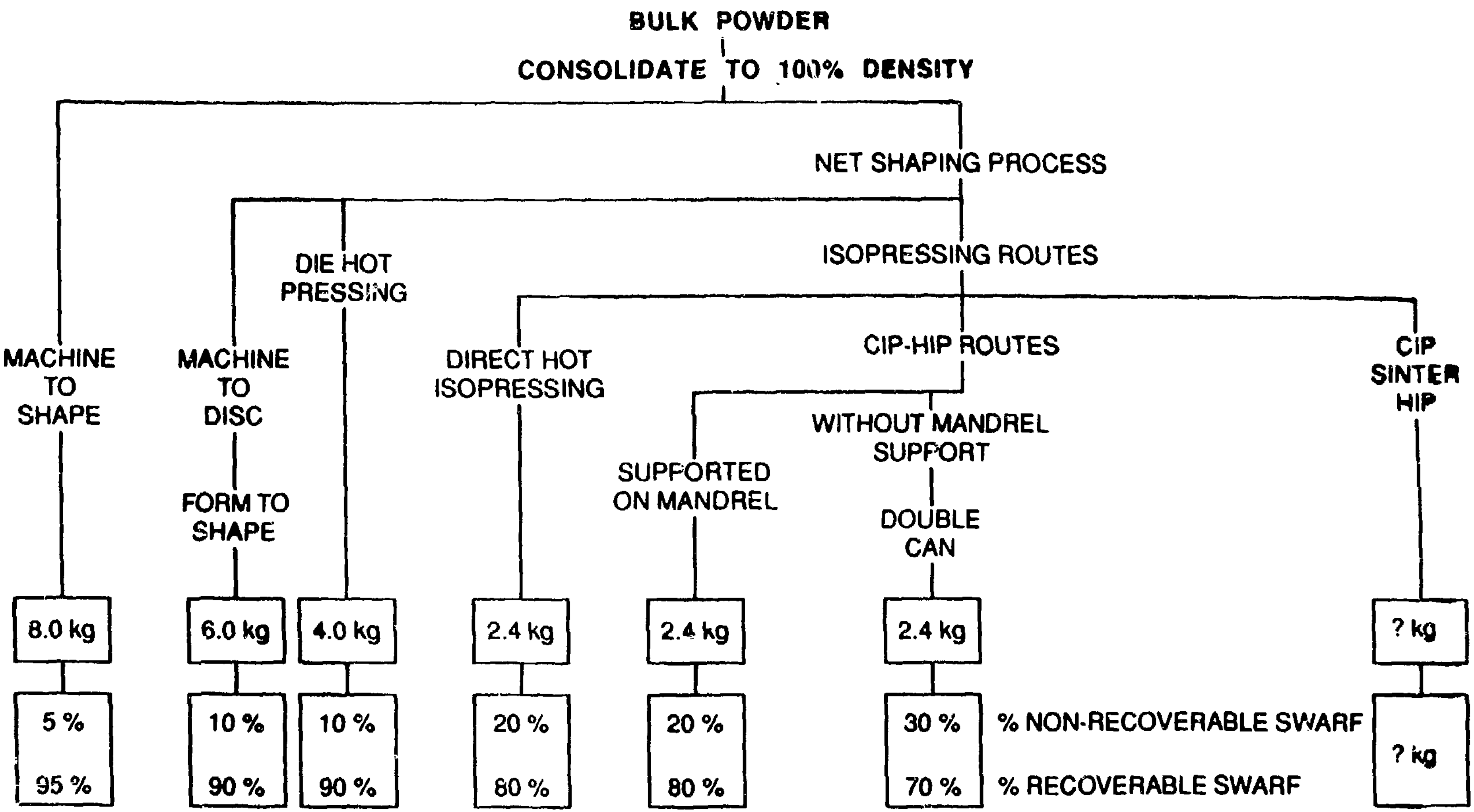
DIE PRESSING WITH A COATED CORE

ISOPRESSING WITH THE DOUBLE CAN TECHNIQUE.

AND SUMMERISES THE PRESSENT STAGE OF DEVELOPMENT

Rauk 2

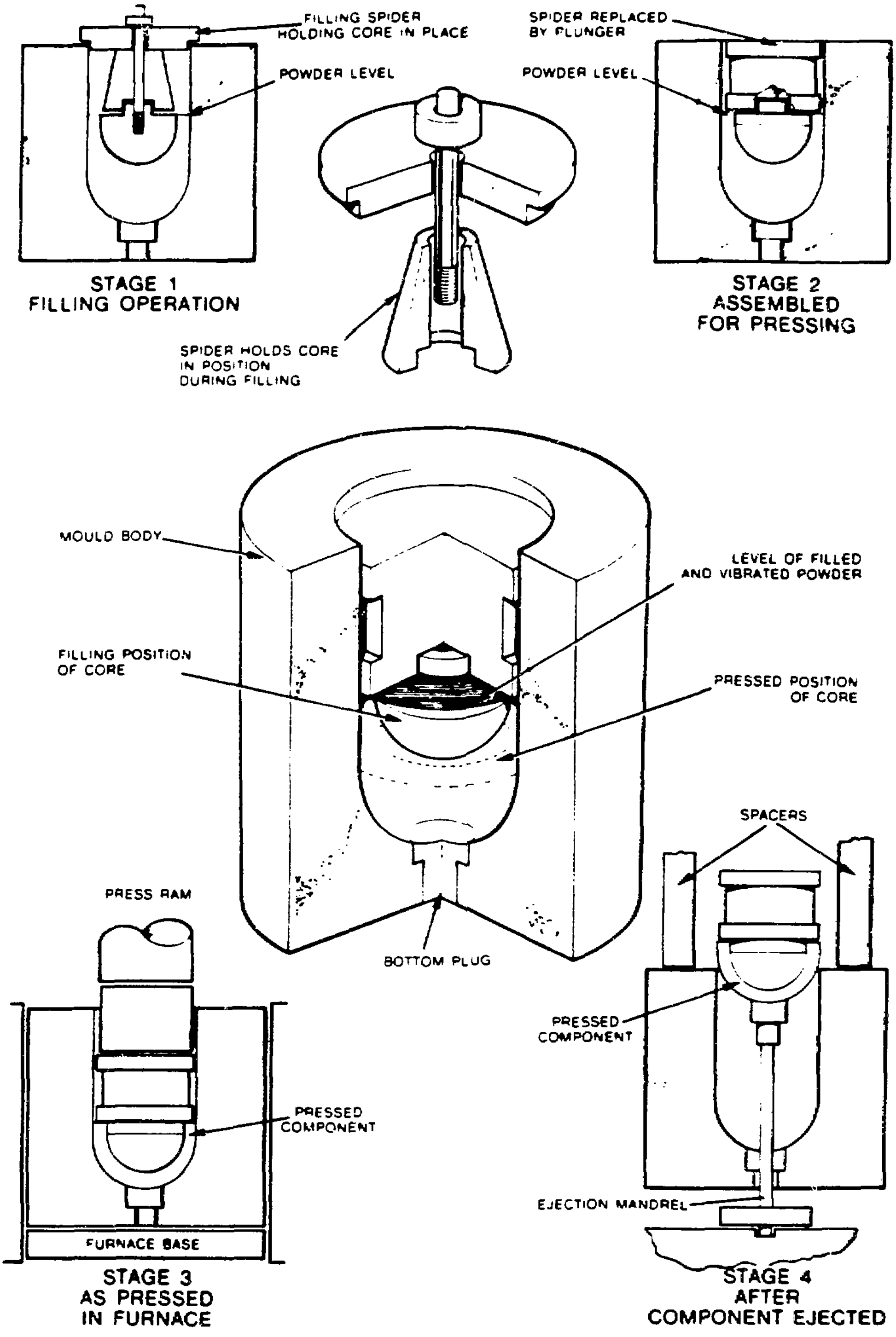
POWDER PREFORM PRODUCTION



FI(I. POSSIBLE POWDER PRODUCTION ROUTES FOR THE MANUFACTURE OF BERYLLIUM PREFORMS

The weights shown are estimates for same preform produced by various routes. Bottom line shows estimated distribution of swarf arisings between dirty swarf not reclaimable as powder of any specification and swarf reclaimable as powder of lower specification.

2



Diagrammatic representation of the die hot pressing of beryllium powder to produce near net shaped components.

VACUUM or DIE HOT PRESSING

FIGURE 2

APPROVED

PRODUCTION

IN USE 15 YEARS

PROBLEMS

POOR DIE LIFE

PROCESS OPERATOR DEPENDANT

ONE PER DAY

POOR NET SHAPE

4

SLID

REVIEW OF CURRENT AWRE DEVELOPMENT WORK IN THE PRODUCTION OF BERYLLIUM PREFORMS BY POWDER METALLURGY

POOR NET SHAPE

↓

THICK WALL PREFORM
to resist cooling stresses
between graphite core and preform

↓

SOLUTION
STAINLESS STEEL MANDREL

↓

PROBLEM
REACTION BETWEEN STAINLESS STEEL AND BERYLLIUM
MANDREL LIFE - ONE PRESSING AT 1050

↓

DEVELOPMENT

ERBIUM OXIDE COATING

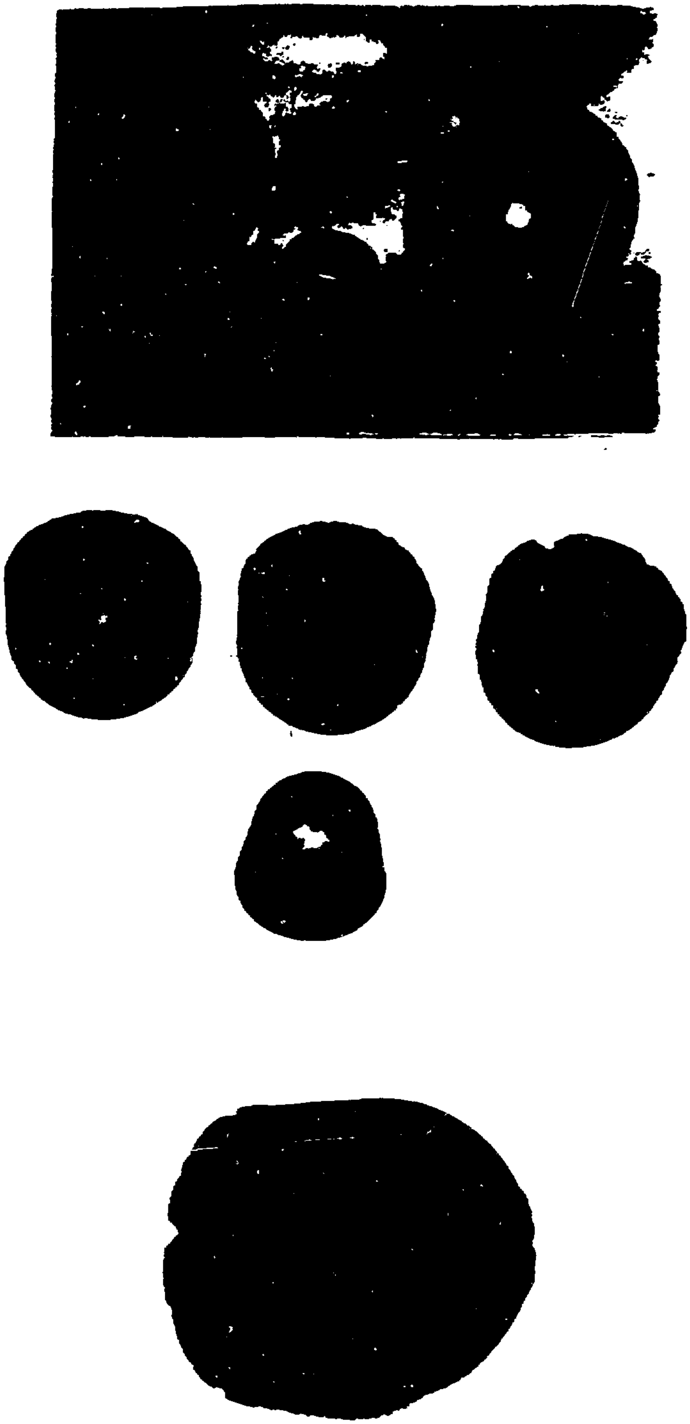
↓

THREE RUNS BEFORE RE- COATING

FIGURE 3

5

REVIEW
6

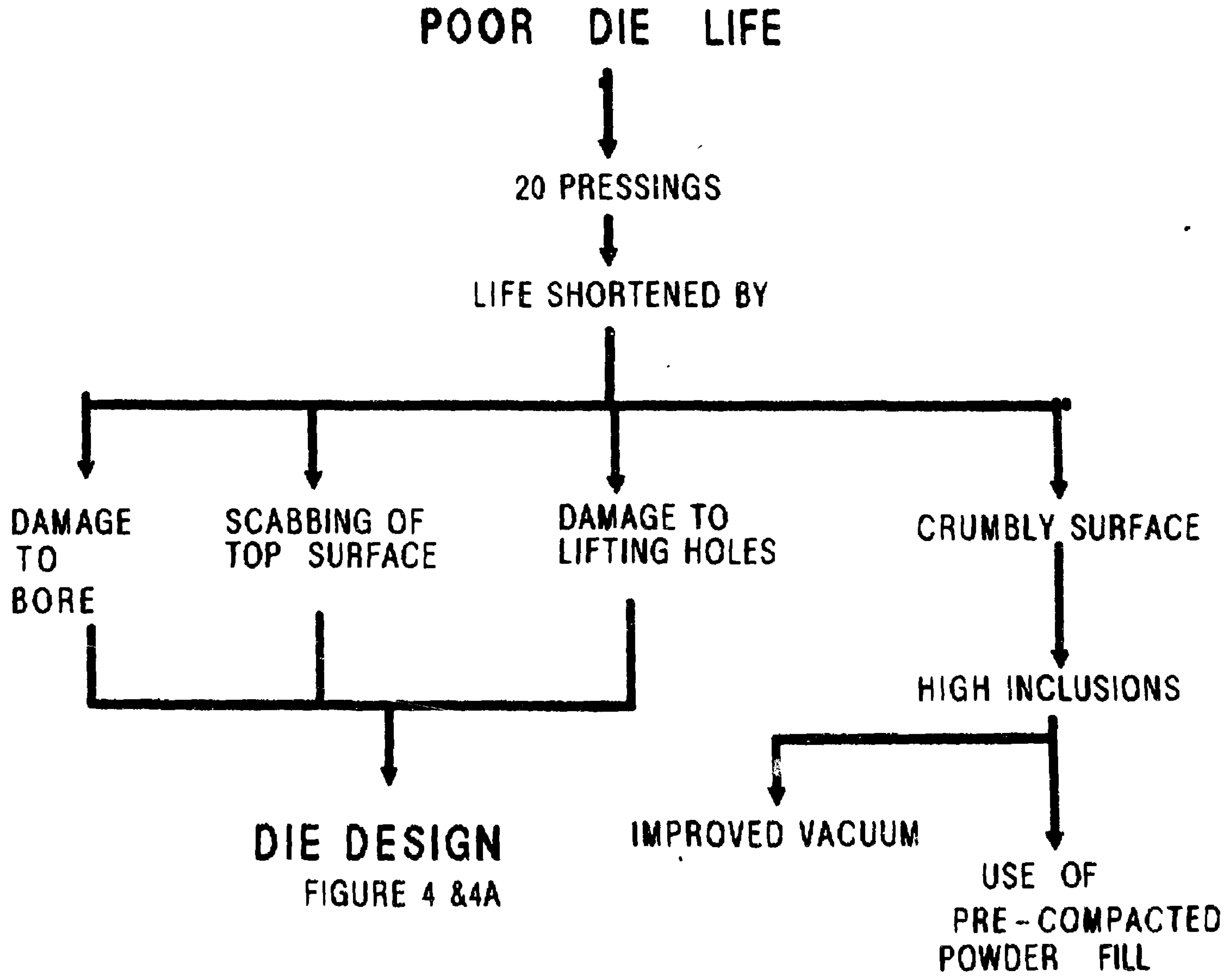


Two series of 110mm diameter preforms produced by hot pressing using an erbia coated stainless steel mandrel. In each series the coating failed after three pressing cycles.
(a) Coated stainless mandrel size as used for graphite
(b) Coated stainless mandrel large size
(c) Large size graphite core (cracked preform)

6

Section through Thin walled preforms produced by
(A) Graphite core
(B) Erbia coated stainless steel mandrel

SLIDE 8



POOR DIE LIFE

20 PRESSINGS

LIFE SHORTENED BY

DAMAGE TO BORE

SCABBING OF TOP SURFACE

DAMAGE TO LIFTING HOLES

CRUMBLY SURFACE

HIGH INCLUSIONS

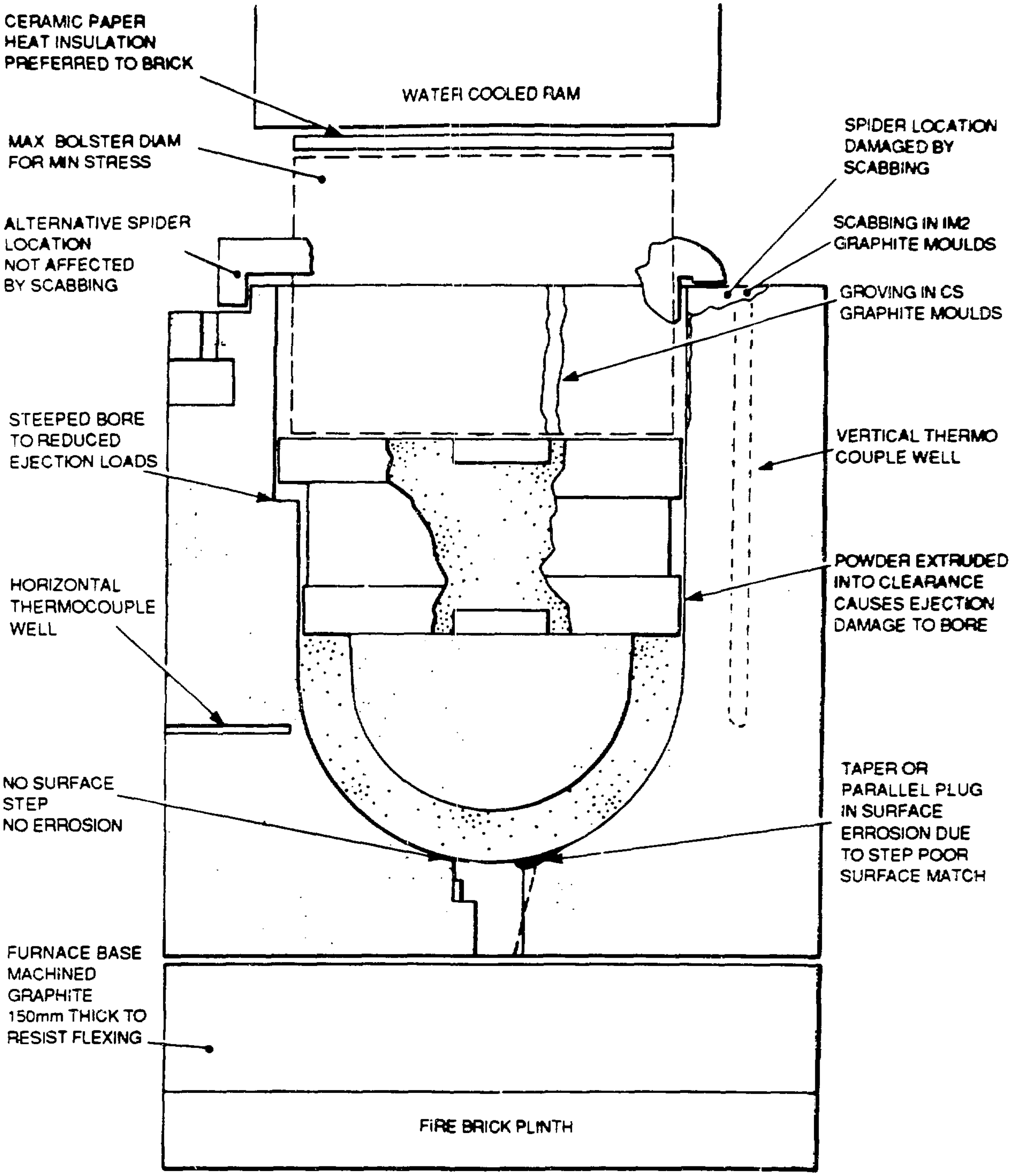
DIE DESIGN

FIGURE 4 &4A

IMPROVED VACUUM

USE OF PRE-COMPACTED POWDER FILL

REVIEW
#9



9

Design of a hot pressing assembly with modifications to reduce wear.

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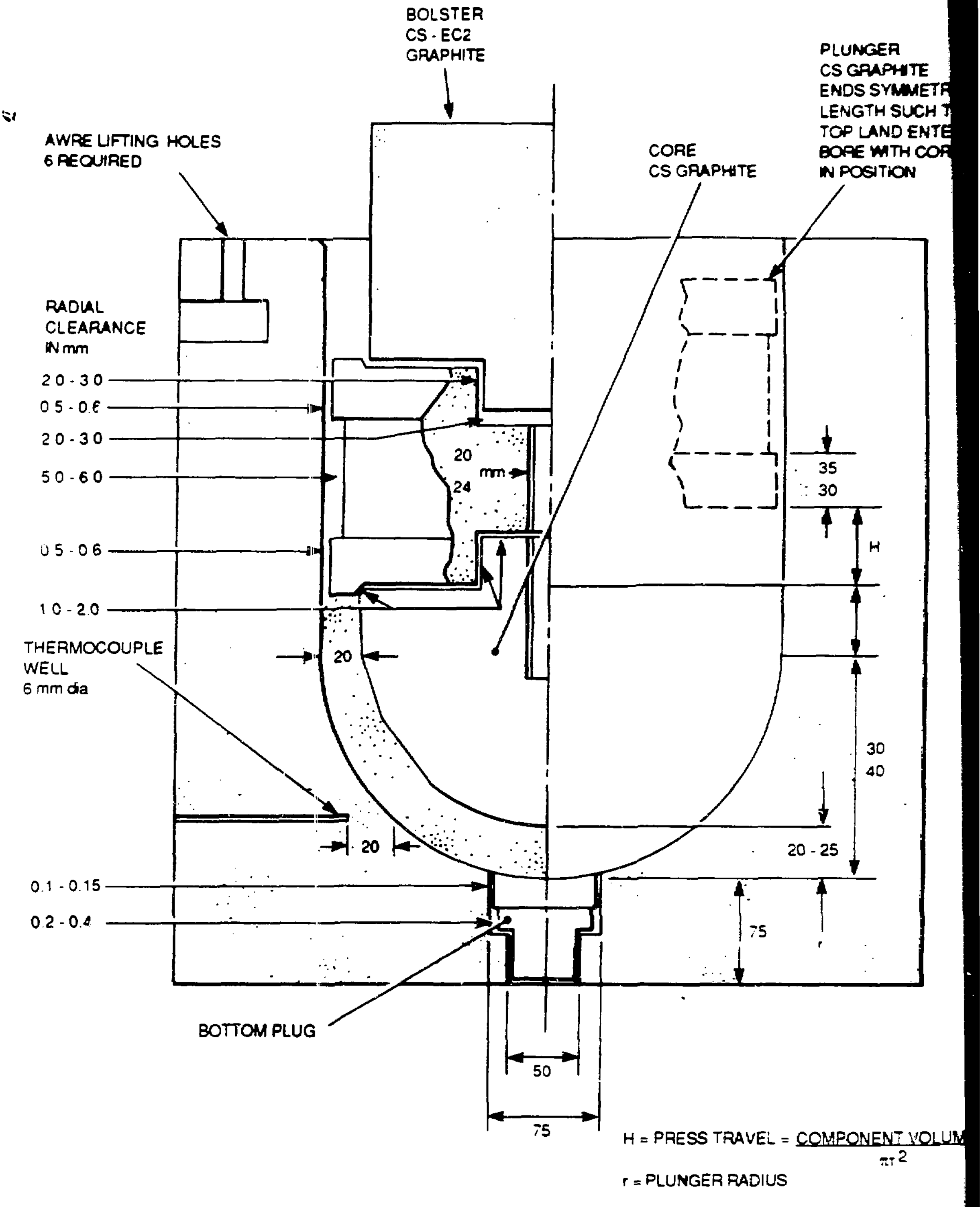


Typical ovoid hot pressing mould showing - material - design - clearances and minimum lengths

SLID
10

SLOW PROCESS

ONE PREFORM PRESS PER DAY

↓

TOOLING DESIGNED TO PRESS

TWO PREFORMS IN SINGLE DIE

↓

PROBLEM
FILLING OF BOTTOM CAVITY DIFFICULT
DENSITY OF BOTTOM PREFORM
LESS THAN 100%

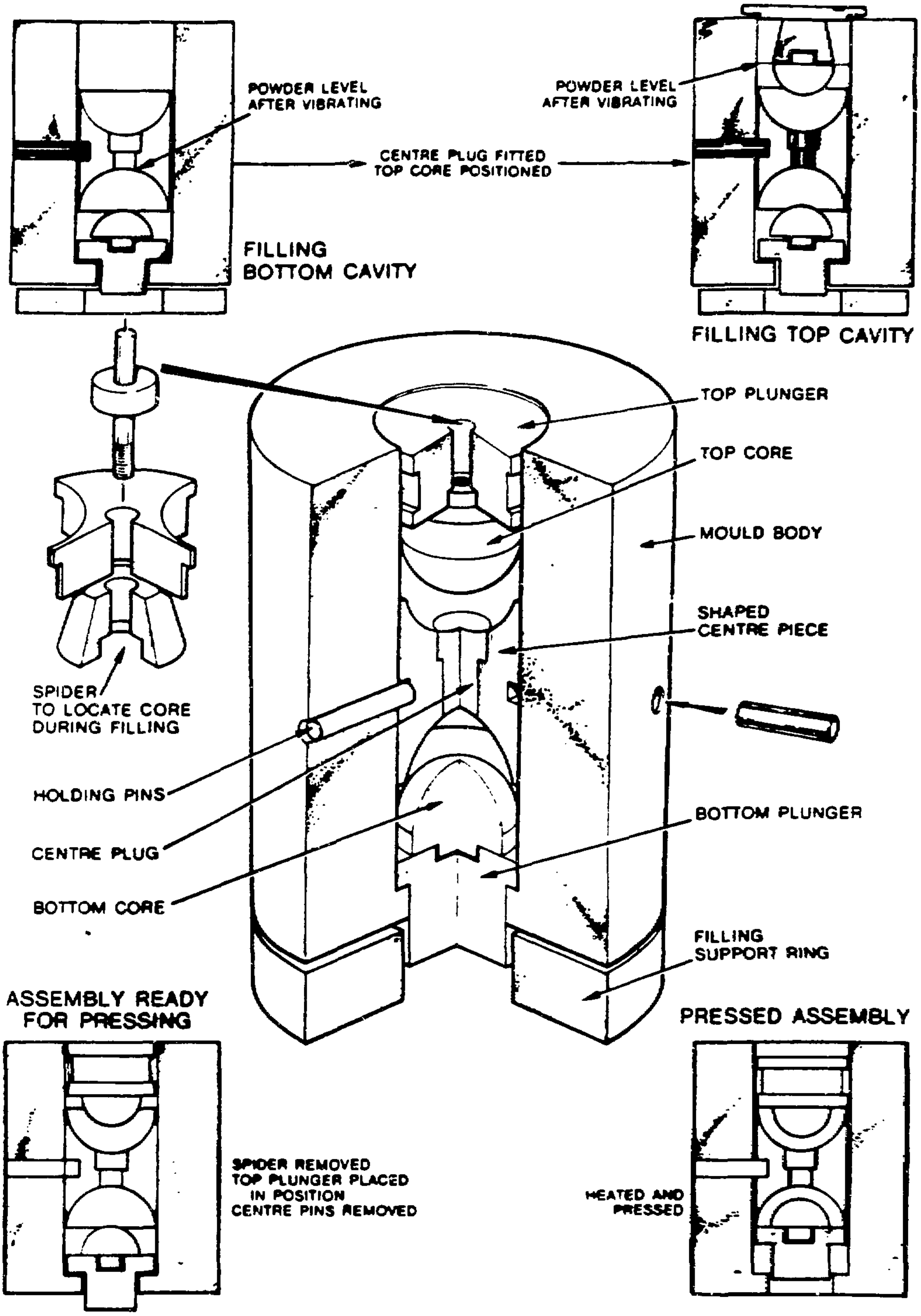
↓

PRECOMPACTED POWDER FILL IN BOTTOM CAVITY

CIPed POWDER PREFORM

A

II



Die design developed to produce two preforms in one pressing operation. Process steps shown.

SLIDE #15

REVIEW OF CURRENT AWRE DEVELOPMENT WORK IN THE PRODUCTION OF BERYLLIUM PREFORMS BY POWDER METALLURGY

HOT PRESSING OF PRECOMPACTED POWDER CIPed PREFORM

PURPOSE
REDUCE POWDER MOVEMENT
IMPROVE POWDER FILL
IMPROVE PROCESS CLEANLINESS

PROBLEM

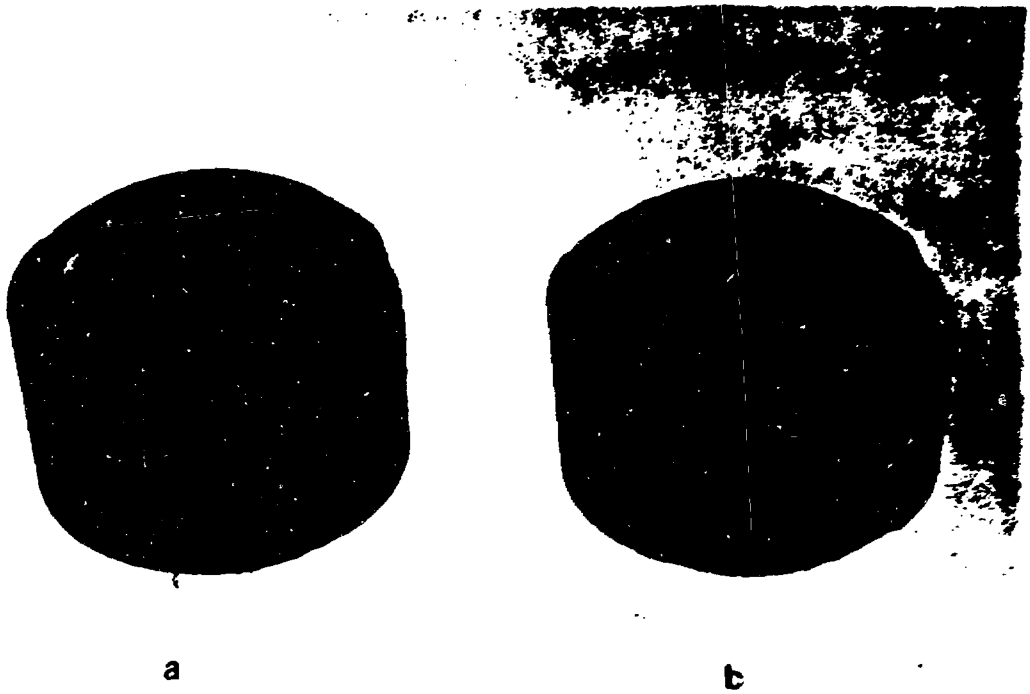
CIPed PREFORM CONTRACTION NOT AS PREDICTED
POOR FIT BETWEEN PREFORM AND DIE CONTOUR

RESULT

GREEN PREFORM CRACKED IN DIE
DURING INITIAL PRESSING
CRACKS HEAL UP DURING FINAL DENSIFICATION

SURFACE OF PREFORM FURROWED

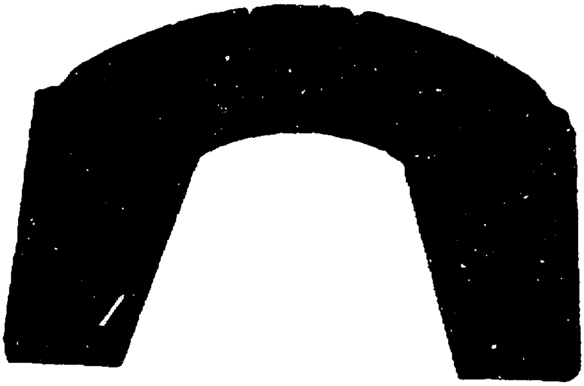
15



Two preforms produced in one pressing operation using tooling described in Figure 5.
Preform (a) pressed in bottom cavity from CIPed preform fill. Cracked on spherical surface due to shape mismatch.
Preform (b) pressed in top cavity from powder fill.

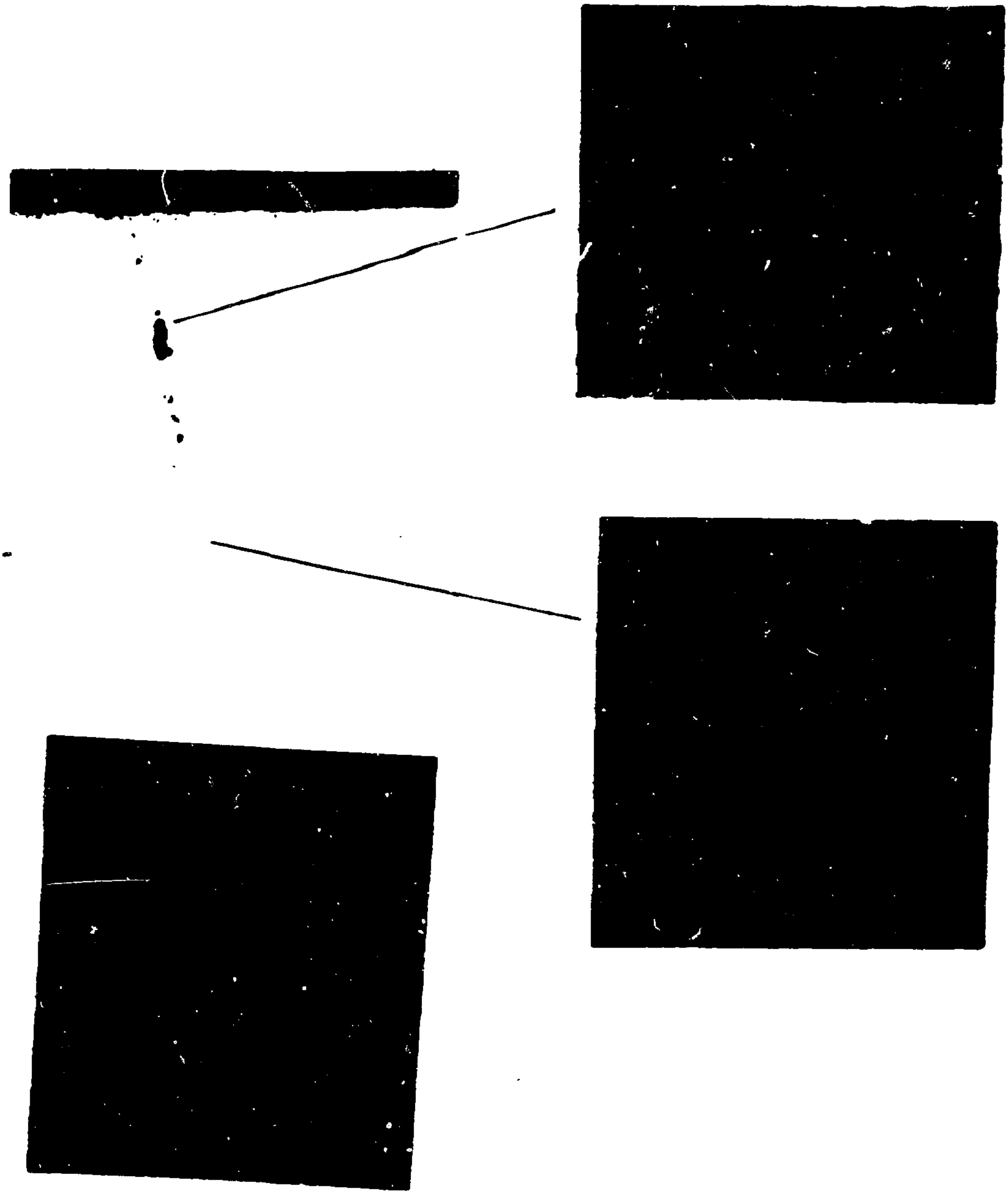
14

REVIEW
(3



Section of CIP hot pressed preform (Figure 6 (a)) through area of crack. Crack appears confined to surface.

15



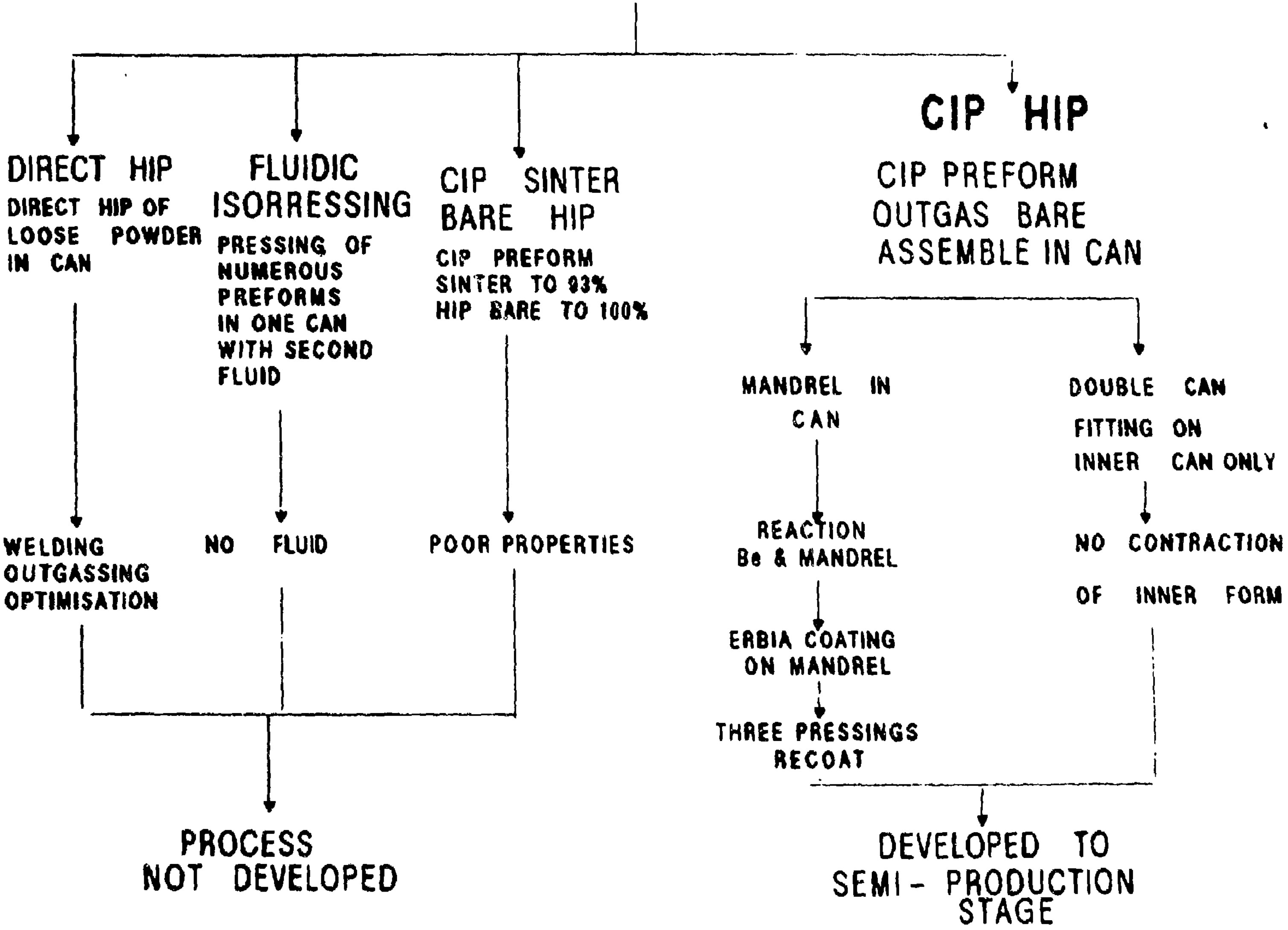
Section of CIP hot pressed preform (Figure 6a) through area of crack. Crack appears confined to surface.

16

SLIDE 14

REVIEW OF CURRENT AWRE DEVELOPMENT WORK IN THE PRODUCTION OF BERYLLIUM PREFORMS BY POWDER METALLURGY

DEVELOPMENT OF ISOPRESSING ROUTE



DIRECT HIP
DIRECT HIP OF LOOSE POWDER IN CAN
WELDING
OUTGASSING
OPTIMISATION

FLUIDIC ISORRESSING
PRESSING OF NUMEROUS PREFORMS IN ONE CAN WITH SECOND FLUID
NO FLUID

CIP SINTER BARE HIP
CIP PREFORM
SINTER TO 93%
HIP BARE TO 100%
POOR PROPERTIES

PROCESS NOT DEVELOPED

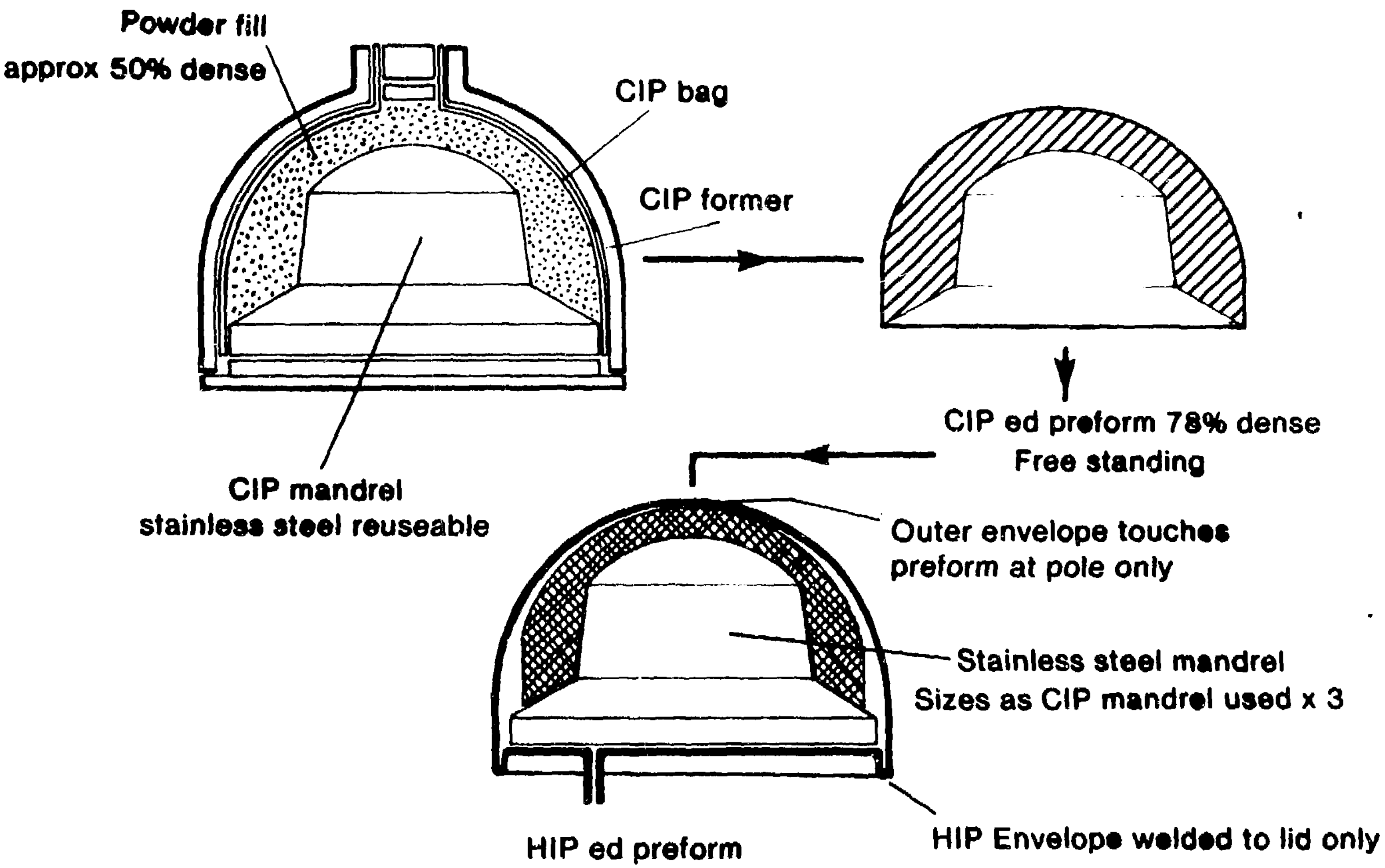
CIP HIP
CIP PREFORM
OUTGAS BARE
ASSEMBLE IN CAN

MANDREL IN CAN
REACTION Be & MANDREL
ERBIA COATING ON MANDREL
THREE PRESSINGS
RECOAT

DOUBLE CAN
FITTING ON INNER CAN ONLY
NO CONTRACTION OF INNER FORM

DEVELOPED TO SEMI-PRODUCTION STAGE

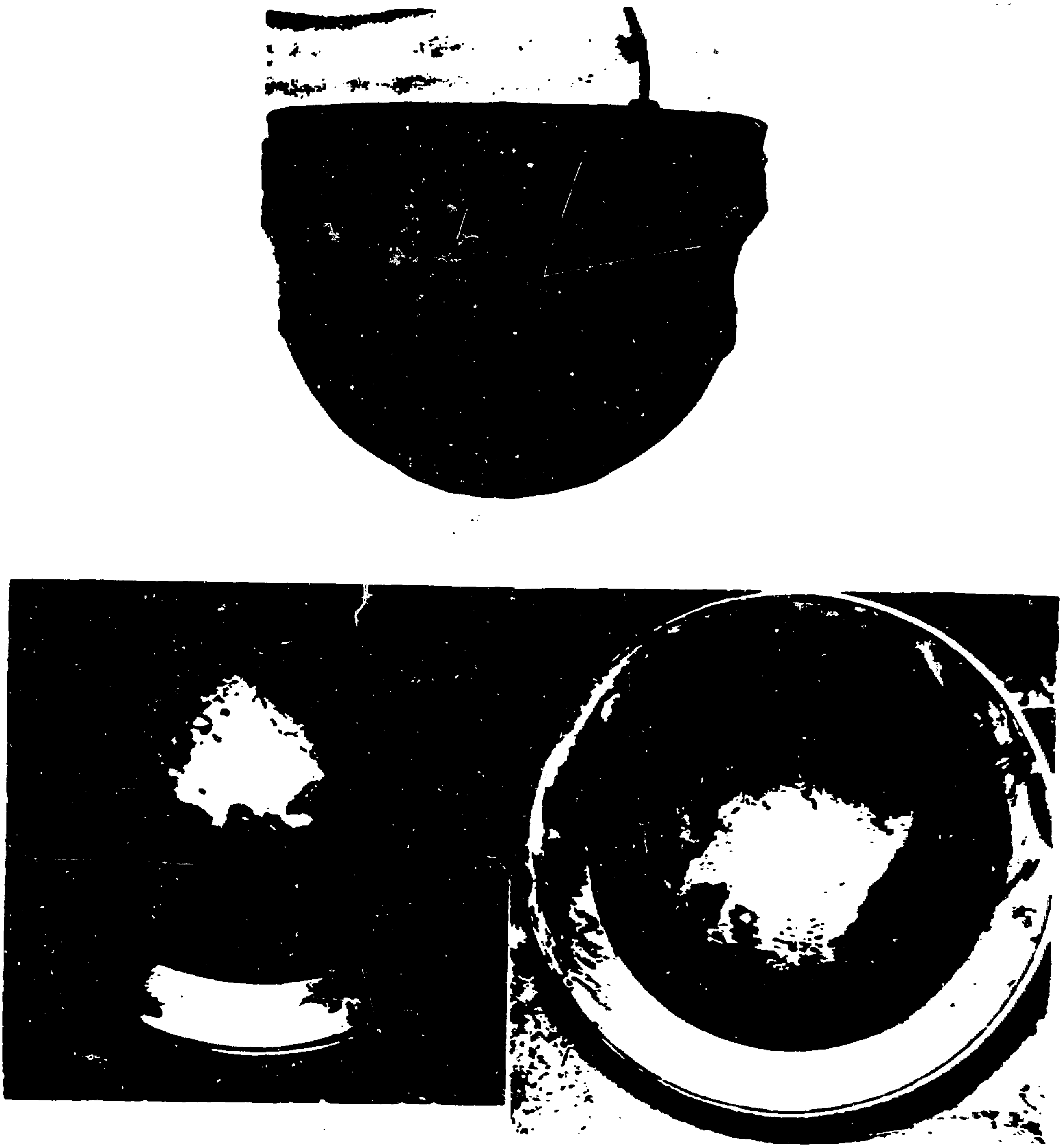
REVIEW
FG
17



Diagrammatic representation of an isopressing process based on the CIP-HIP route using a mandrel in the hot isopressing stage.

Review

19



200mm diameter preform produced by CIP-HIP (mandrel) process. Mandrel uncoated stainless steel.

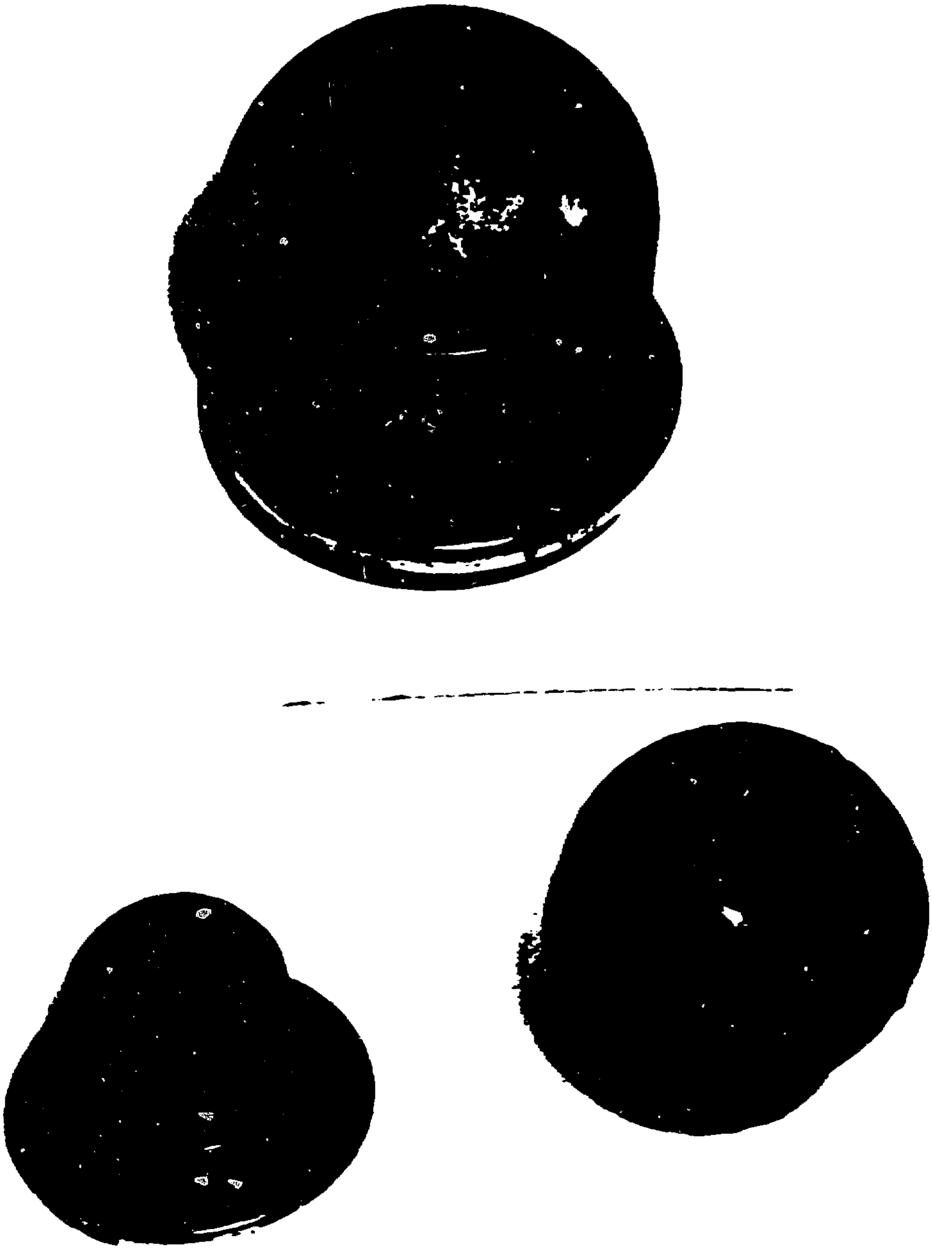
assembly as pressed

Mandrel interior surface of preform

19

RE

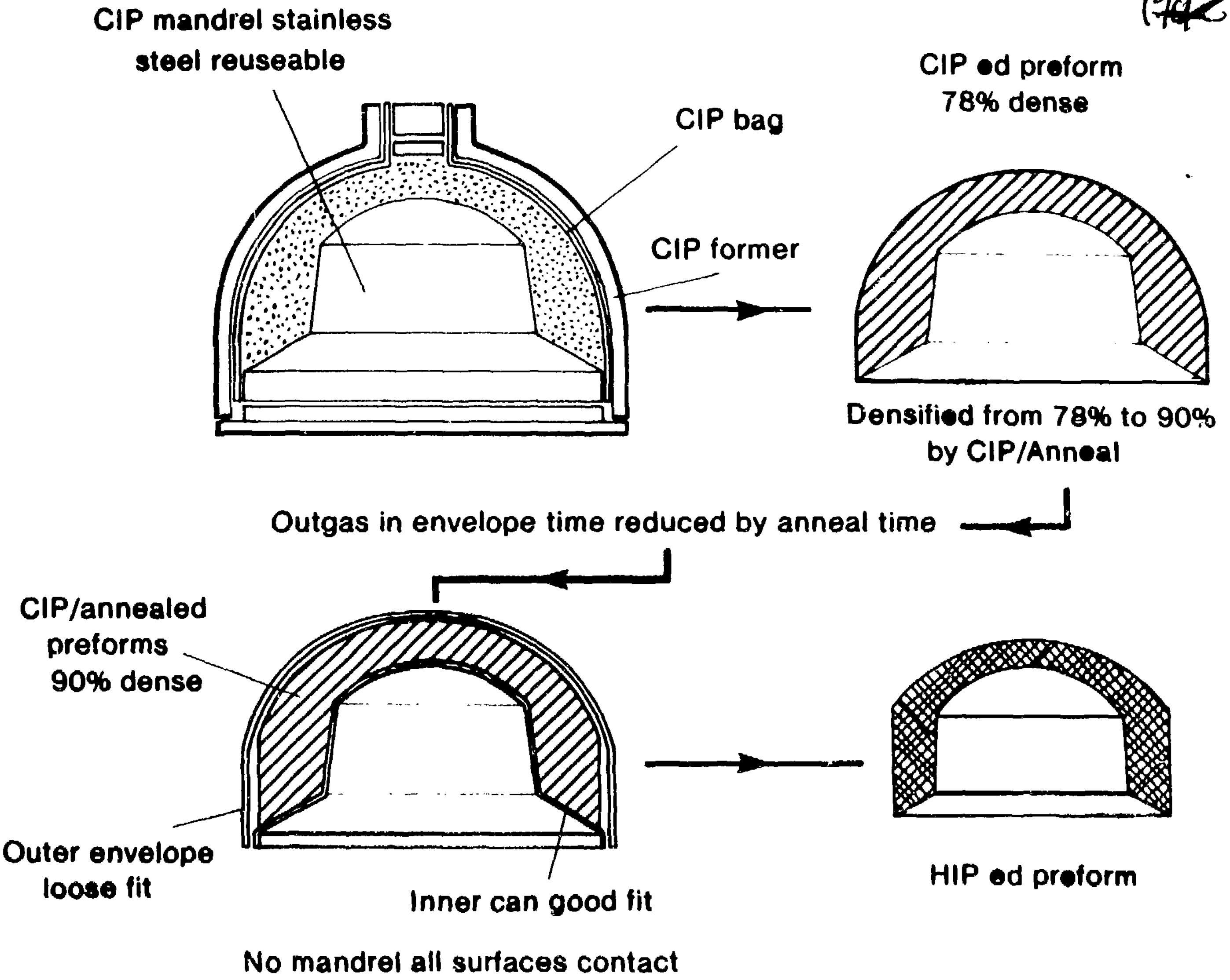
1



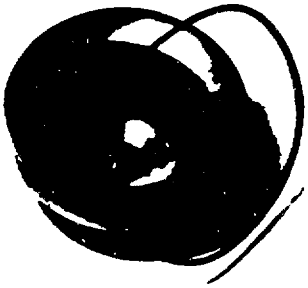
110mm diameter preform produced by mandrel HIP process. First pressing of a series using an erbia coated mandrel. No evidence of reaction between mandrel and preform inner surface. Erbia coated mandrel used. Some damage to coating due to preform movement during can machining.

20

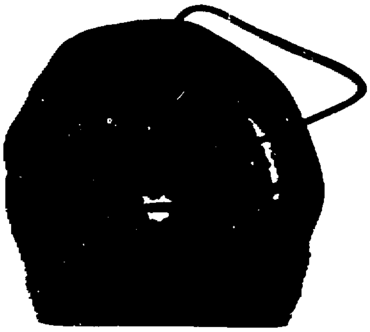
REVIEW 14



Diagrammatic representation of an isopressing process based on the CIP-HIP route using a double can in the hot isopressing stage.



CIPed PREFORM AS CANNED



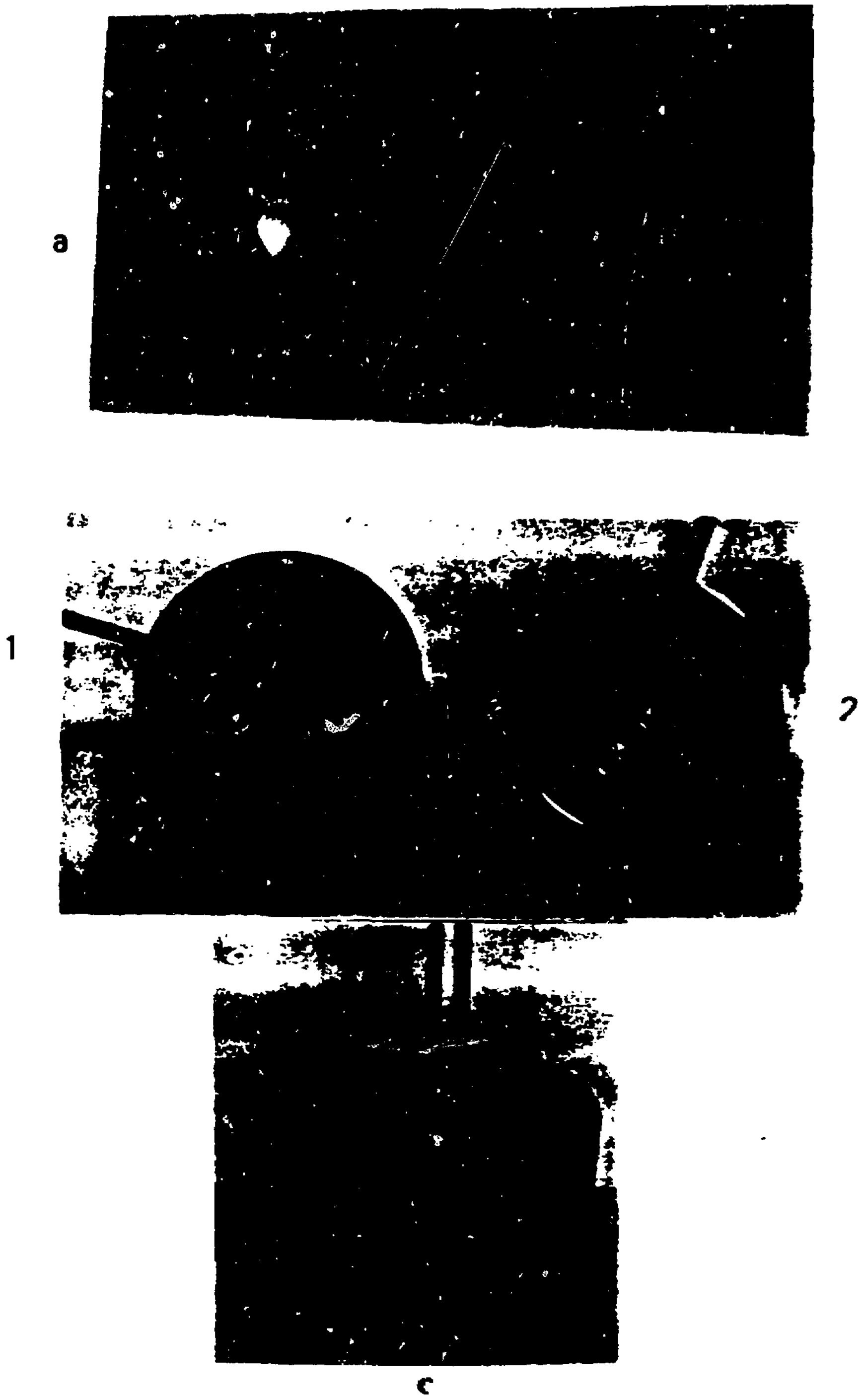
AFTER HIPing

PREFORM AS DECANNED

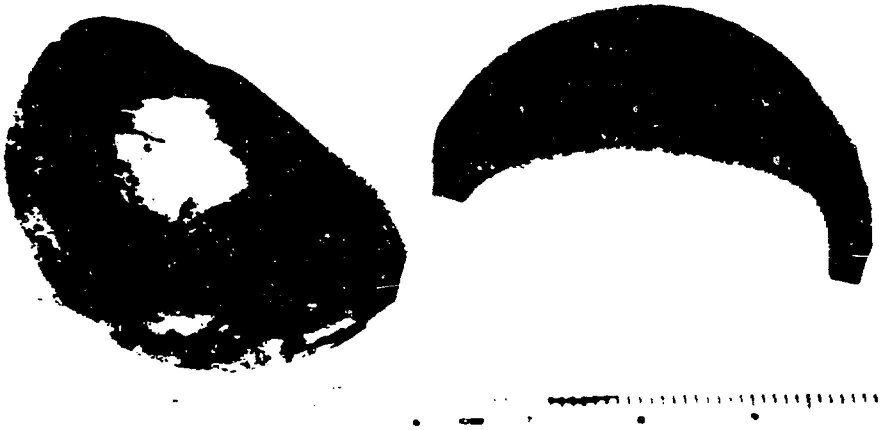
200mm diameter preform produced by double can HIP process.

22

Review 2

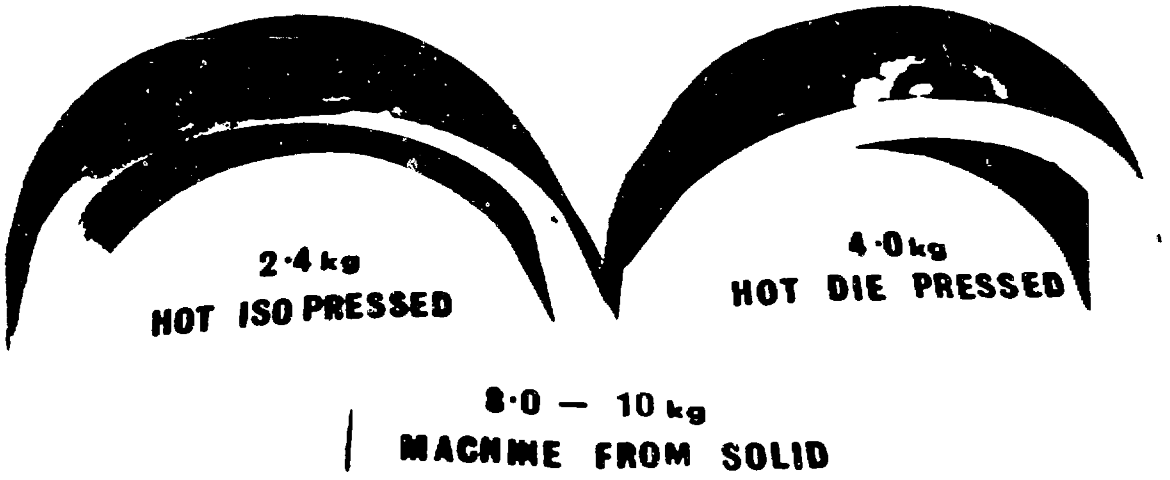


Stages in hot isopressing 110mm diameter preform in a double can
- (a) Outer and inner can
- (b) Can inner surface (1) as pressed
  (2) as welded
- (c) Outer surface as pressed



110mm diameter preform produced by double can HIP process

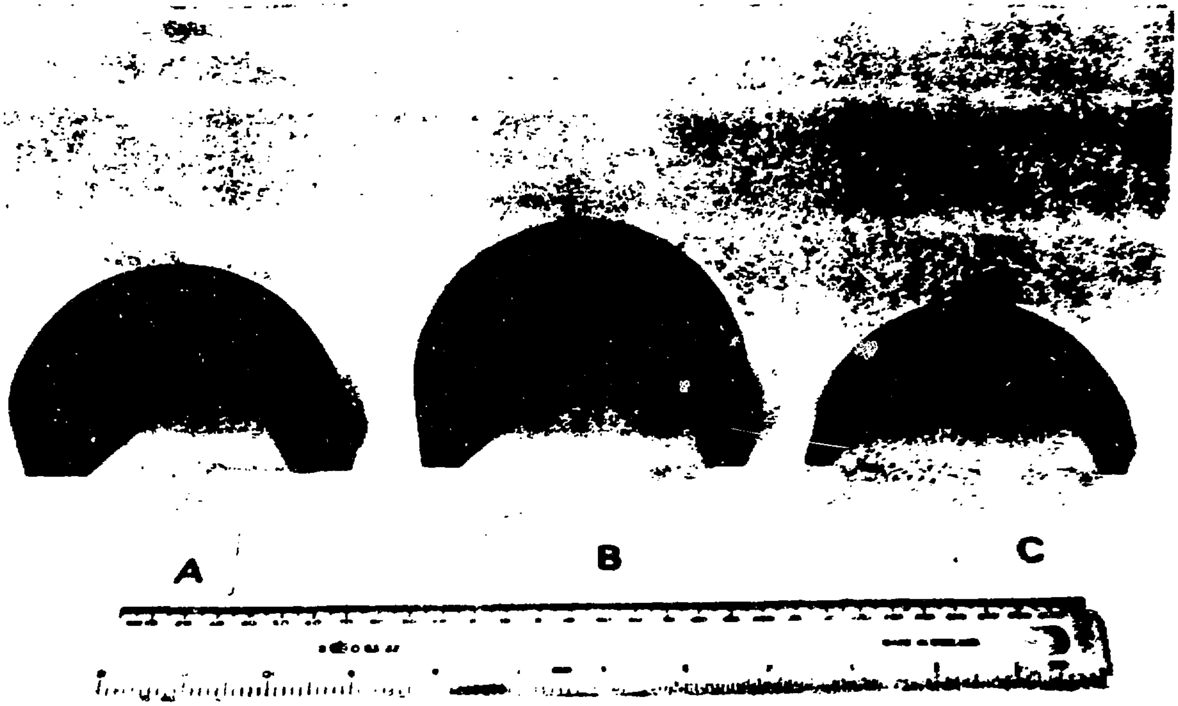
REVIEW



Sections through hot isopressed and a die hot pressed preform to show weight saving advantages of isopressing.
The machine from solid weight shown would be the weight of a cylindrical billet required to enable the preform to be machined from a regular billet.

25

REVIE
3



Cross section through 110 mm preforms produced by:
- (a) Die hot pressing with graphite core
- (b) Coated stainless steel core
- (c) Hot isopressing of a CIPed preform in a double can.

DEVELOPMENT OF BERYLLIUM NEAR-NET-SHAPE TECHNOLOGY AT BRUSH WELLMAN INC.

Monty Kuxhaus
Senior Engineer

Brush Wellman Inc
South River Road
Elmore, Ohio 43416

Current status of the capabilities to produce near-net-shape beryllium shapes by direct HIPing, and the development activities planned for the near future at Brush Wellman are reviewed. Generic shapes and typical size capabilities are addressed. The development plans and activities for the use of CIP as a preform technique are also addressed. The relationship between the two processes and the generic advantages which guide their selection for specific applications are considered.

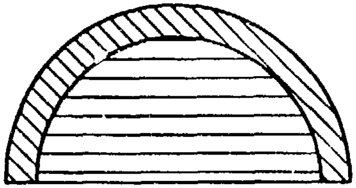
Editor's Note: The visuals for this presentation are not shown here because of reproduction constraints.

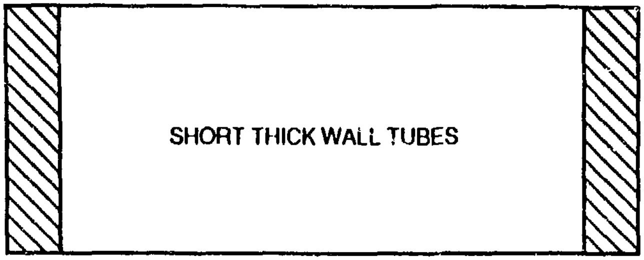
Production of New Geometrical Shapes

- Irregular rectangular blocks
- Hemi's
- Box types
- Tubes
- Light weight optics
- Complex shapes

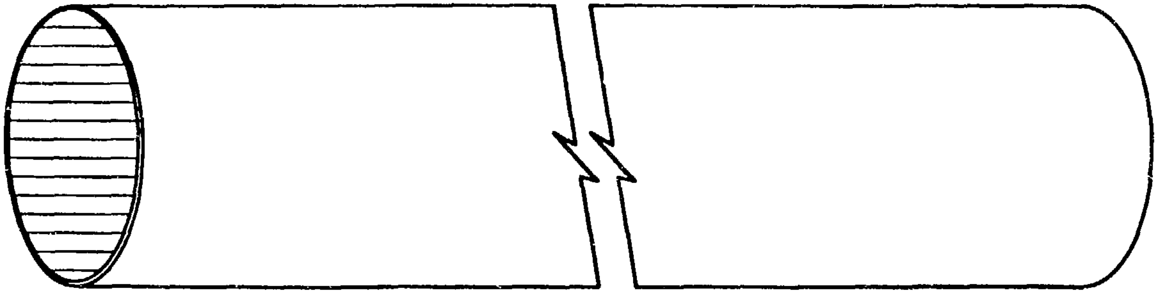
EXAMPLES OF N-N-S PARTS BEING CONSIDERED FOR DEVELOPMENT IN THE NEAR FUTURE

HEMI'S SOLIDS &SHELLS

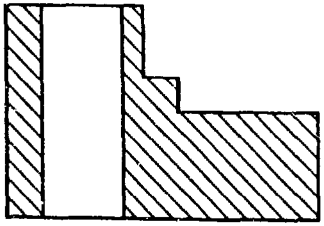


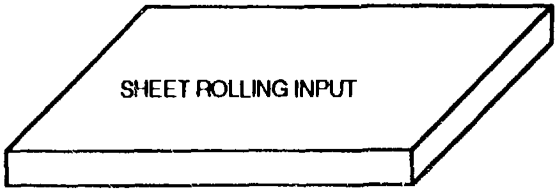


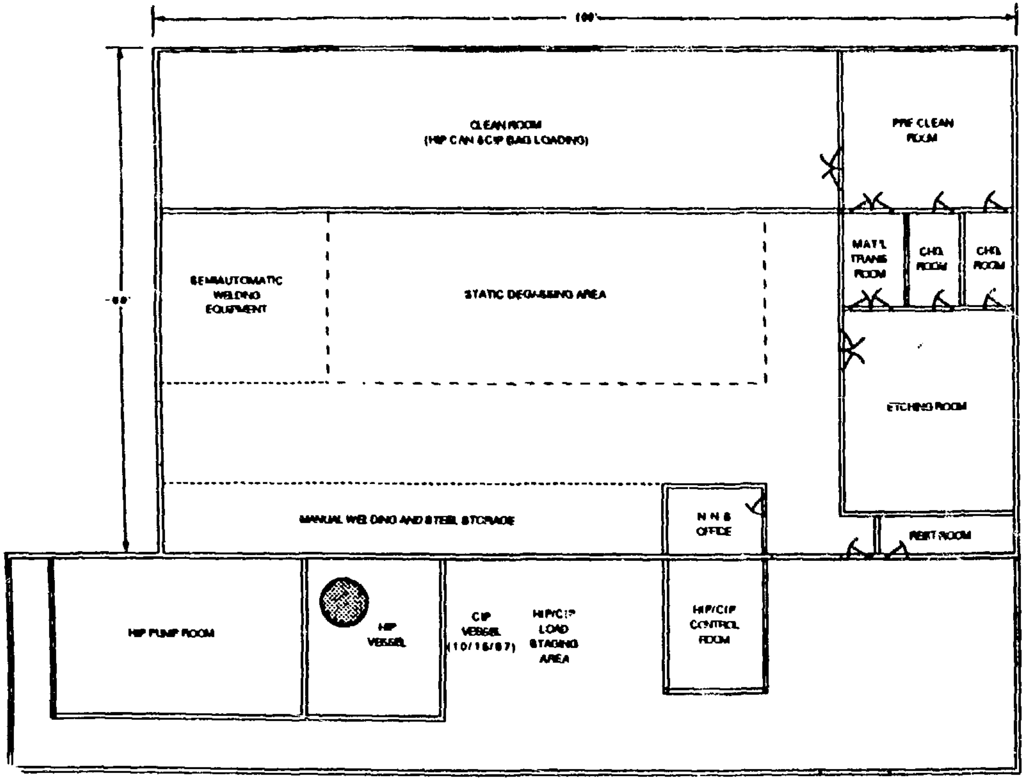
LONG THIN WALL TUBES



NUMEROUS COMPLEX SHAPES






100'
60'
CLEAN ROOM
(HIP CAN & CIP BAG LOADING)
PRE CLEAN ROOM
MAT'L TRANS ROOM
CHG. ROOM
CHG. ROOM
SEMIAUTOMATIC WELDING EQUIPMENT
STATIC DEGASSING AREA
ETCHING ROOM
MANUAL WELDING AND STEEL STORAGE
N N S OFFICE
REST ROOM
HIP PUMP ROOM
HIP VESSEL
CIP VESSEL (10/15/87)
HIP/CIP LOAD STAGING AREA
HIP/CIP CONTROL ROOM

Improved Process Productivity

- More efficient material flow through the area
- Semi automated welding systems
- More efficient powder loading systems
- Partial automation of the HIP can degassing systems
- Pilot scale bulk powder degassing (80 lbs/batch)
- New larger and automated vacuum sintering furnace

Improve the Skills of the Personnel

- Participation of engineers in professional societies
- Attendance of engineers at technical seminars
- Improved and documented working procedures
- More and improved training of the work force
- Planning and documentation of training

Produce High Quality Products

- High density
- Isotropic properties
- Minimization of x-ray inspection indications
- Dimensional consistency

New Processes

- HIP
- CIP/Sinter
- CIP/Sinter/HIP
- CIP/HIP
- Cold Press/Sinter/HIP

Cost Reduction

- Reduced engineering time to bring a part into production
- Reduced cost of input materials to the operations
- Improved material yields
- Reduced machining
- Reduced inspection and testing
- Improved process productivity

THE DEVELOPMENT OF A COMPACTION DIAGRAM FOR THE HOT CONSOLIDATION OF BERYLLIUM POWDER

by

G.Heald and H.G.Rhodes, AWRE

Summary

Compaction diagrams show the progress of densification in a material as a function of time, over a range of isobaric or isothermal pressing conditions. A diagram representing the compaction of S65 grade beryllium powder has been constructed using computer generated data. The predicted densification rates for temperatures below 873K are found to be much higher than those experienced in practice. The evaluation of the densification process for temperatures above 873K is being studied.

FIG 1. COMPACTION DIAGRAM

Tool Steel Ashby's Data

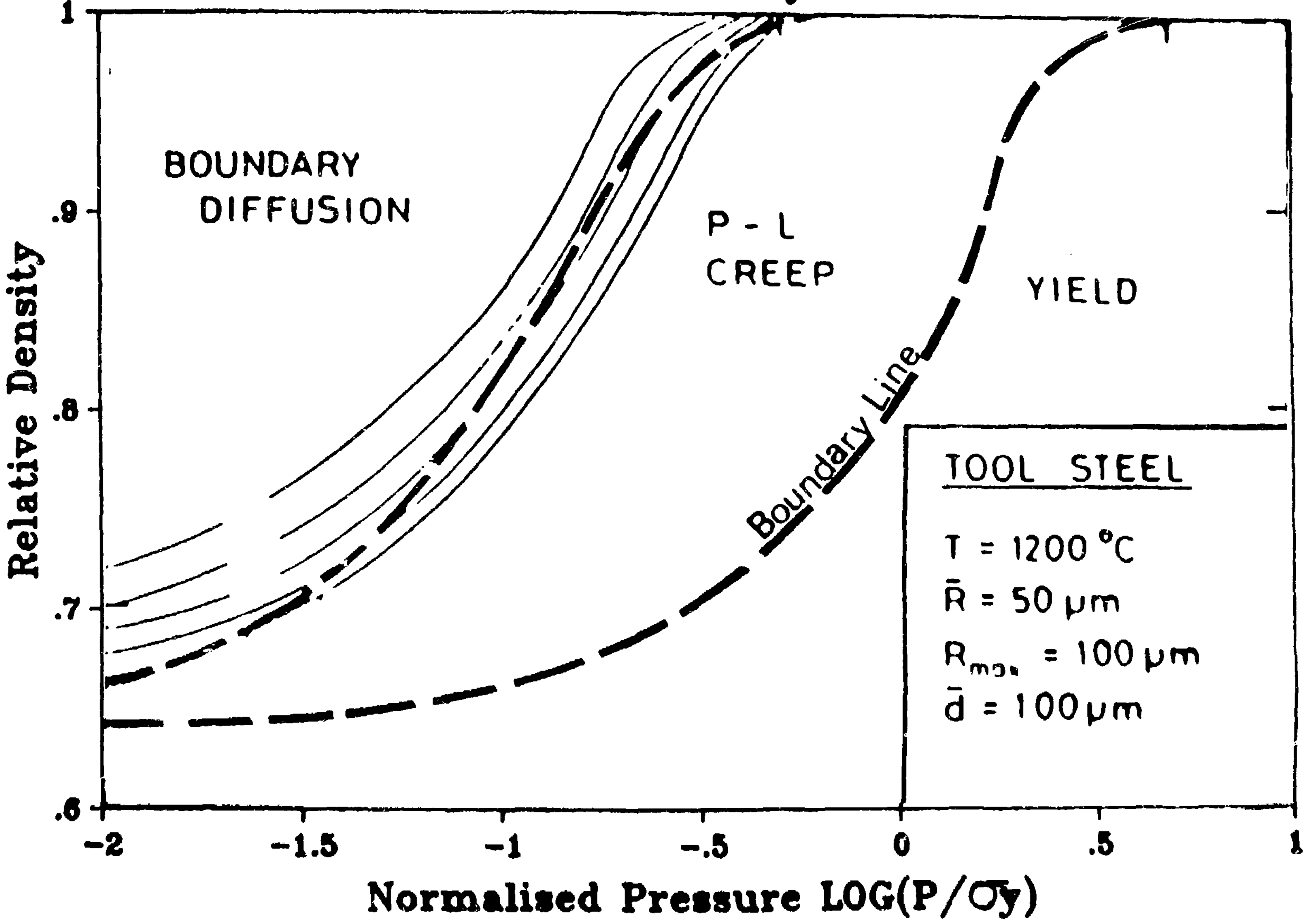


FIG 2. COMPACTION DIAGRAM

Tool Steel Powder T=1473 K

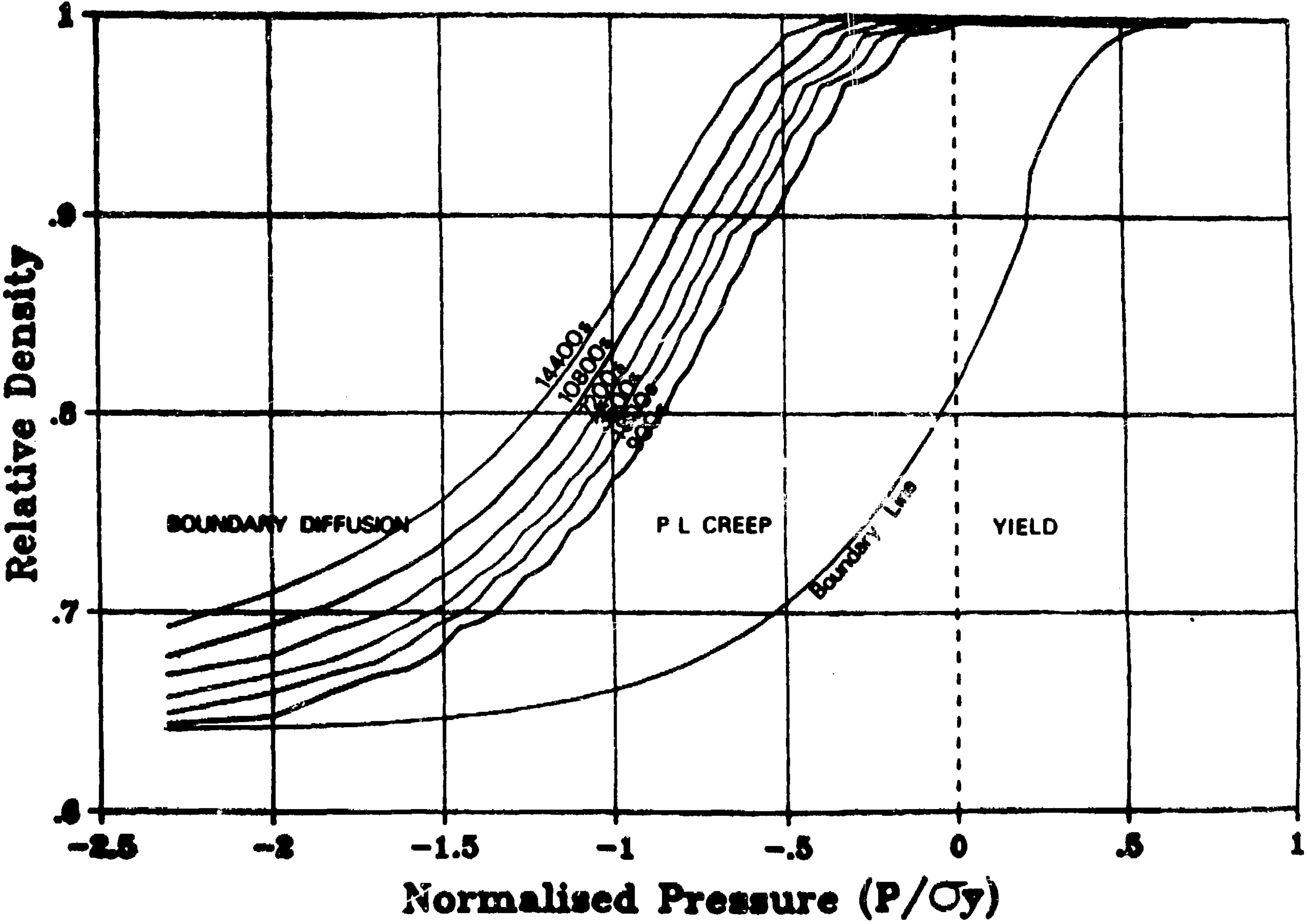


FIG 3. Be COMPACTION [illegible]

QMV Powder T=[illegible] K

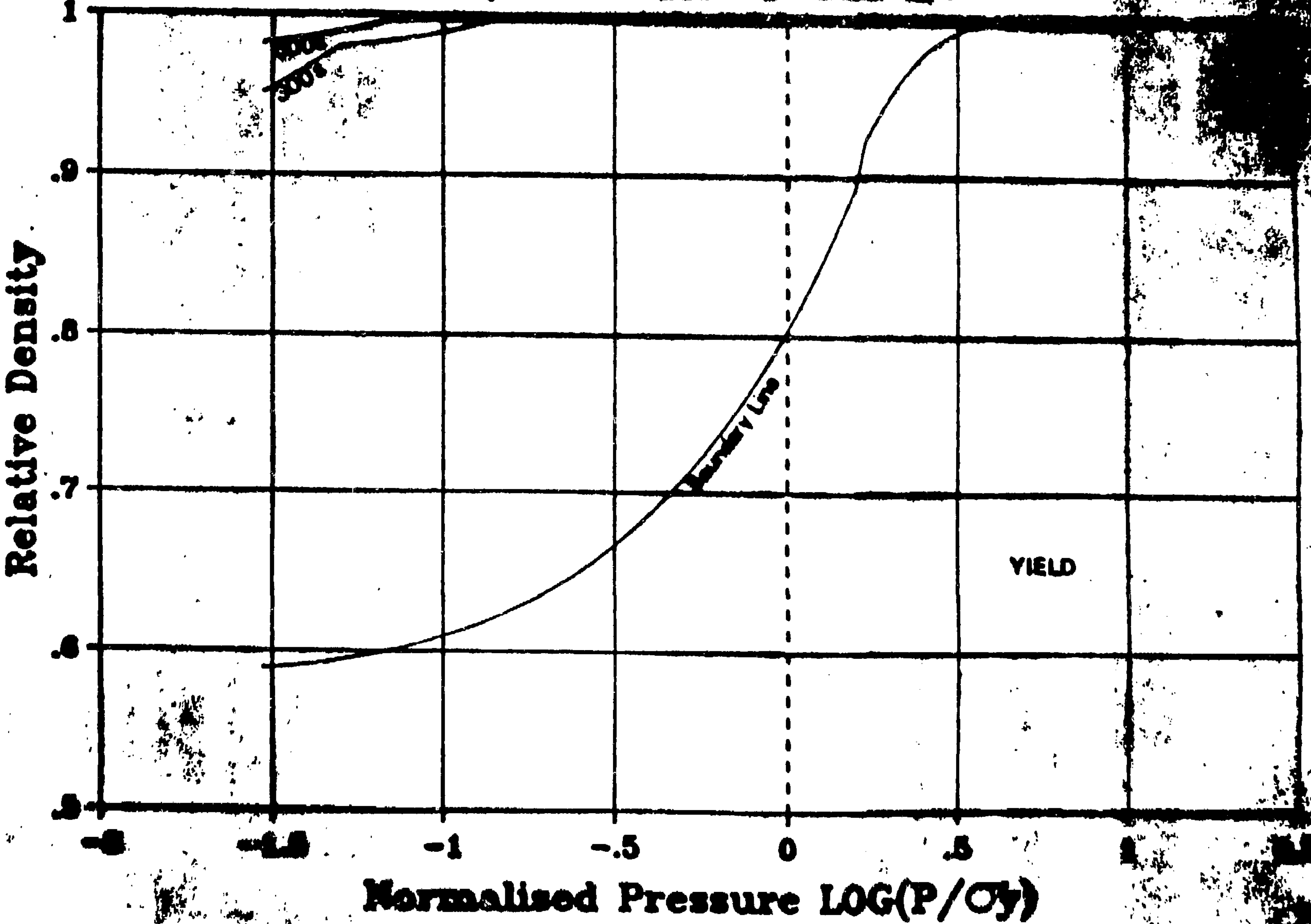


FIG 4. COMPACTION DIAGRAM

QMV Powder T=923 K

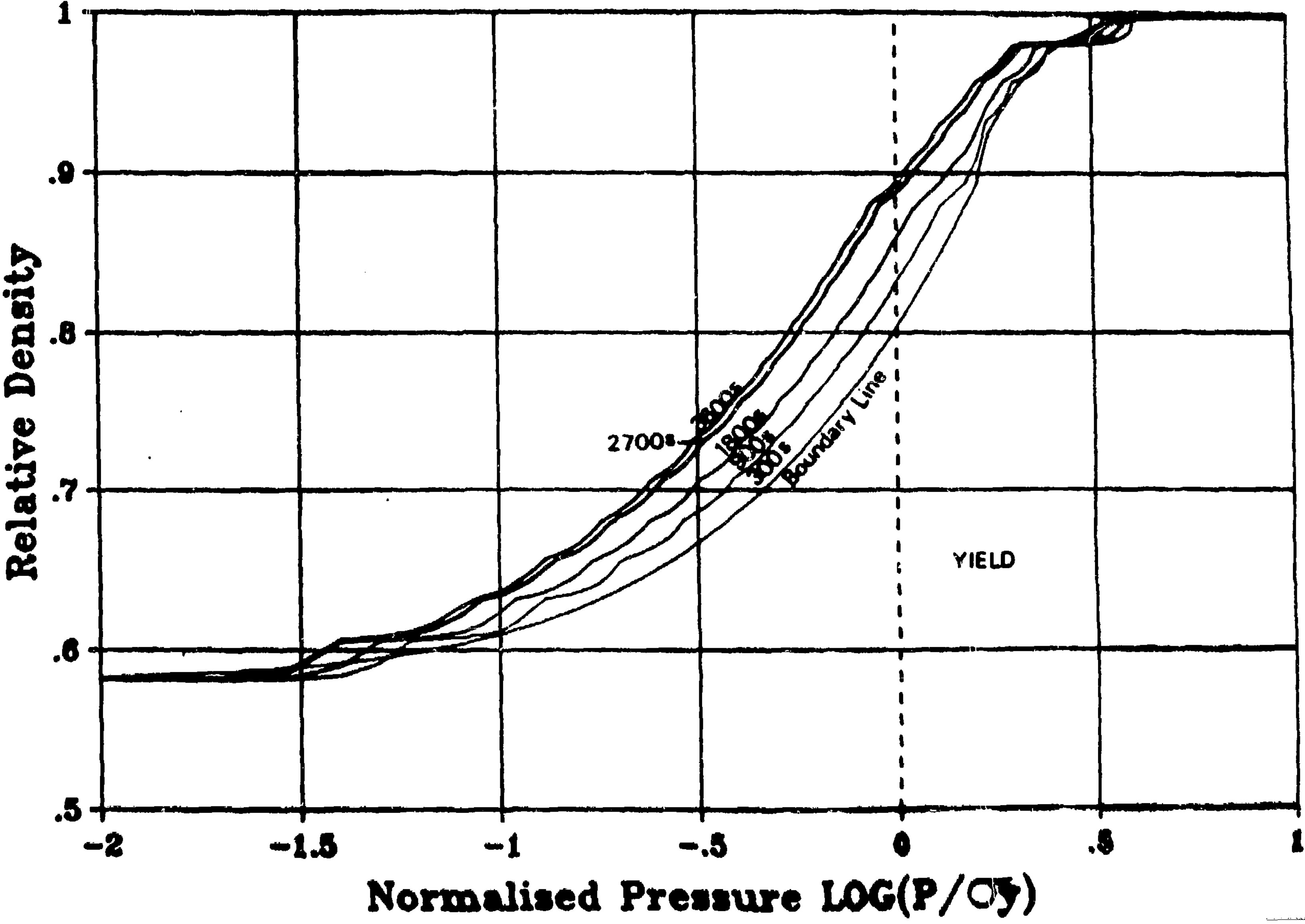


FIG 5. COMPACTION DIAGRAM

Be Preform T=923 K

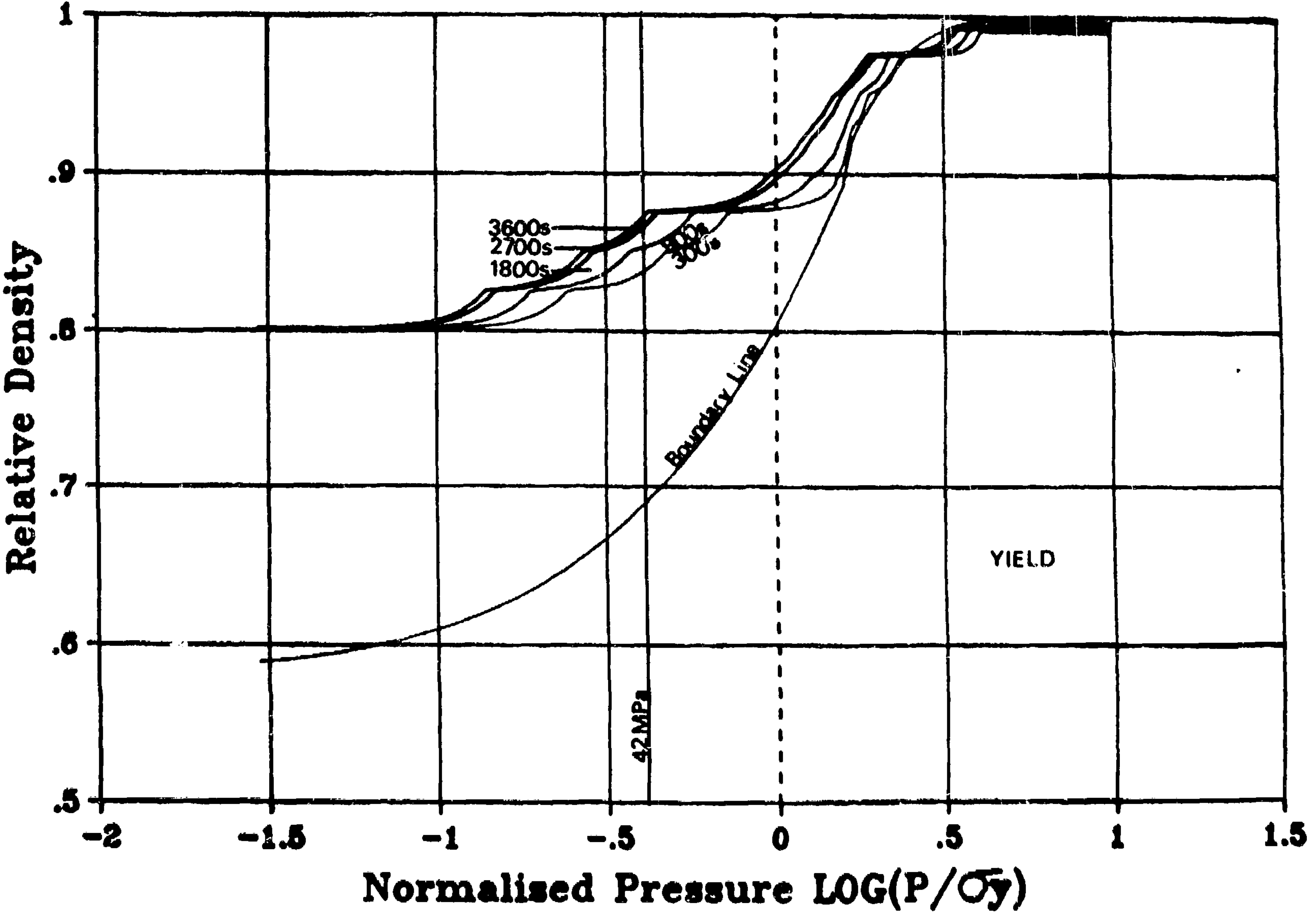


FIG 6. COMPACTION DIAGRAM

QMV Powder T=1003 K

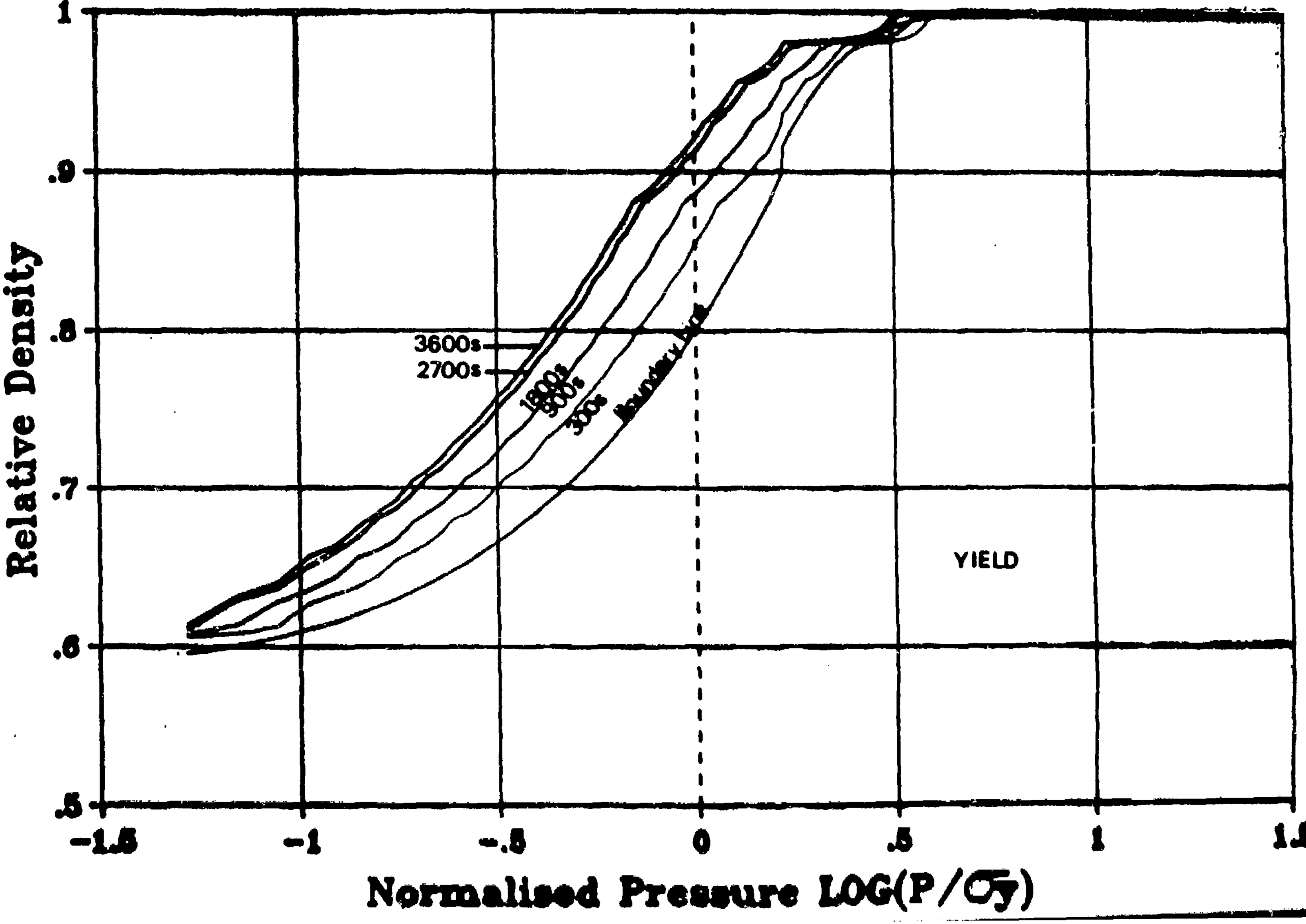


FIG 7. COMPACTION DIAGRAM

QMV Powder T=1068 K

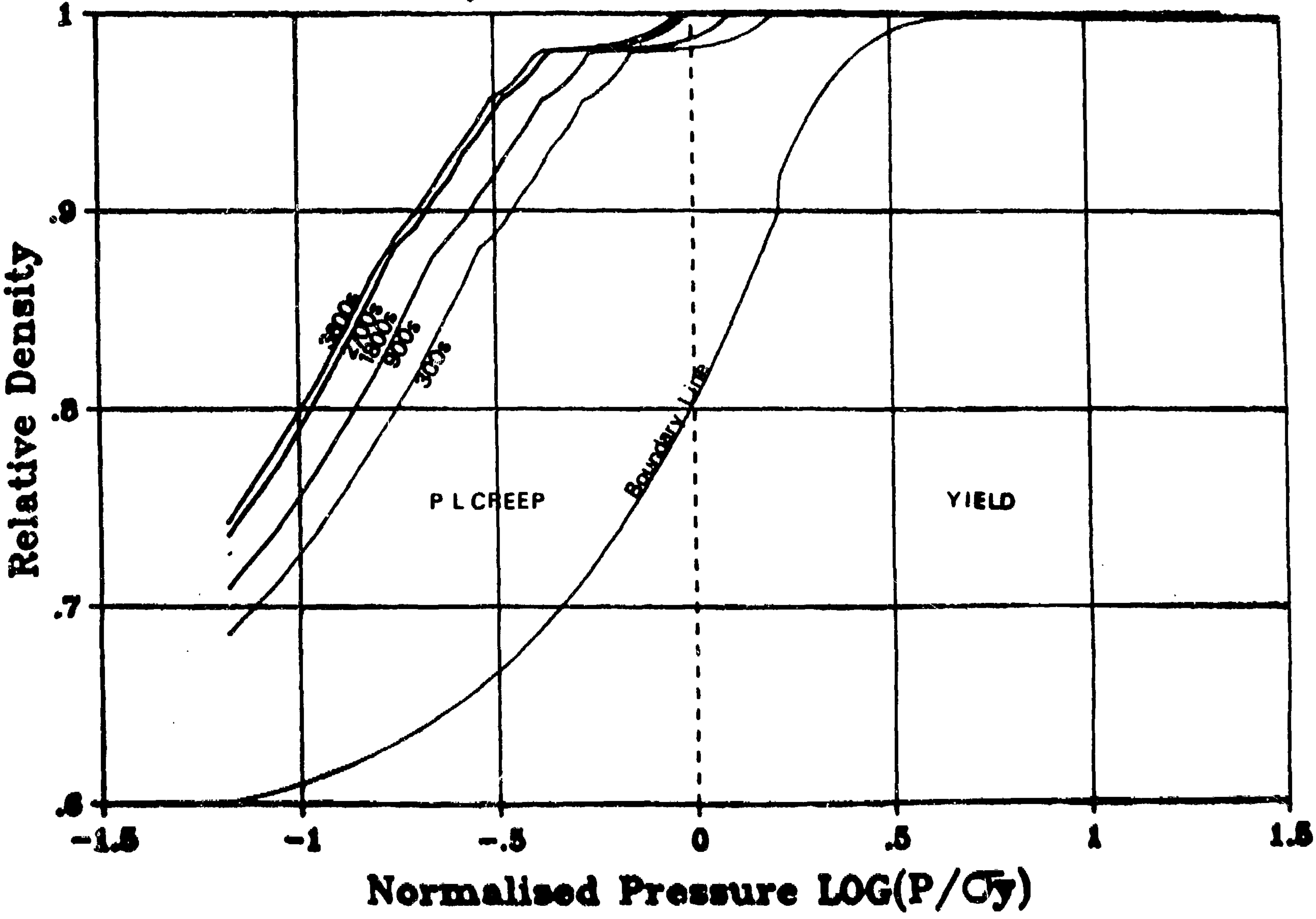
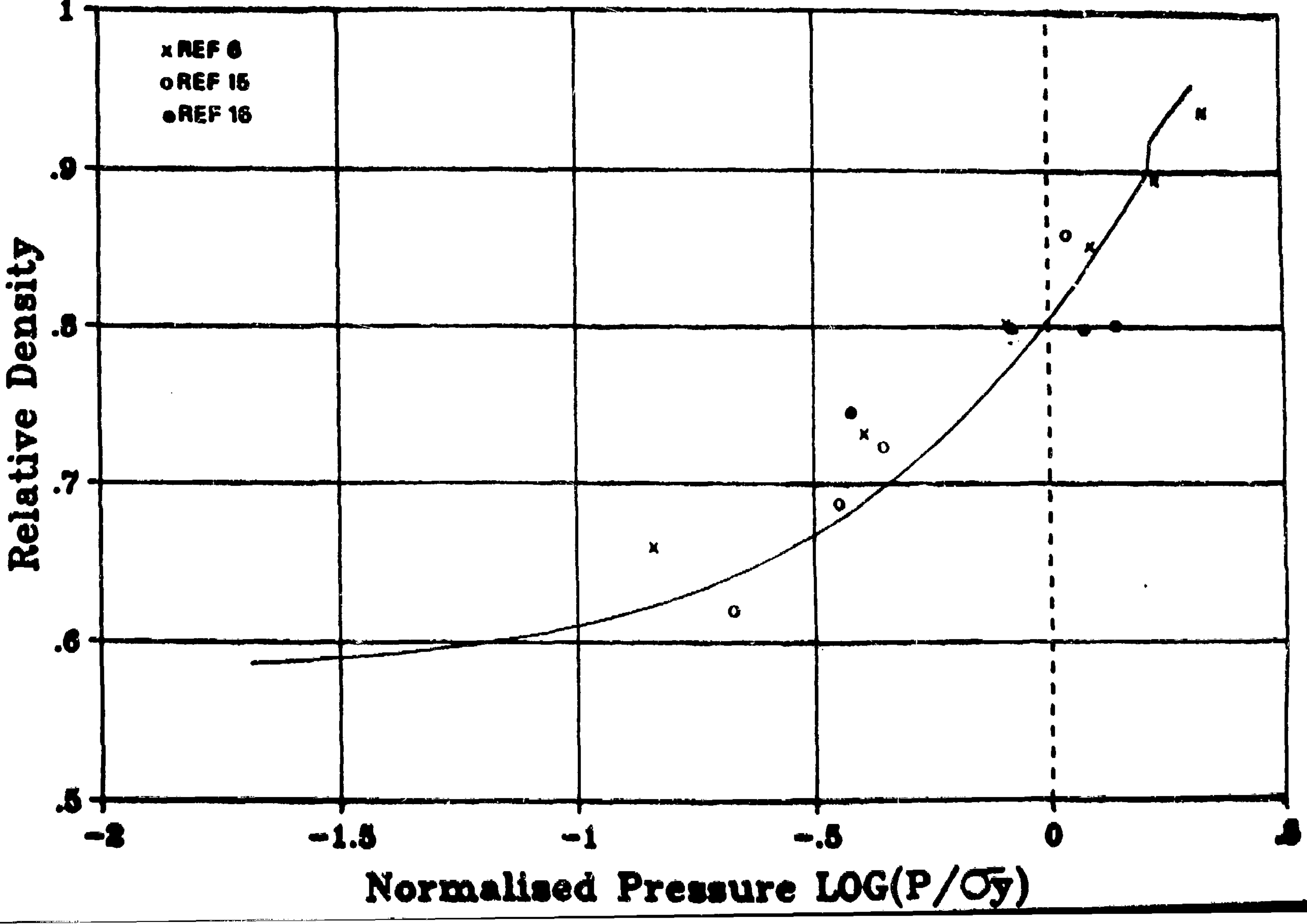


FIG 8. COLD COMPACTION DIAGRAM

QMV Powder T=298 K



ISOSTATIC PRESSING OF BERYLLIUM POWDER INTO NEAR NET SHAPES

J. L. Krankota, RFP

Abstract

Beryllium powder was hot isostatically pressed into hemishells using double-walled steel cans. Two methods of producing the hemishells were investigated: 1) hot isostatically pressing (HIP) of cold isostatically pressed (CIP) preforms and 2) direct HIP of loose-packed powder. Stainless steel cans were used initially but could not be easily separated from the beryllium. Mild steel cans were subsequently used and were found to be easily removed by dissolution in 10 vol % nitric acid, with no attack on the beryllium. A finite element computer simulation was employed to help solve a buckling problem in the outer wall of the cans. Buckling of the can was eliminated by thickening the outer walls. Finite element predictions were in agreement with actual results. Vacuum outgassing for more than 8 hours at 650°C was required to produce microstructures with very little residual gas contamination, revealed by thermal-induced-porosity tests. Thin hemishells (1/4 inch thick or less) cracked during processing. A thicker hemishells (3/8 inch) directly hot isostatically pressed from S200F powder did not crack and was machined into a finished part. The mechanical properties of this part met the specification for high strength beryllium product. Little distortion of the steel cans supported in graphite collars occurred during HIP. Since little advantage of cold isostatically pressing powder into preforms was realized, it appears that direct HIP is the recommended process for fabricating beryllium hemishells from powder.

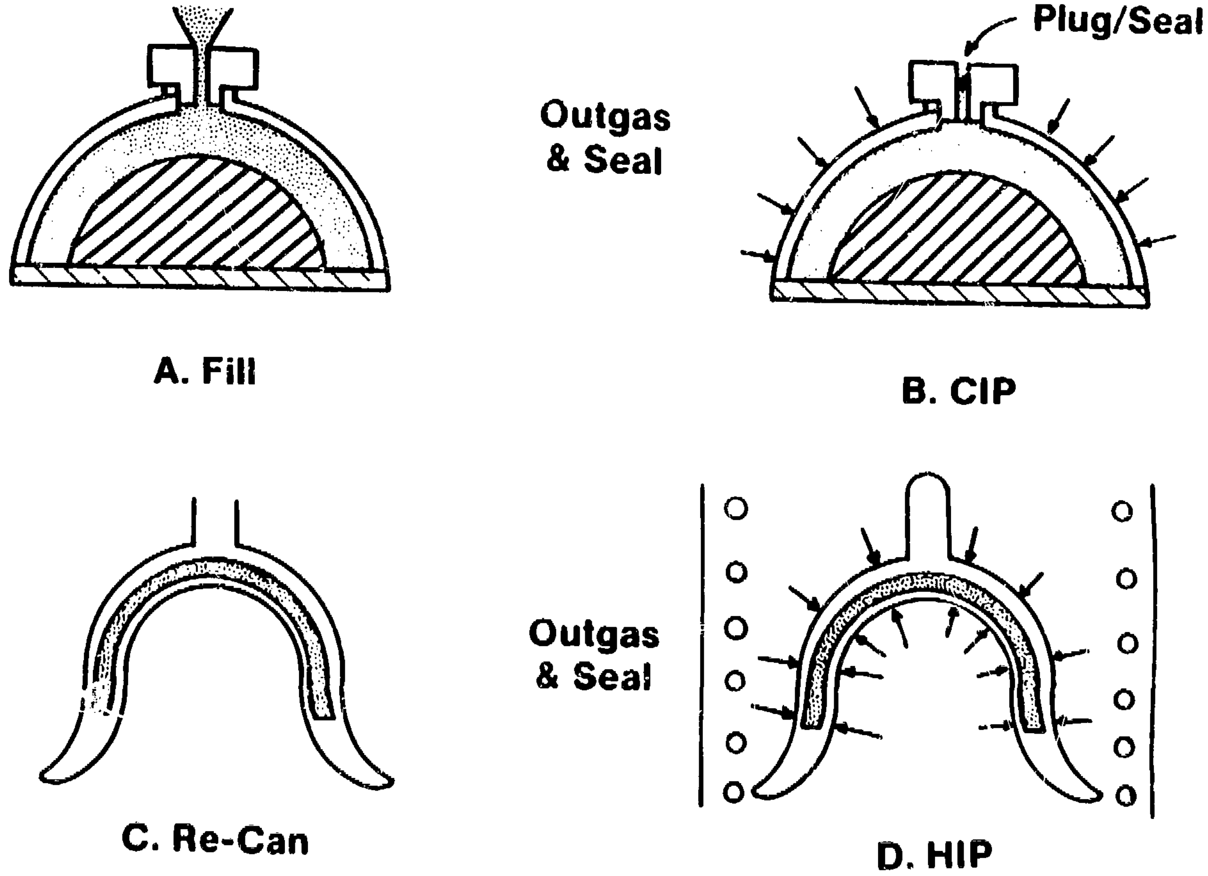
Objective

- Experimental approach
  - Process schematics
  - Outgas and HIP parameters
  - Characterization
- Experimental results
  - Design specifics
  - FEA
  - Dimensional analysis
  - Properties
- Material utilization
- Summary/conclusions
- Future work

Objective

Examine the feasibility of direct HIP and CIP/HIP processing for producing WR components

CIP/HIP Process



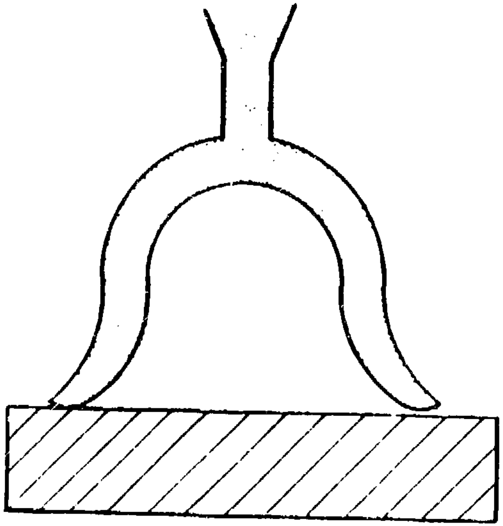
A. Fill

B. CIP

C. Re-Can

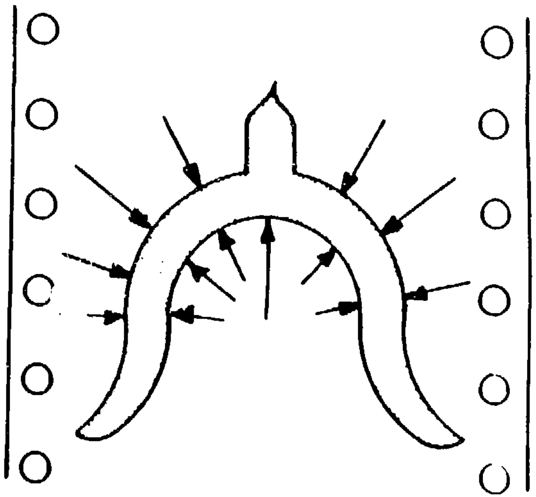
D. HIP

Direct HIP Process



Shaker Table
A. Fill Container

Outgas
& Seal



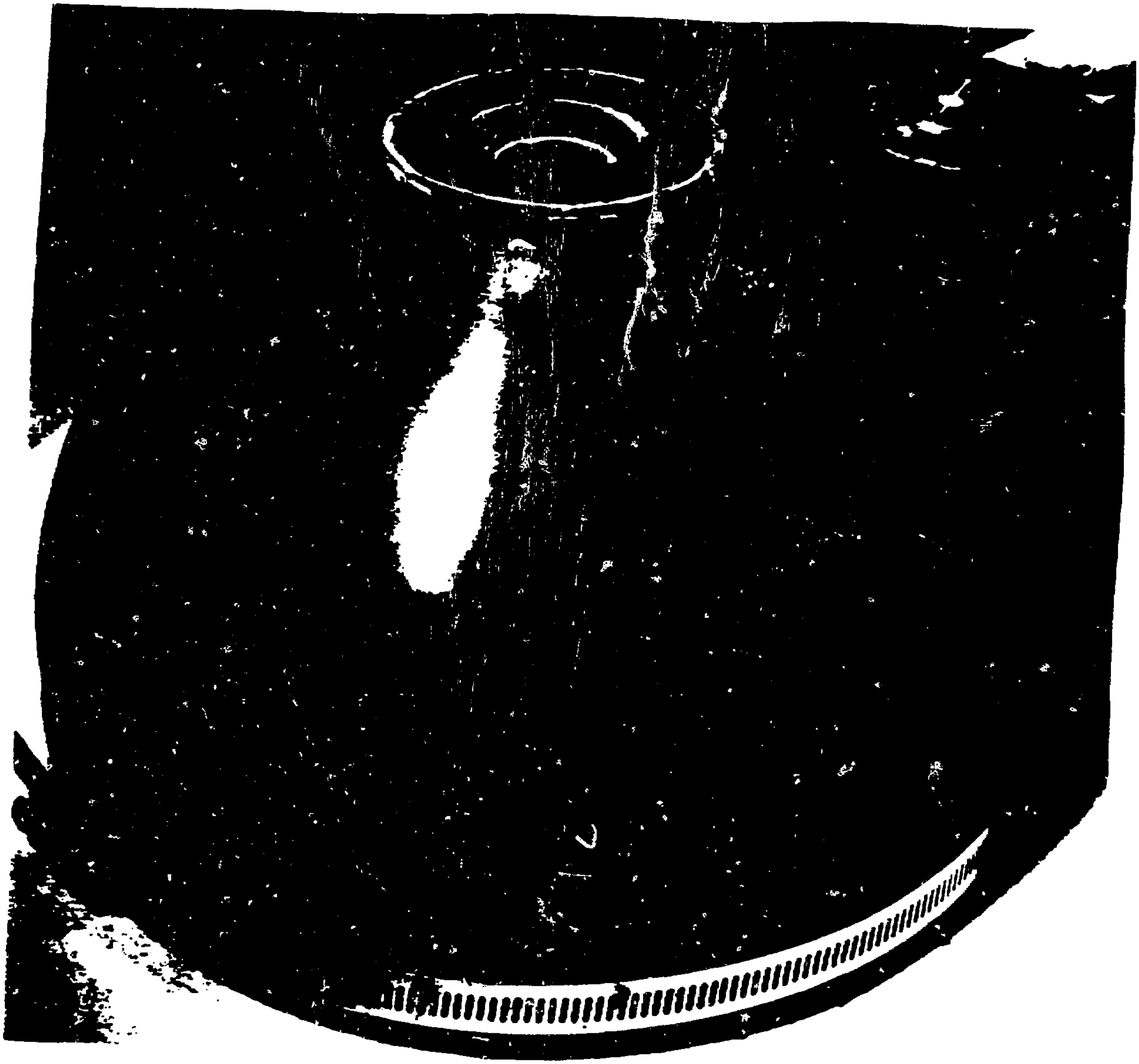
B. Hot Isostatically
Press

Outgas and HIP Parameters

- Outgas
  - Time - 1-20 hours
  - Temperature - 650° C
- HIP
  - Time - 4 hours
  - Temperature - 1000° C
  - Pressure - 15,000 psi

Characterization

- Dimensional control
- Properties
    - Physical
    - Mechanical
    - Microstructural
- T.I.P. response
- Finite Element Analysis (FEA)



Photograph of polyurethane hemishell CIP bag on aluminum mandrel with filter on bottom of plug seal

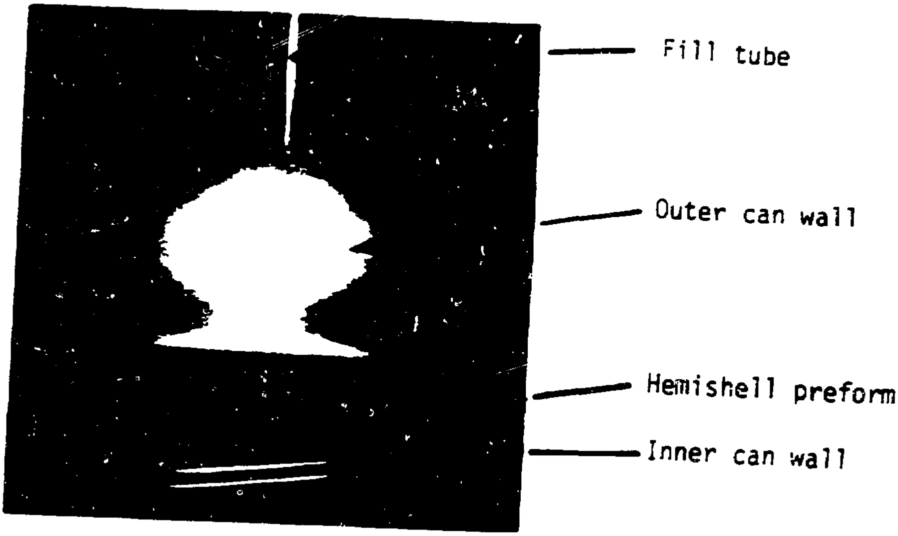
Inside of shell

Outside of shell

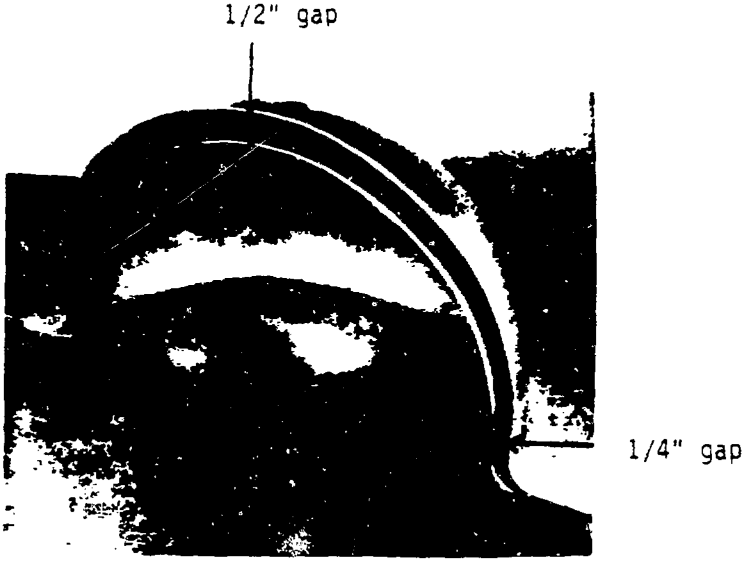
Aluminum mandrel: note Be powder which stuck to polar region of mandrel

Photographs of beryllium preform and the aluminum mandrel on which it was CIPed.

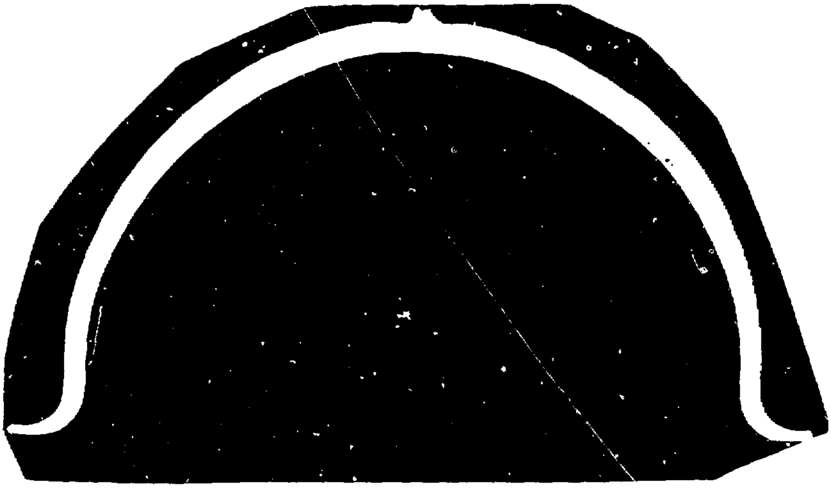
CIPED POWDER PREFORM IN HIP CAN



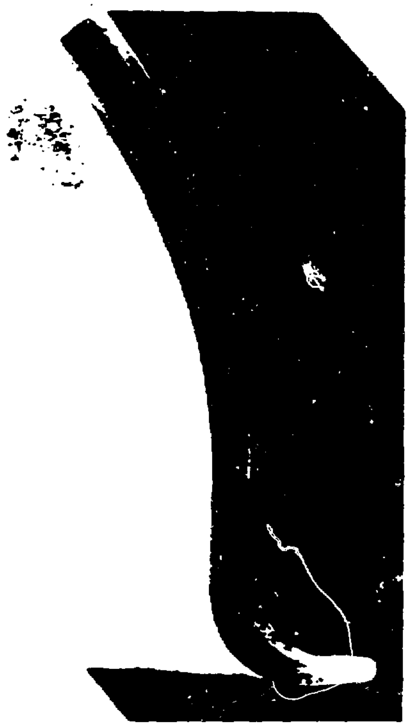
Photograph of cold isostatically pressed powder preform inserted between can walls prior to brim welding.



Photograph of cut section through large hemishell can before filling with powder. Inner can wall has a 4-inch radius and outer can wall has a 4-1/4 inch radius.

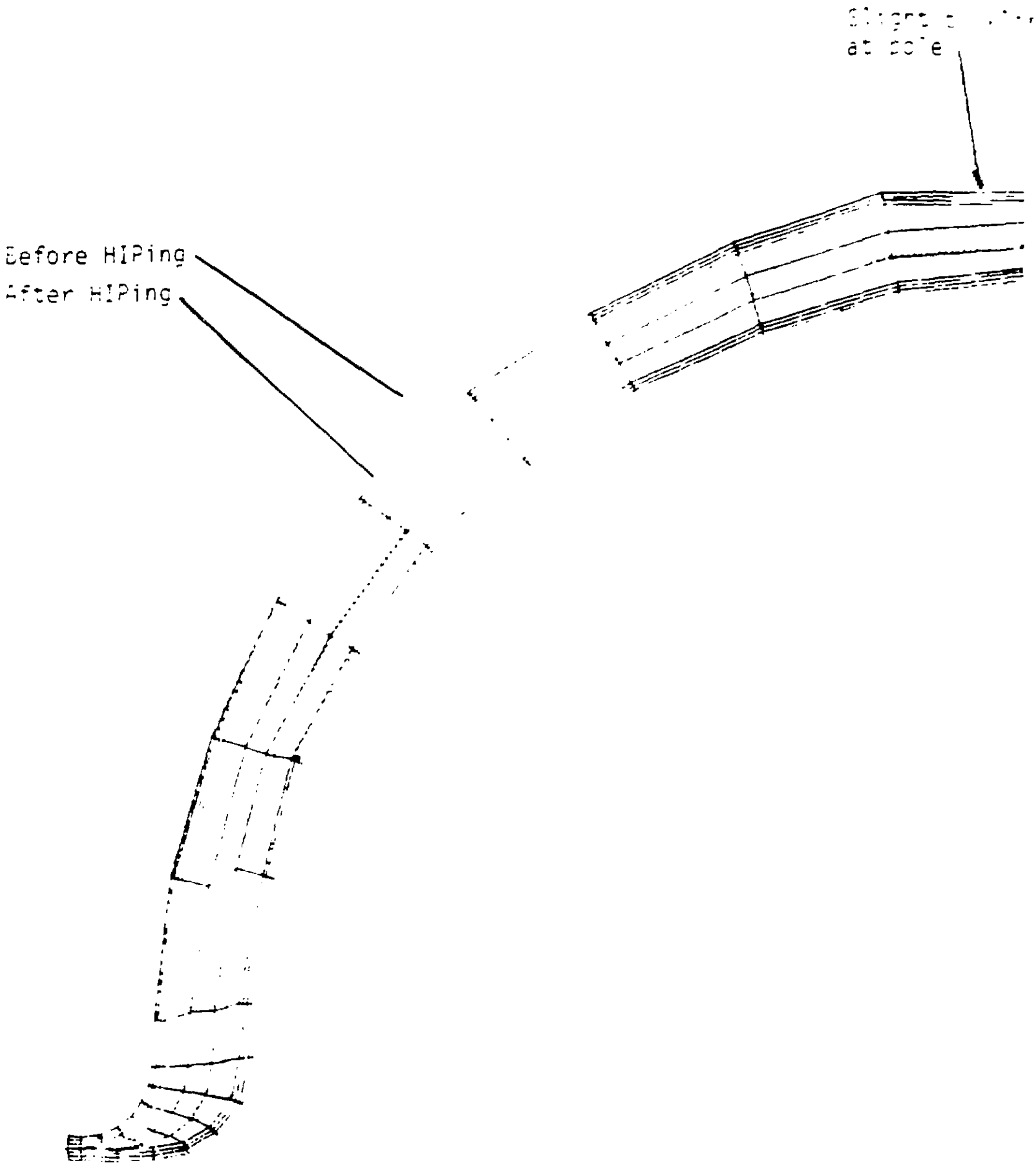


Full half section of hemishell before removal of SS can

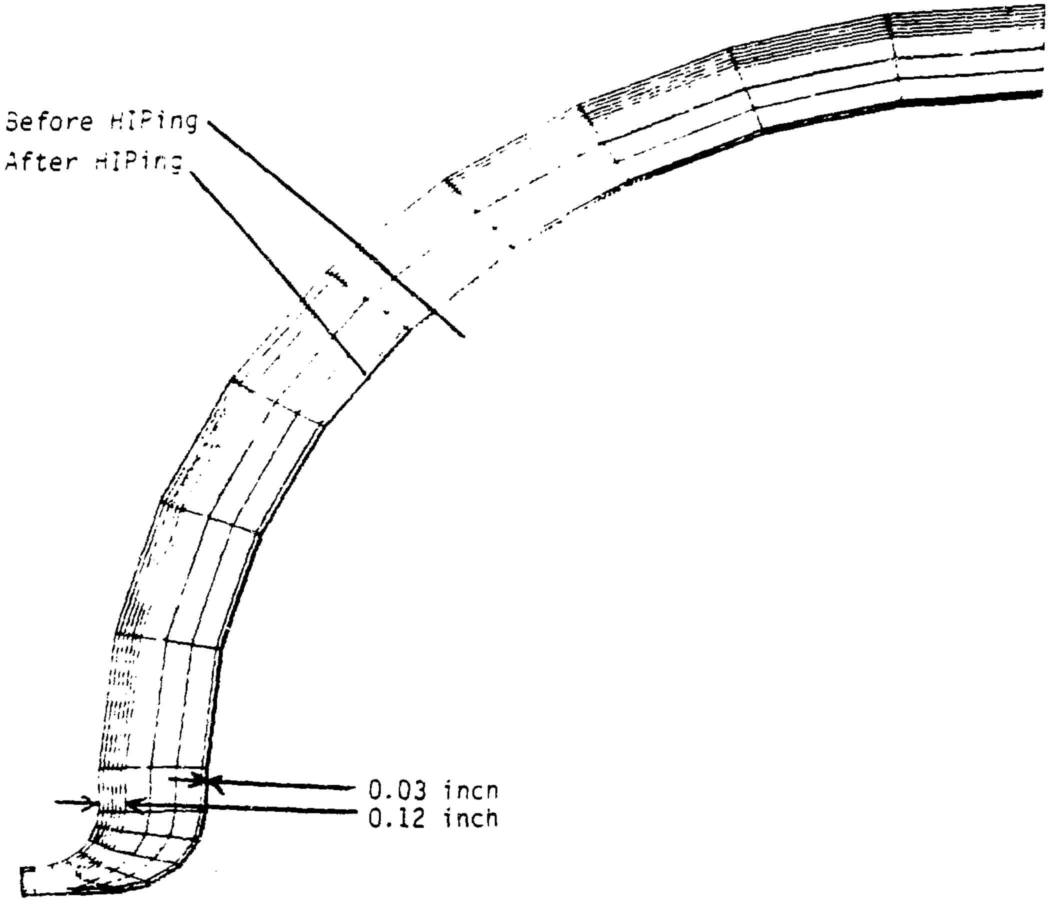


Closeup of waist/brim area

Photographs of large hemishell directly HIPed from beryllium powder (Direct HIP-1)



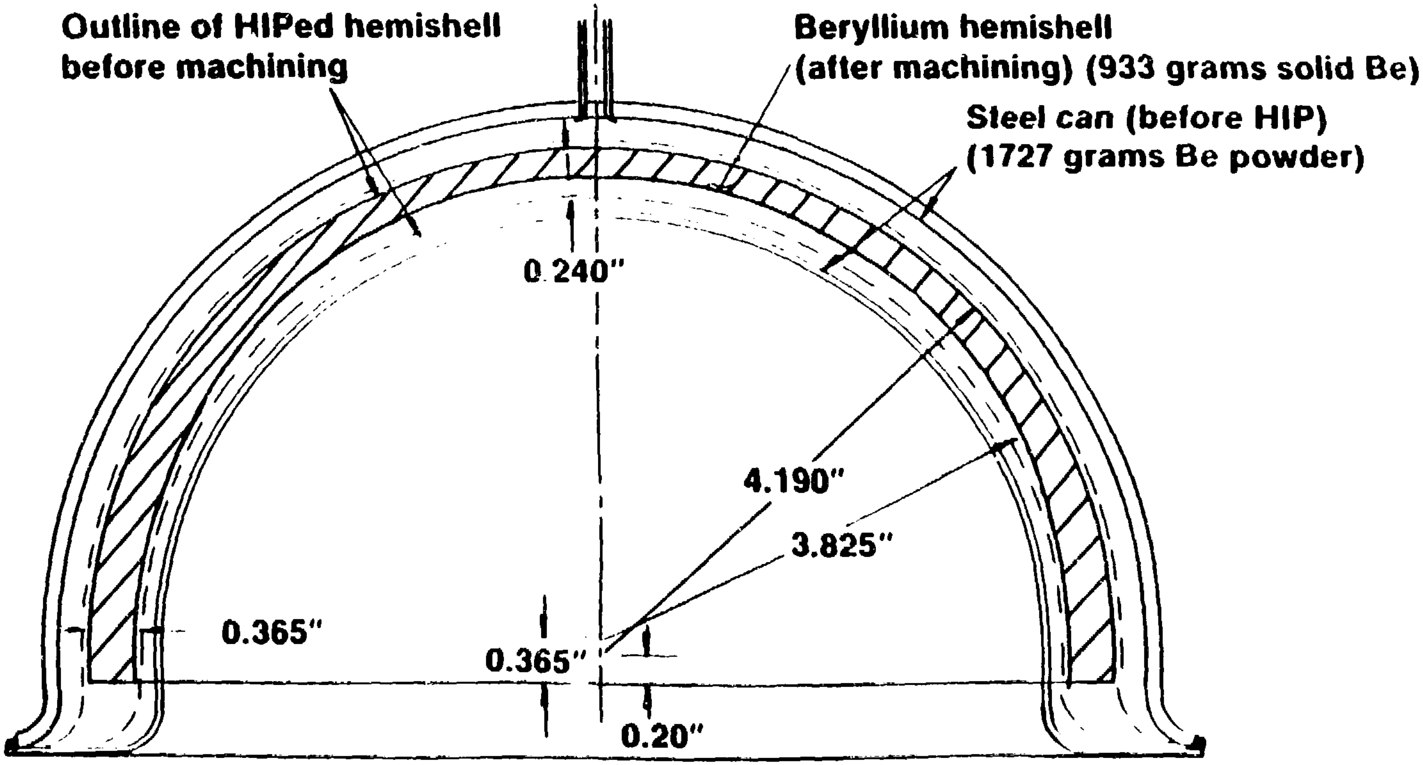
Plots of large hemishell with same outer and inner wall thickness before and after a simulated HIP cycle on the finite element model



Plots of large hemishell with thick outer wall and thin inner wall before and after a simulated HIP cycle on the finite element model

Rockwell International

Diagram of Profile of Direct HIP-4 Beryllium Hemishell Before and After HIPing in Steel Hemican and Finish Machining



Rockwell International

Composition and Density of HIPed Beryllium Hemishells

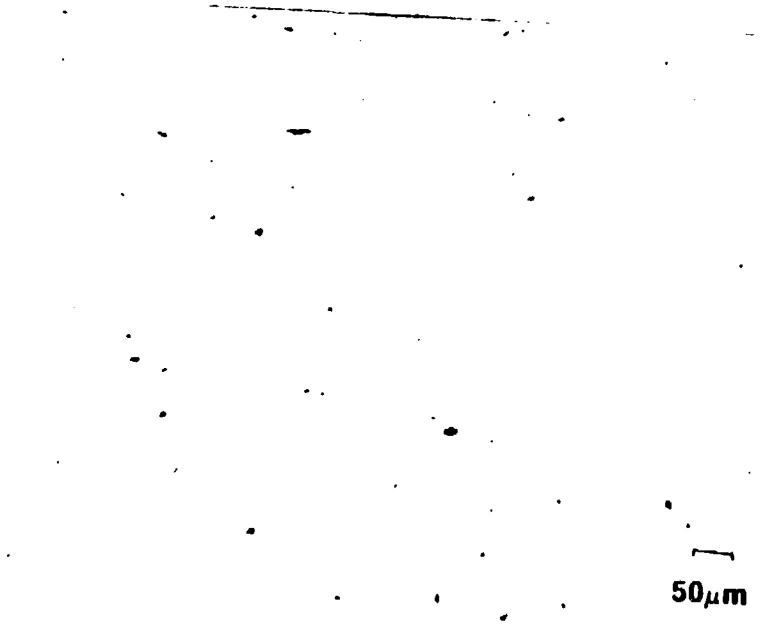
Part	Iron (ppm)	Aluminum (ppm)	BEO (Wt%)	Density (gm/cm³)
SP-200F Powder	800	300	0.7	—
Direct HIP-4	855	305	1.10	1.8536
CIP/HIP-4	1005	325	1.12	1.8508
WR Specifications				
RM255116 (A)	1200	700	0.9	1.8534
RM255116 (B)	1800	1600	2.0	1.8620
RM253939	1000	600	1.2	1.8557

Rockwell International

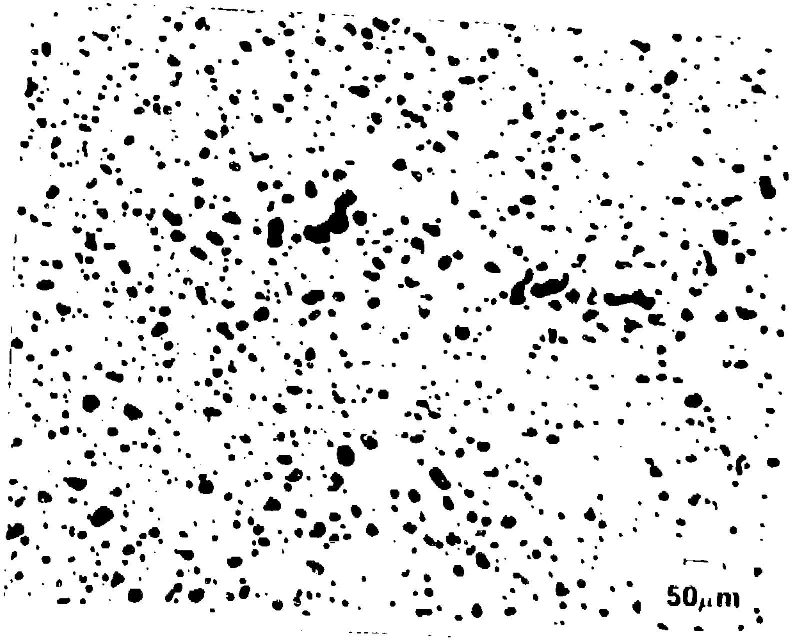
Mechanical Properties of HIPed Beryllium Hemishells

Part	Yield Strength (ksi)	Ult. Tens. Strength (ksi)	% Elong.
Direct HIP-4	48 (max) 38 (min)	69 (max) 61 (min)	5.2 (max) 3.2 (min)
CIP/HIP-4	42 (max) 38 (min)	61 (max) 58 (min)	5.4 (max) 3.4 (min)
WR Specifications			
RM253939	50 (max) 35 (min)	— 50 (min)	3.0 T&L
RM255116 (A)	38 (max) 25 (min)	— 35 (min)	2.0 T/1.0 L
RM255116 (B)	25 (min)	35 (min)	1.0 T&L

Thermally Induced Porosity/Test in Beryllium
(T.I.P.)



Direct HIP-4
20 Hours



CIP/HIP-4
1 Hour

100X

Material Utilization

- Over 50% recovery with HIP
- 28-30% recovery with current processing

Summary/Conclusions

- Good shape control with direct HIP
- No buckling with thicker outer can wall
- Mild steel can easy to remove with nitric acid
- Properties meet high strength Be specification
- T.I.P. revealed porosity and insufficient outgassing
- FEA is a useful tool

Future Work

- Make WR component
- Examine HDI issue
- Further develop FEA

EVALUATION OF THE STRENGTH OF GRAPHITE DIES USED IN THE HOT PRESSING OF BERYLLIUM BILLETS

by

D. Roberts (AWRE)

Summary

Graphite tooling is used for the hot pressing of beryllium billets. The load applied during a typical pressing operation, gives a calculated die hoop stress approaching the UTS of the graphite used for the tooling. Despite this fact, the incidence of die failure is low. It was thought that the development of near net shape pressing techniques, would require hot pressing loads higher than those used for typical billets. In view of the die hoop stresses produced by the higher pressing loads, a preliminary investigation was undertaken to determine the bursting strength of graphite dies. It was found that depending upon the pressing temperature and billet aspect ratio, failure occured at calculated hoop stresses at least twice the UTS of the graphite. It was concluded that the actual hoop stresses were less than the calculated values as a result of the applied load being attenuated by die friction effects.

EVALUATION of THE STRENGTH OF GRAPHITE DIES

SLIDE 1

AWE and ROF Cardiff use graphite dies for the hot pressing of beryllium powder. The reasons for using graphite are availability compatibility machinability and cost. The strength of the graphite used CS is low

SLIDE 2

Experience of hot pressing has shown that dies made from the CS graphite give satisfactory service and did not fail by bursting Calculation by lame formula show that the hoop stresses induced in the dies by an applied load of about 0.5 tpi are about half the ultimate strength of the graphite. At such a high hoop stress some die failures could be expected in a production run

SLIDE 3
SLIDE 3A

The possible reason for the absence of failure is that some of the applied load is attenuated by die or powder friction

SLIDE 4

A similar effect was reported by Bustamante in about 1960 in the cold pressing of metal powders in steel dies. He showed that the measured strain in a cold pressing die varied from half to a fifth of the predicted value.

Assuming a similar effect for the hot pressing of beryllium , and the confidence that had been built up absence of die failure , the use of dies at the high apparent working hoop stresses was acceptable.

In developing improved net shaping and possibly in using CIPed preforms the applied load of 0.5 tpi may have to be increased . If it was increased to 1tpi then the calculated hoop stresses in a die would be higher than the strength of graphite. This would appear unrealistic and there would be little confidence in carrying out any development work involving this loading.

What was required was to show that a similar attenuation effect occurred in beryllium hot pressing and to measure the amount of attenuation.

SLIDE 5

Measuring the strain in graphite dies at about 1100 C would be difficult. What was required was an estimate of the applied load at which a die would fail. This could be easily measured by increasing the applied load on a die until failure occurred. Using lame formula the calculated hoop stress at failure could be calculated from the applied load at failure . The differance betwern the calculated strength and the tensile strength of graphite would be a measure of the attenuation of the applied load in the die

To carry out this work a double acting die was designed and manufactured at various wall thickness

SLIDE 6

The die was manufactured at 75mm id and 112 125 150 and 200 mm od. Applying lame formula to the dies the induced hoop stress in each case would exceed the applied pressure by 2.5 2.13 1.67 and 1.33 respectively.

SLIDE 7

Twenty dies were burst tested under various conditions of temperature and powder fill height and powder

The results are shown in

SLIDE 8

SLIDE 9

and summarised later

Considering first
Lame formula

SLIDE 10

The formula applies to a infinite long cylinder under either internal or external fluid pressure such that the applied pressure is equal to the internal pressure and to the internal radial pressure.

In powder pressing
Considering the die as a cylinder
The cylinder is only stressed or strained over a short length about its centre. The unstrained wall area will give some support to the stressed area and therefore reduce the overall strain or stress in the wall.

SLIDE 11

Secondly the radial pressure seen by the wall is derived from the applied pressure (applied load/ die area) after transmission through both the plunger and billet. In transmission the forces must be attenuated by both plunger / die, plunger/ powder, and powder friction.

Considering the results summarised in

SLIDE12

The first result Die stresses with water. Failure occured at a hoop stress equal to the quoted tensile strength of graphite.Showing no stress attenuation. In this case the billet or charge aspect ratio is uncertain as the the billet was made up of a sandwich of water between two rubber end plugs.

SLIDE 13

All the other results show attenuation of stress when the calculated hoop stress at temperature is compared with the room temperature strength of graphite.
The variation can be considered under three effects

ONE Variation with billet aspect ratio

SLIDE 14

There appears to be two relations
First a rapid increase in attenuation as the aspect ratio of the billet decreased below 0.5 or as billet height decreases
Secondly a slow decrease in attenuation as aspect ratio increases above 0.5
Both effects are the result of friction

At low aspect ratio the friction between plunger faces and powder or billet restricts the outward flow of either powder or billet and therefor the radial stress on die wall
As the billet aspect ratio increase the effect of plunger face friction remains constant, but the billet or powder column will tend to barrel out increasing wall stress

At an aspect ratio of about 2 the attenuation of stress would be about 2. As a in powder pressing billet aspect ratio is limited to about 2. it could be considered that in powder pressing the minimum attenuation is about 2.

The second effect is that of die wall thickness

SLIDE 15

This is again as expected from the original consideration
In this case very few results were available and to investigate the effect fully work would be required at various die diameter at constant billet aspect ratio.

The third effect noticed is that of pressing temperature on the attenuation ratio.

SLIDE 16

At medium billet aspect ratio
beryllium at 1100 attenuates stress at about 2

copper at 1000 attenuated stress at about 2

Beryllium at 900 and 750 attenuate stress at about 4 6

Alumina at 1100 attenuates by about 4---6

In the case of beryllium at 1100 and copper at 1000 the billets would be fully dense but the remainder would be about 80 to 90% dense

The results are similar to the work of Bustamante who showed that at room temperature lead attenuated the die strain by 2 but other metals attenuated strain by about 4

Again lead would be expected to be 100% dense and the other metals about 80%

SLIDE 17

As explained earlier EC 2 graphite is not use for dies some tests were carried out on EC2 dies. In the first test compression failure of the plunger occurred without any damage to the die body. AS the compressive strength of graphite is higher than its tensile strength and in the die under test the die geometry would intensify (Lame Formula) the applied pressure by about 1.3. Then the plunger failure before die failure indicates a n attenuation of about 1.6 * 1.3*2= 2.6

The other EC2 results when expressed as attenuation appear to agree with the CS graphite results

The dies all appeared to fail by longitudinal cracking with the broken fragments left standing after the failure.

SLIDE 18
SLIDE 19
SLIDE 20

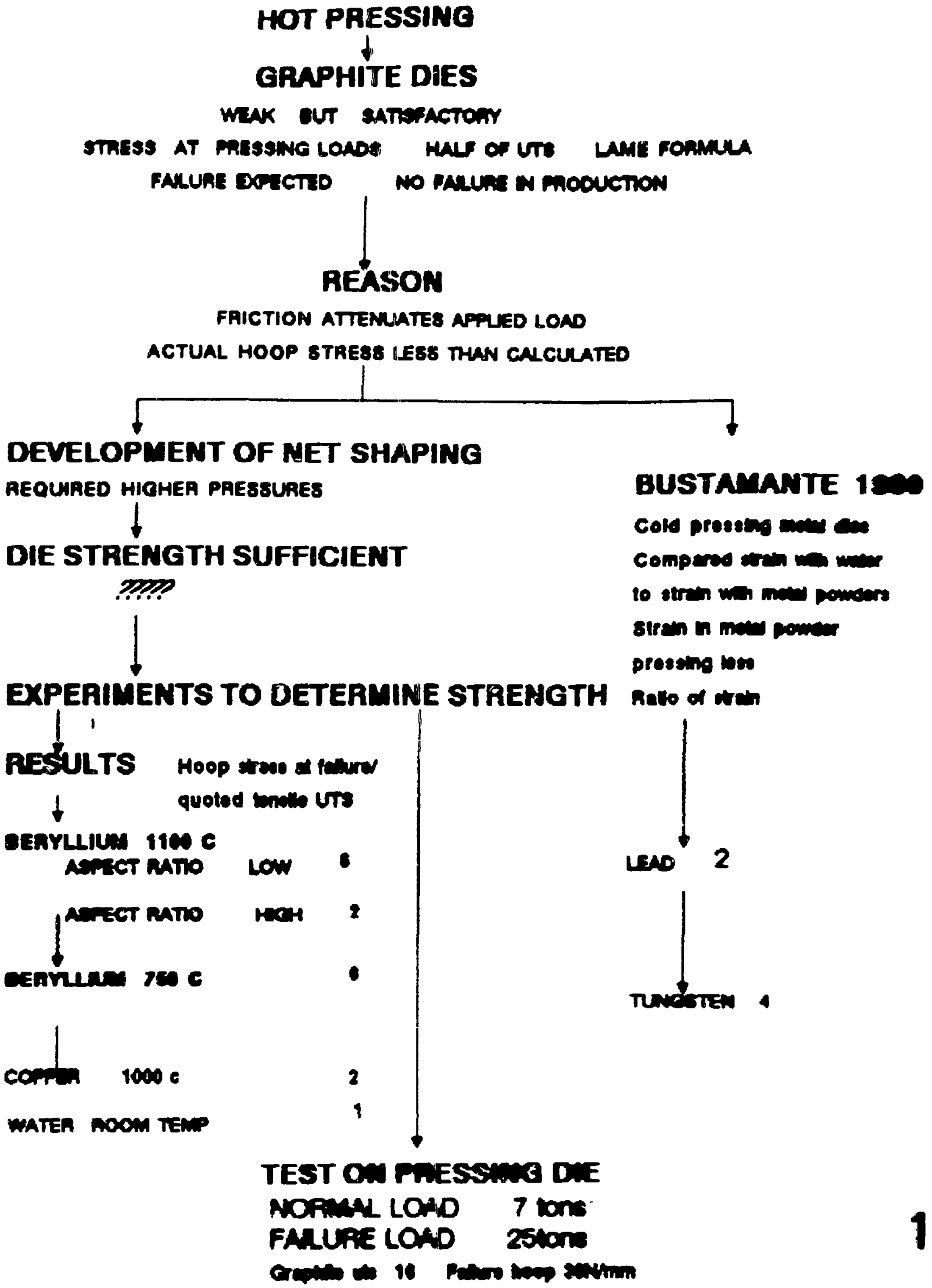
Assuming a similar failure in a production die then there would be little damage to furnace provided some clearance was allowed between die and furnace body.

SLIDE 21

TO GAIN FULL CONFIDENCE IN THE EXPERIMENTAL RESULTS A 110MM DIAMETER PREFORM DIE WAS TESTED TO DESTRUCTION THE RESULTS GIVE AN ATTENUATION RATIO OF ABOUT 2

SLIDE I

EVALUATION OF THE STRENGTH OF GRAPHITE DIES USED IN THE HOT PRESSING OF BERYLLIUM BILLETS



HOT PRESSING

GRAPHITE DIES

WEAK BUT SATISFACTORY

STRESS AT PRESSING LOADS HALF OF UTS LAME FORMULA

FAILURE EXPECTED NO FAILURE IN PRODUCTION

REASON

FRICTION ATTENUATES APPLIED LOAD

ACTUAL HOOP STRESS LESS THAN CALCULATED

DEVELOPMENT OF NET SHAPING

REQUIRED HIGHER PRESSURES

DIE STRENGTH SUFFICIENT

?????

EXPERIMENTS TO DETERMINE STRENGTH

RESULTS

Hoop stress at failure/
quoted tensile UTS

BERYLLIUM 1100 C
ASPECT RATIO LOW 6
ASPECT RATIO HIGH 2
BERYLLIUM 750 C 6
COPPER 1000 c 2
WATER ROOM TEMP 1

BUSTAMANTE 1[illegible]

Cold pressing metal dies
Compared strain with water
to strain with metal powders
Strain in metal powder
pressing less
Ratio of strain

LEAD 2

TUNGSTEN 4

TEST ON PRESSING DIE

NORMAL LOAD 7 tons
FAILURE LOAD 25tons
Graphite uts 18 Failure hoop 30N/mm

1

SLIDE 2

CS graphite	UTS	Compressive	32 N/mm^2
		Tensile	15--20 N/mm^2
		Flecture	15 N/mm^2
		Density	1.70 (Perosity 23%)
EC 2 Graphite	UTS	Compressive	67.0 N/mm^2
		Tensile	27.0 N/mm^2
		Flecture	34.0 N/mm^{2}n
		Density	1.77 (Perosity 17%)

TABLE

Manufacturers quoted strength values for Graphite used for the manufacture of hot pressing dies for beryllium at AWRE.

2

SLIDE 3

Die od mm	Die id mm	base shape	powder weight Kg	aspect ratio	applied load tons	applied press N/mm^2	hoop factor	calc hoop stress	[illegible] hoop/ UTS
500	370	billet	10Kg	0.14	30t	2.78	3.42	9.0	.6-.45
500	240	near net	4Kg	0.17	40t	8.81	1.58	14.0	.93--.7
500	175	near net	2.6Kg	0.36	30t	12.43	1.28	12.8	.85--.64
500	300	billet	22kg	0.6	30t	4.23	2.13	9.1	.61--.46
300	75	billet	400g	0.66	7.0t	15.79	1.15	17.6	1.17--.88
300	108	near net	750g	0.2	7	7.61	1.35	10.4	.69--.52
					14.0	15.32	1.35	20.8	1.39--1.04

TABLE 3

Typical values of applied loads used in the hot pressing of beryllium preforms at 1100° C in graphite dies

SLIDE #3A

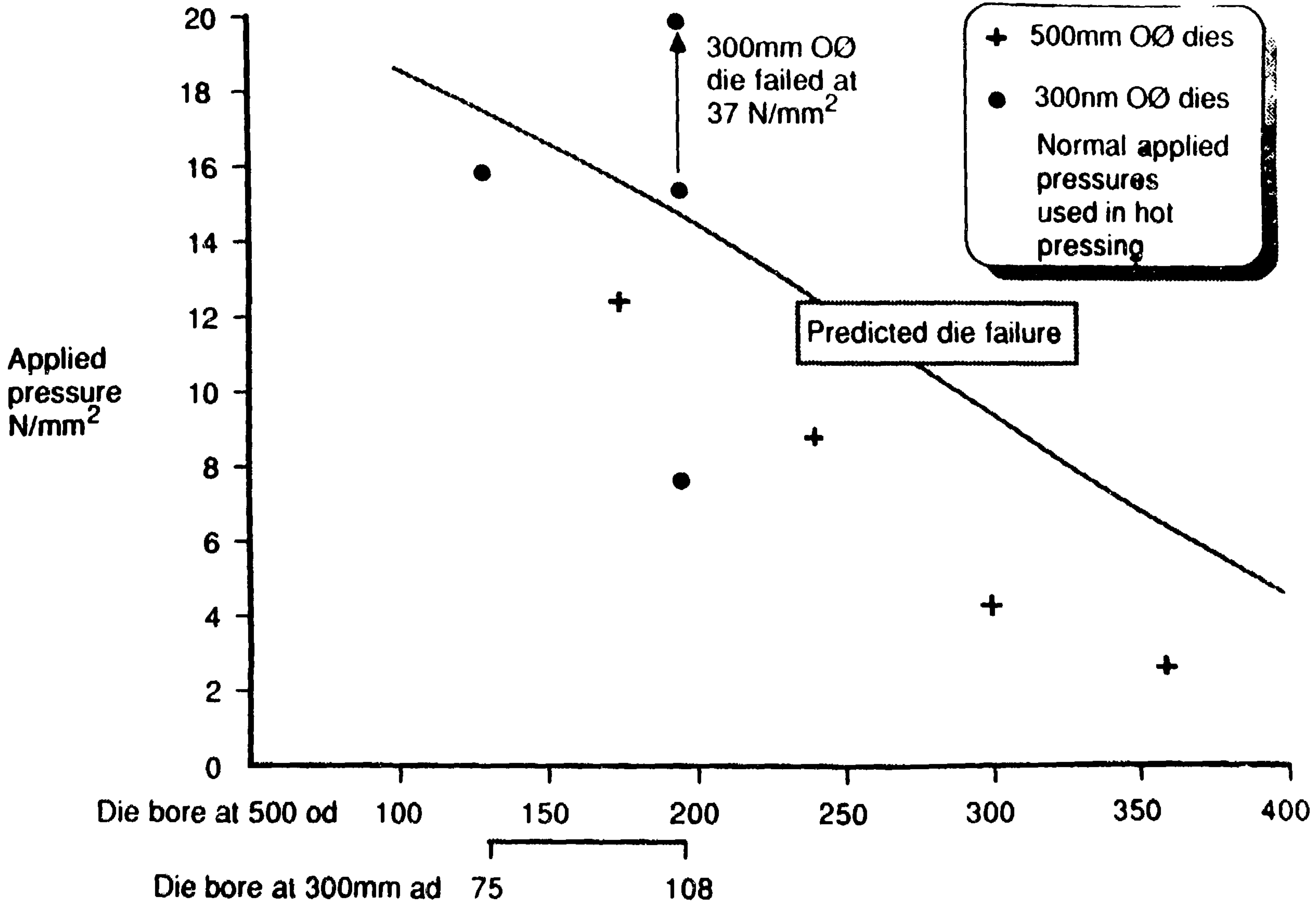


FIGURE 3a PRESSURES APPLIED IN DIE HOT PRESSING OF BERYLLIUM AT 1100°C
Line shows applied pressure predicted to burst a die

SLIDE 34

EVALUATION OF THE STRENGTH OF GRAPHITE DIES USED IN THE HOT PRESSING OF BERYLLIUM POWDER

GRAPHITE DIES

REASON AVAILABLE
COMPATIBLE
COST

WEAK

SATISFACTORY

IN USE NO FAILURE BY BURSTING

LAME FORMULA

HOOP STRESS = $\frac{\text{INTERNAL PRESSURE} * (R*R + r*r)}{(R*R - r*r)}$

FRICTION ATTENUATES STRAIN TO ORDER 2---6 IN COLD PRESSING (BUSTAMANTE 1960)

NET SHAPING DEVELOPMENT

HIGHER APPLIED LOADS POSSIBLE

CURRENT APPLIED LOADS OVER HALF CALCULATED DIE BURSTING LOAD

4

3L17C 5

EVALUATION OF THE STRENGTH OF GRAPHITE DIES USED IN THE HOT PRESSING OF BERYLLIUM POWDER

PROBLEM

DETERMINE STRAIN or STRESS in HOTPRESSING D

↓

STRAIN DETERMINATION
DIFFICULT
TEMPERATURE 1100 C

↓

REQUIRED

ESTIMATE OF LOAD AT WHICH
DIES WILL FAIL

↓

METHOD

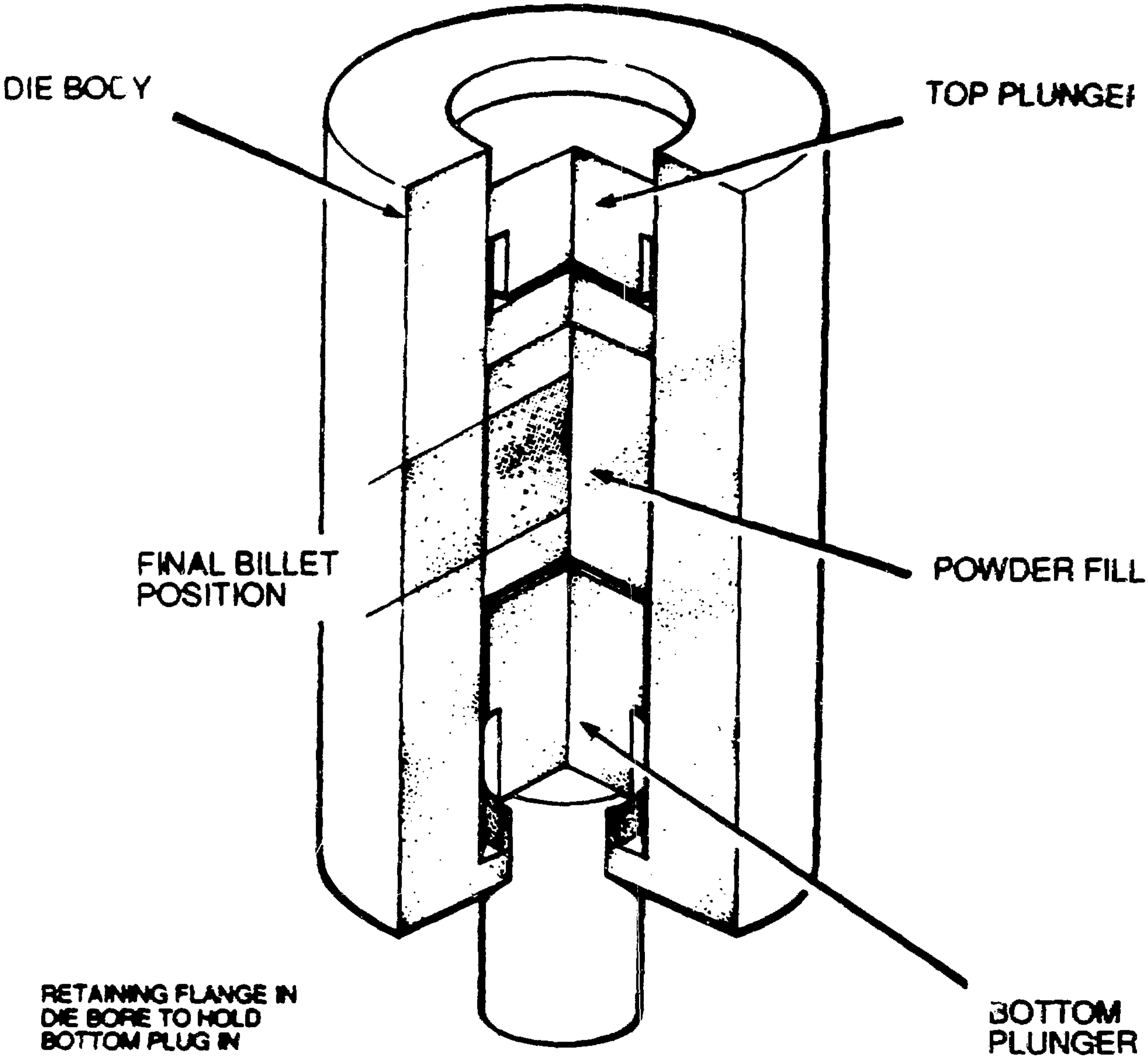
BURST A DIE BY INCREASING LOAD
UNTIL DIE FAILS

↓

DESIGN

DOUBLE ACTING DIE

SLIDE 6



F

DIE DESIGNED TO EVALUATE HOOP STRESSES GENERATED IN GRAPHITE DIES USED IN THE HOT PRESSING OF BERYLLIUM POWDER

6

SLIDE 7

DIE OD mm	DIE ID mm	HOOP STRESS INTENSITY factor
200	75	1.33
150	75	1.67
125	75	2.13
115	75	2.5

FORMULA

Hoop stress = Applied pressure * (k+1) / (k-1)

Where $k = (R/r)^2$.

Letting

(k+1) / (k-1) = hoop stress intensity factor

Then

Hoop stress = applied pressure * stress intensity fac

Determination of hoop stress of experimental dies used

SLIDE. 8

			At failure		
Powder &test temp. °C	die od mm	die id mm	applied load tons	applied pressure N/mm^2	hoop stress N/mm^2
water room temp	125	75	3.5	7.81	16.5
Be 1100	200	75	19.5	44	58
1100	150	75	12.5	28	47
1100	125	75	8	18	38
1100	115	75	6.5	14.5	36
760	125	75	19.0	42.9	91.1
Cu 980	125	75	8	18	36
Alumina 1100	125	75	18.0	40.6	86.3

Experimental results CS graphite dies at constant billet aspect ratio H/D (Billet ht/ Billet diameter)of 0.49 ie 0.5

SLIDE 9

powder weight gms	billet aspect ratio	At failure		
		applied load tons	applied pressure N/mm^2	induced hoop stress N/mm^2
900	1.47	6.5	14.66	24.5
600	.98	6.75	15.22	25.4
300	.49	8	18.04	38.4
200	.32	11	24.81	62.0
100	.16	20	45.11	96.1

CS GRAPHITE

Effect of Billet Aspect Ratio on Die Hoop Failure Stress

Die diameter 75mm id 125mm od pressed at 1100^{0}C

Aspect ratio = billet height/ billet diameter

9

SLIDE 10

EVALUATION OF THE STRENGTH OF GRAPHITE DIES USED IN THE HOT PRESSING OF BERYLLIUM POWDER

RESULTS

SUMMARISED TABLE 8

DISCUSSION

STRESS IN CYLINDER UNDER HYDRAULIC PRESSURE
LAME FORMULA

$$\text{HOOP STRESS} = \frac{\text{INTERNAL PRESSURE} \times (R \times R + r \times r}{(R \times R - r \times r)}$$

R = OUTER RADIUS r = BORE RADIUS

APPLIES WHEN CYLINDER LENGTH LONG and PRESSURE IS APPLIED BY FLUI

$$\text{IF } k = (R \times R + r \times r)/(R \times R - r \times r)$$

THEN HOOP STRESS = INTERNAL STRESS X k

POWDER PRESSING

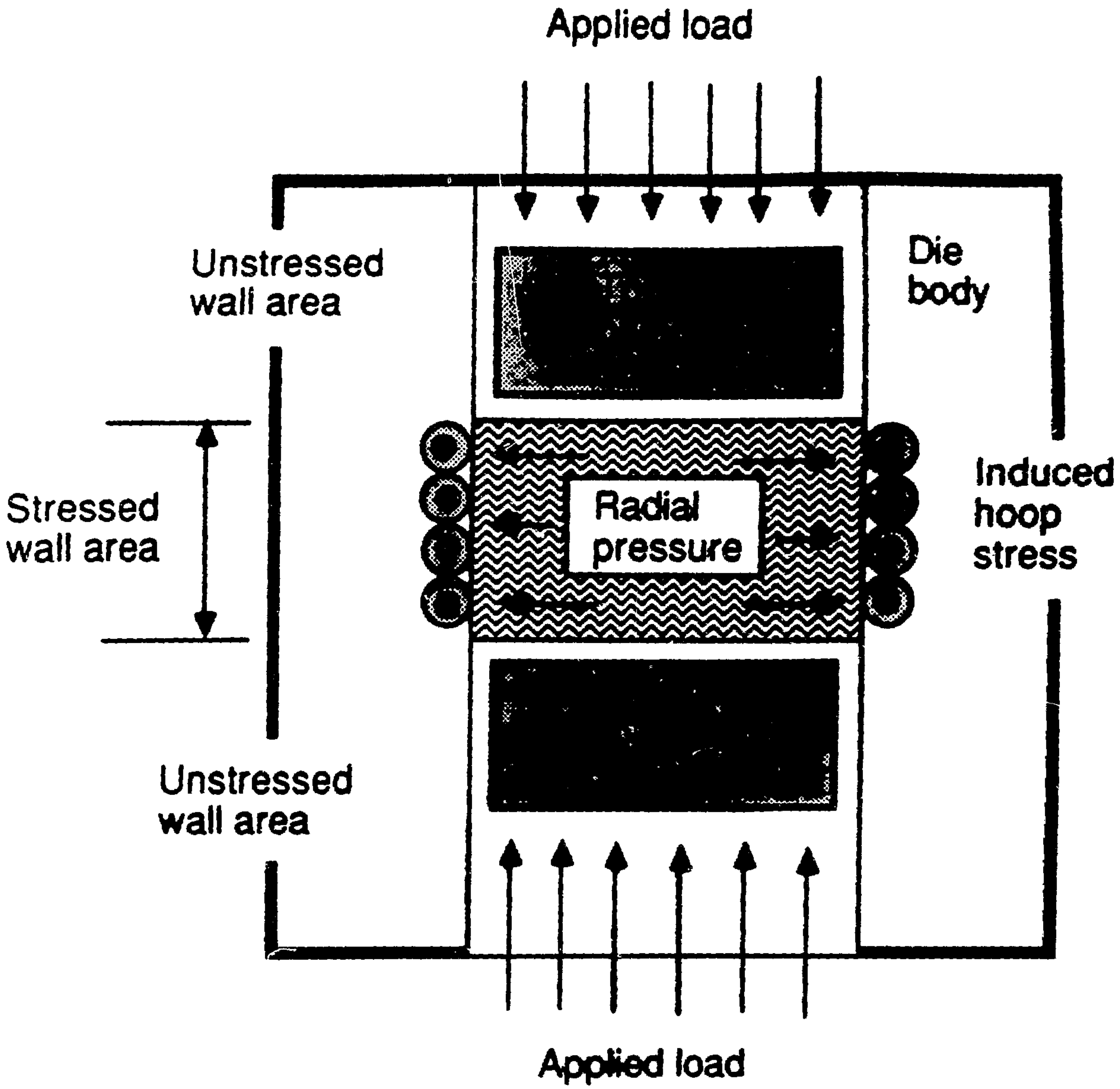
CYLINDER LENGTH SHORT PRESSURE MEDIA POWDER

FRICTION AND DIE WALL LENGTH UNDER STRESS
BOTH REDUCE EFFECTIVE PRESSURE ON DIE

=

STRESS ATTENUATION

SLIDE. 11



STRESSES IN A POWDER PRESSING DIE

SLIDE 12.

DIE MATERIAL	POWDER TEMP °C	POWDER ASPECT RATIO	DIE OUTER DIAMETER	DIE HOOP FACTOR	HOOP STRESS N/MM^2	RATIO HOOP/ TENSILE STRESS
C.S	water 20		125		16.5	1.0
C.S	Be 1100	0.49	200	1.33	58	3.9
			150	1.67	47	3.1
			125	2.13	38	2.5
			115	2.5	36	2.0
	Be 1100	1.47	125	2.13	31.10	2.1
		.98	125	2.13	32.38	2.20
		.49	125	2.13	38.4	2.6
		.32	125	2.13	62.0	4.1
		.16	125	2.13	96.1	6.4
	Be 750	0.49	125	2.13	91.1	6.1
C.S	Cu 1000	.49	125	2.13	36	2.4
			150	1.67	43.1	2.87
	A1203 1100	.49	125	2.13	91.1	6.07
E.C 2	Be 1100	.49	200	1.33	65.86	2.44 *
			150	1.67	75.18	2.79
			125	2.13	76.85	2.85
			115	2.5	64.33	2.38
E.C 2	Be 900	.49	125	2.13	124.8	4.62
	750	.49	125		115.0	4.25
	Cu 950	.49	125		55.7	2.06

Experimental results summarised and calculated hoop stress expressed as ratio of quoted room temperature tensile strength of the respective graphites.

SLIDE 13

EVALUATION OF THE STRENGTH OF GRAPHITE DIES USED IN THE HOT PRESSING OF BERYLLIUM POWDER

DISCUSSION OF RESULTS

↓

DIE PRESSURISED WATER FAILED WHEN HOOP STRESS REACHED UTS OF GRAPHITE
TABLE

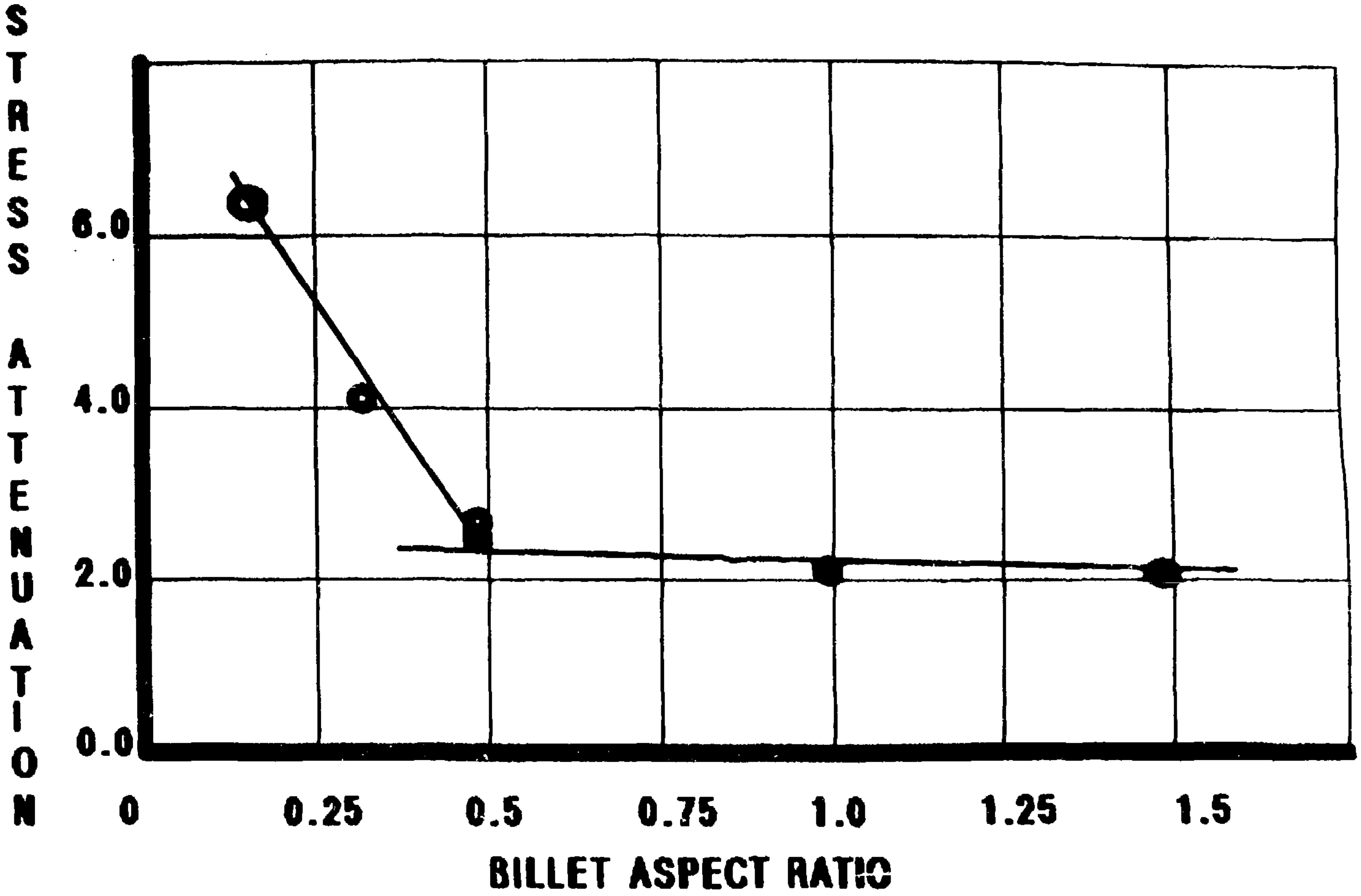
↓

IN ALL OTHERS HOOP STRESS AT BURSTING
WELL ABOVE UTS TABLE

↓

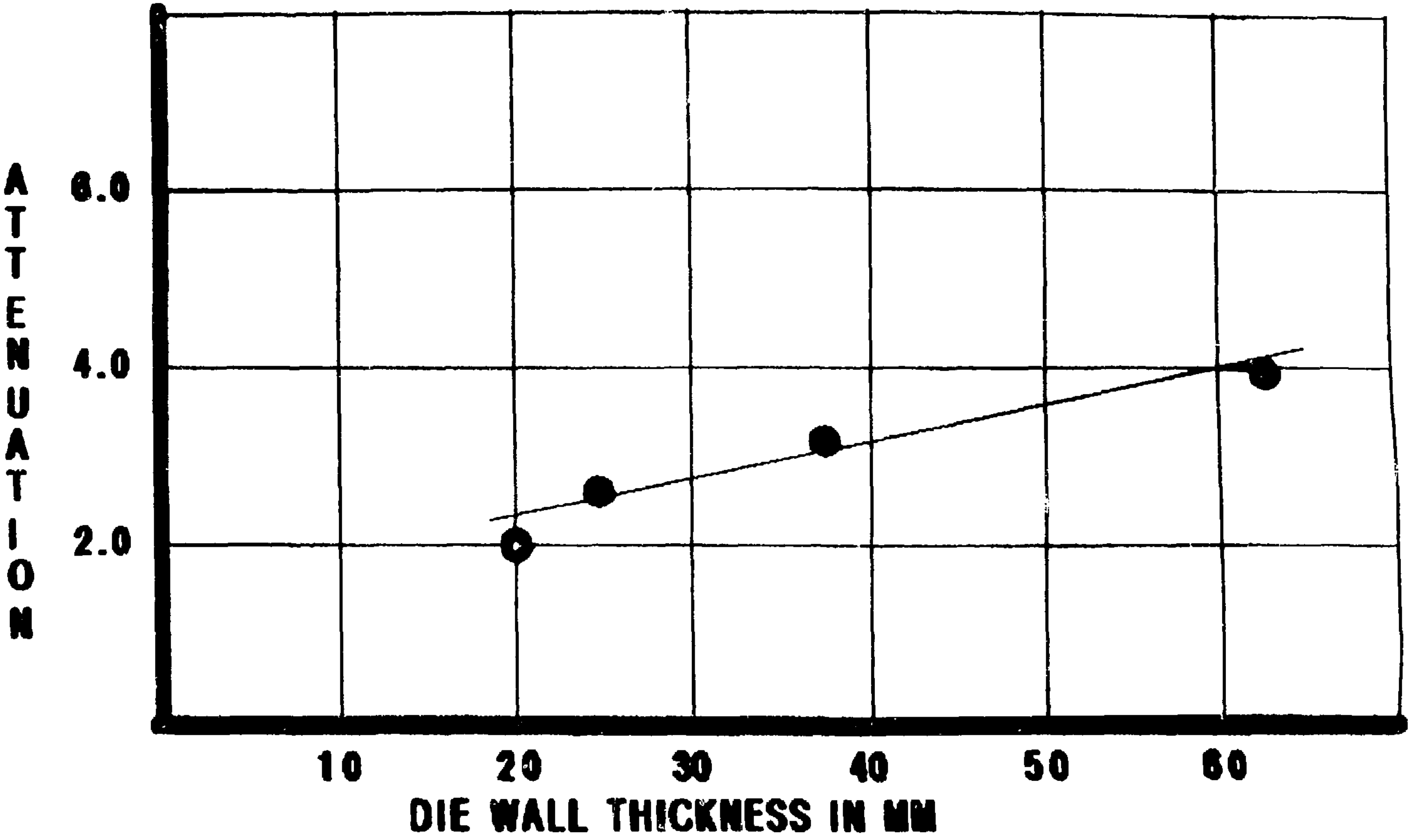
THREE EFFECTS

BILLET ASPECT RATIO FIGURE
DIE WALL TO BILLET HEIGHT FIGURE
EFFECT OF TEMPERATURE



Graph showing relation between billet aspect ratio and stress attenuation in graphite dies during the hot pressing of beryllium powder

SLIDE 15



Variation of Die hoop stress attenuation with die wall thickness at constant billet aspect ratio

EVALUATION OF THE STRENGTH OF GRAPHITE DIES USED IN THE HOT PRESSING OF BERYLLIUM POWDER

SLIDE 16

$$\text{ATTENUATION OF HOOP STRESS} = \frac{\text{CALCULATED STRENGTH OF DIE}}{\text{TENSILE STRENGTH OF GRAPHITE}}$$

BILLET MATERIAL	BILLET ASPECT	ATTENUATION	TEMPERATURE
BERYLLIUM	LOW	6	1100
BERYLLIUM	HIGH	2	1100
BERYLLIUM	MEDIUM	2.5	1100
BERYLLIUM	MEDIUM	6	750
BERYLLIUM	MEDIUM	2.5	1100
BERYLLIUM	MEDIUM	4	900
BERYLLIUM	MEDIUM	4	750
COPPER	MEDIUM	2	1000
ALUMINA	MEDIUM	6	1100

BUSTAMANTE

RESULTS

LEAD RT 2

IRON RT 4

SLIDE 17

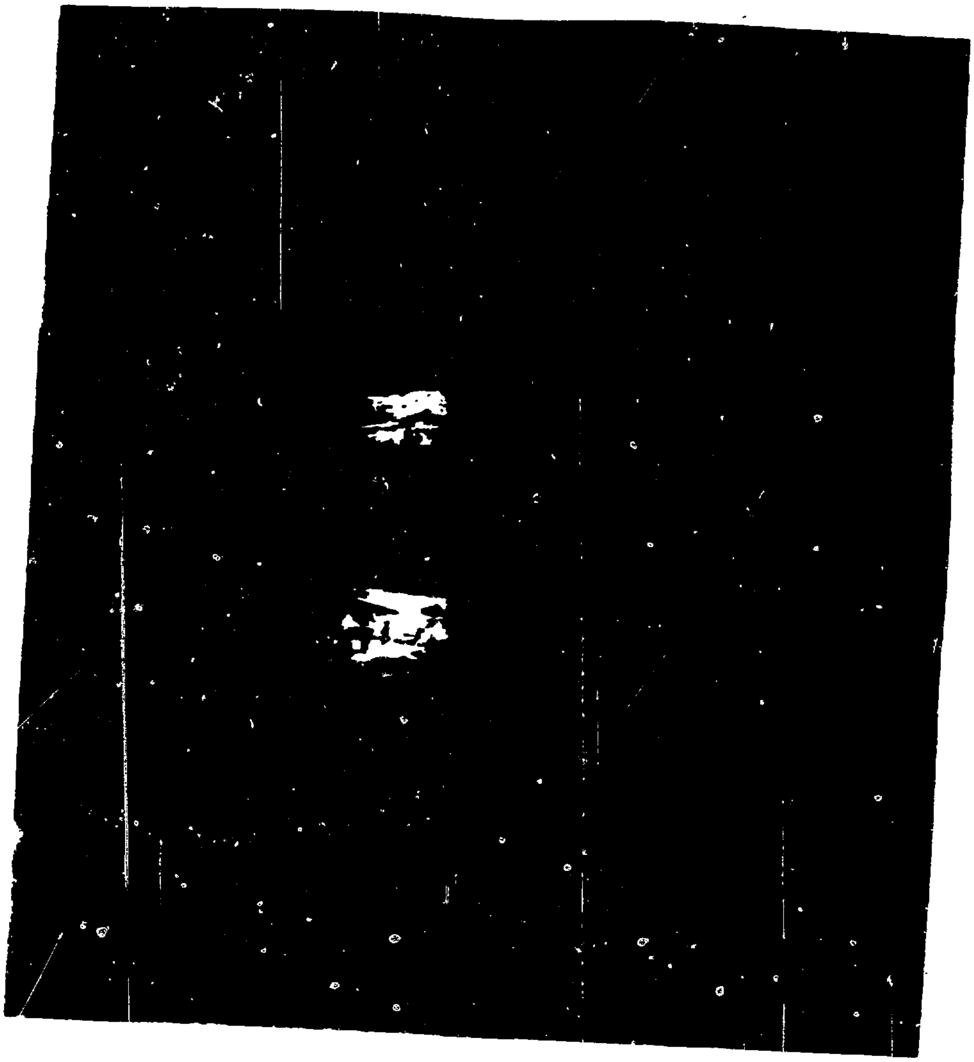
die od mm	die id mm	At failure applied load tons	applied pressure N/mm^2	hoop stress N/mm^2
* 200	75	22	49.62	65.86 *
150	75	20	45.1	75.18
125	75	16	36.1	76.68
115	75	11.5	25.9	64.33

Experimental results of EC2 Graphite dies
Pressing temperature 1100°C powder charge 300g
Billet aspect ratio h/d=0.49

17

* Plunger failure exessive loads Result ignored

SLIDE



1.

Experimental die burst-tested using hydraulic loading at room temperature.
Centre: Water filled balloon used to transmit hydraulic pressure.

18

BEST
[illegible]

Series of three CS graphite dies used to determine the strength of graphite hot pressing dies. 19

[illegible] SLI
[illegible] E

Fractured die shown as in furnace after test (reassembled after removal from furnace).

20

SLIDE 20

11" Experimental die used about 10 times at 7 ton applied loa

Cavity diameter		4.25"	108mm
Outer die diameter		11.00"	280mm
Cavity bottom		Hemispherical	
Parallel skirt on compact length		1"	25mm
Powder charge weight		750 gms	750g
Applied load	normal	7 tons	
	high	14 tons	
Normal pressing pressure		0.5tpi	7.7N/mm^2
high pressing pressure		1.0tpi	15.4N/mm^2
Hoop stress	normal	0.5*1.35	7.7*1.35
	normal	0.675tpi	10.4N/mm^2
	high	1.35tpi	20.8N/mm^2
Load at failure		25 tons	
Applied pressure at failure		1.76tpi	27N/mm^2
Hoop stress at failure		36.7N/mm^2	

Comparison of pressing practice load with values attained in a press to destruction test on an actual mould
Results of actual hoop failure test on CS Graphite die

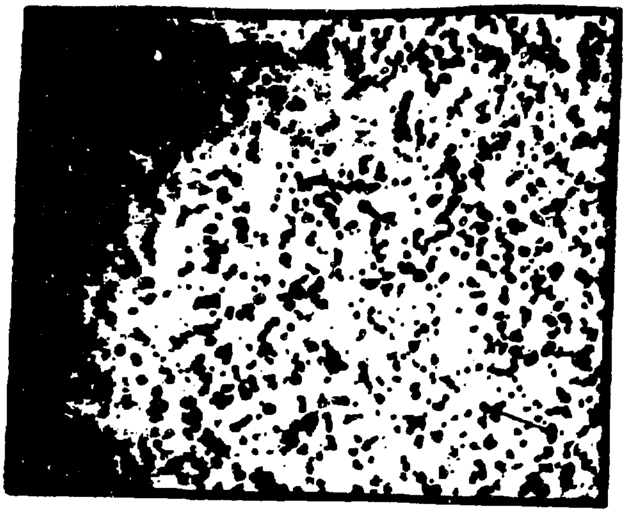
THE KINETICS OF VOID FORMATION IN ANNEALED HOT ISOSTATICALLY PRESSED BERYLLIUM.

by

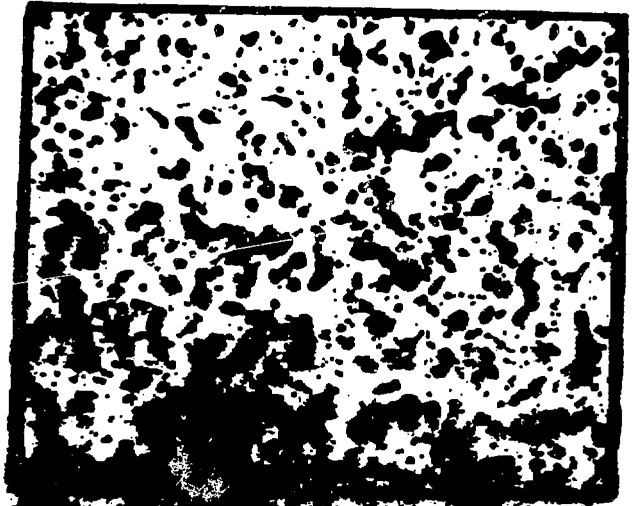
J.Ansell and J.S.White (AWRE)

Summary

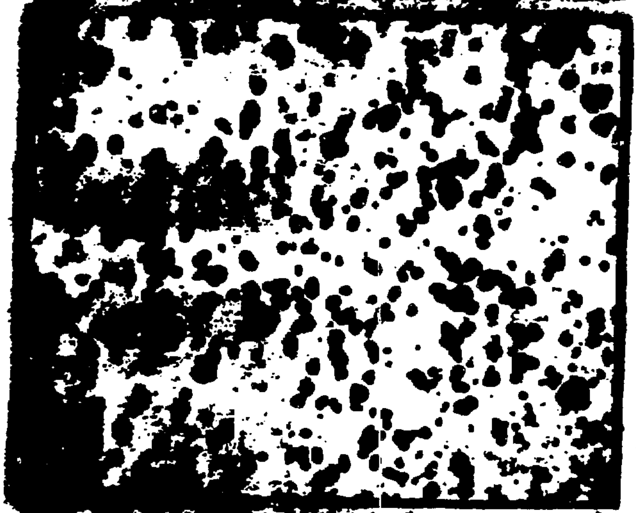
It has been qualitatively established that hot isostatically pressed (HIP) beryllium is susceptible to swelling on subsequent annealing. Previous workers have attributed this behaviour to the incomplete outgassing of the steel cans used to contain the beryllium during the HIPing operation. The kinetics of the swelling process have been studied dilatometrically in the temperature range 750 - 1150°C. The data show that approximately 30 v/o void formation was produced in the experimental samples by high temperature annealing. In addition, the kinetic data also suggest that the mechanism of void formation is different for annealing temperatures above and below 850°C.



Specimen Heat Treated at [illegible]°C
for 24hrs.
Density = 82·8%

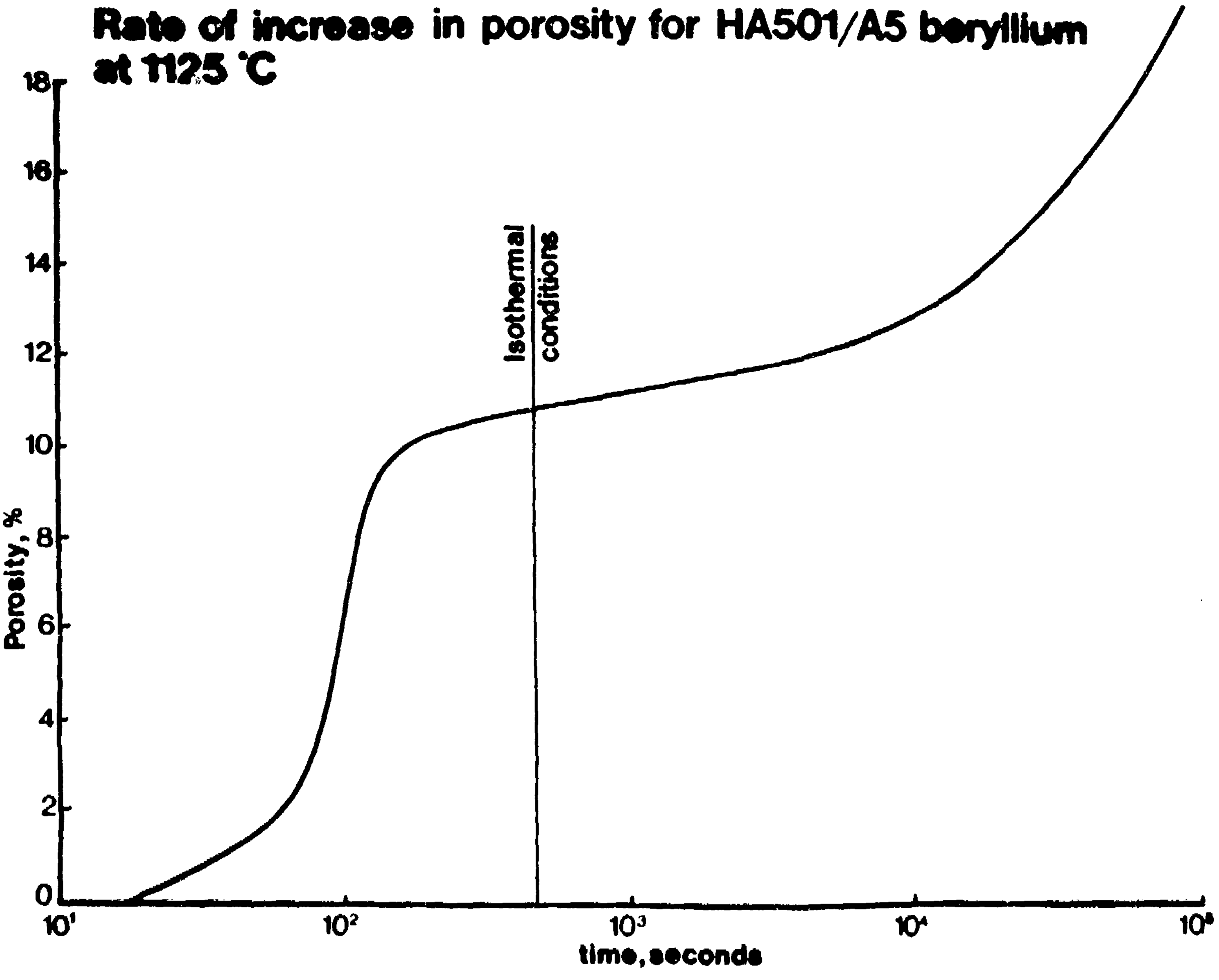


Specimen Heat Treated at 1060°C
for 24hrs
Density = 81·5%



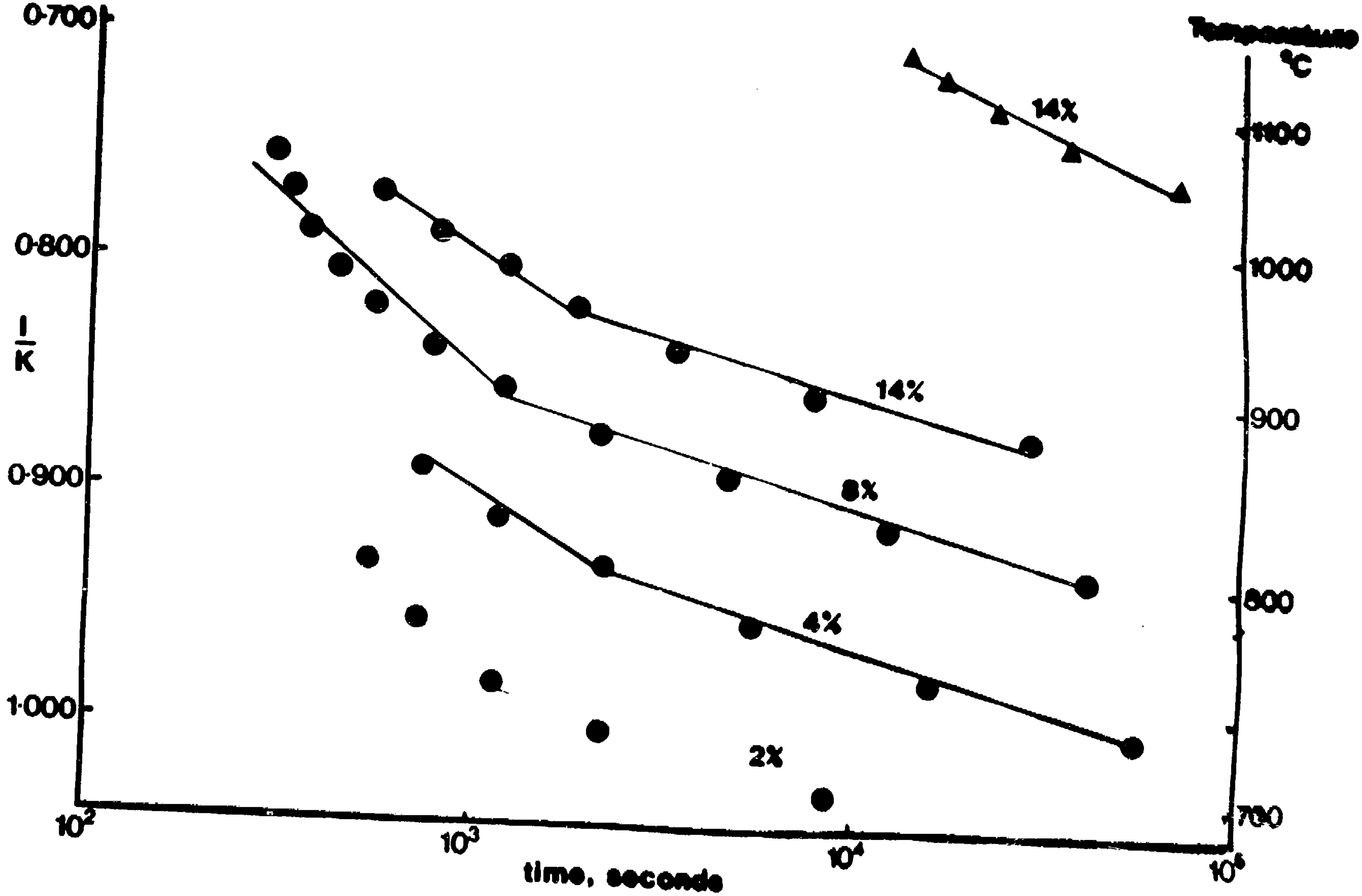
Specimen Heat Treated at [illegible]°C
for 24 hrs followed by [illegible]°C
for 18 hrs
Density = 81·1%

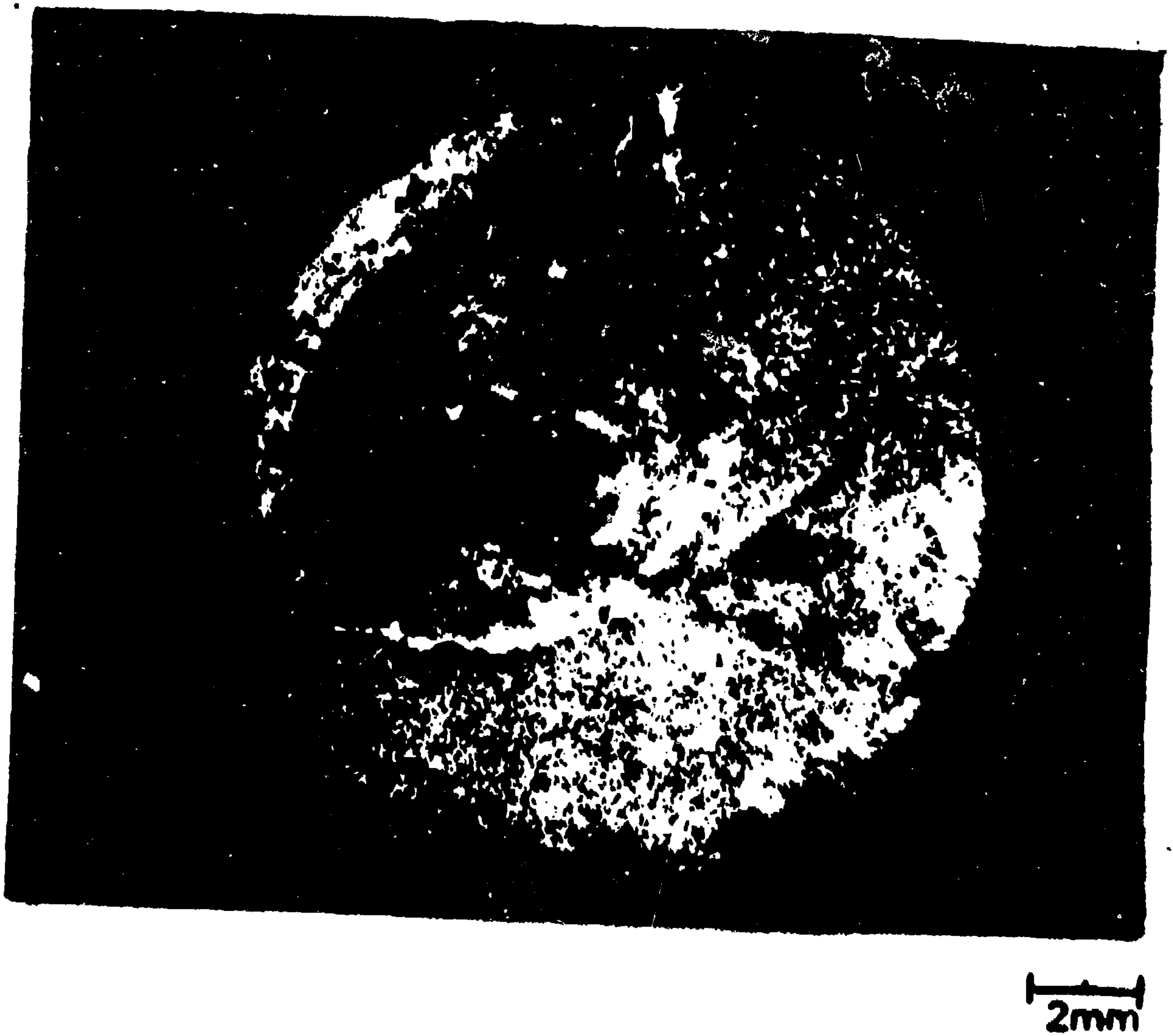
[illegible] COMPARISONS AFTER DIFFERENT [illegible]
PRODUCE SIMILAR DENSITIES.



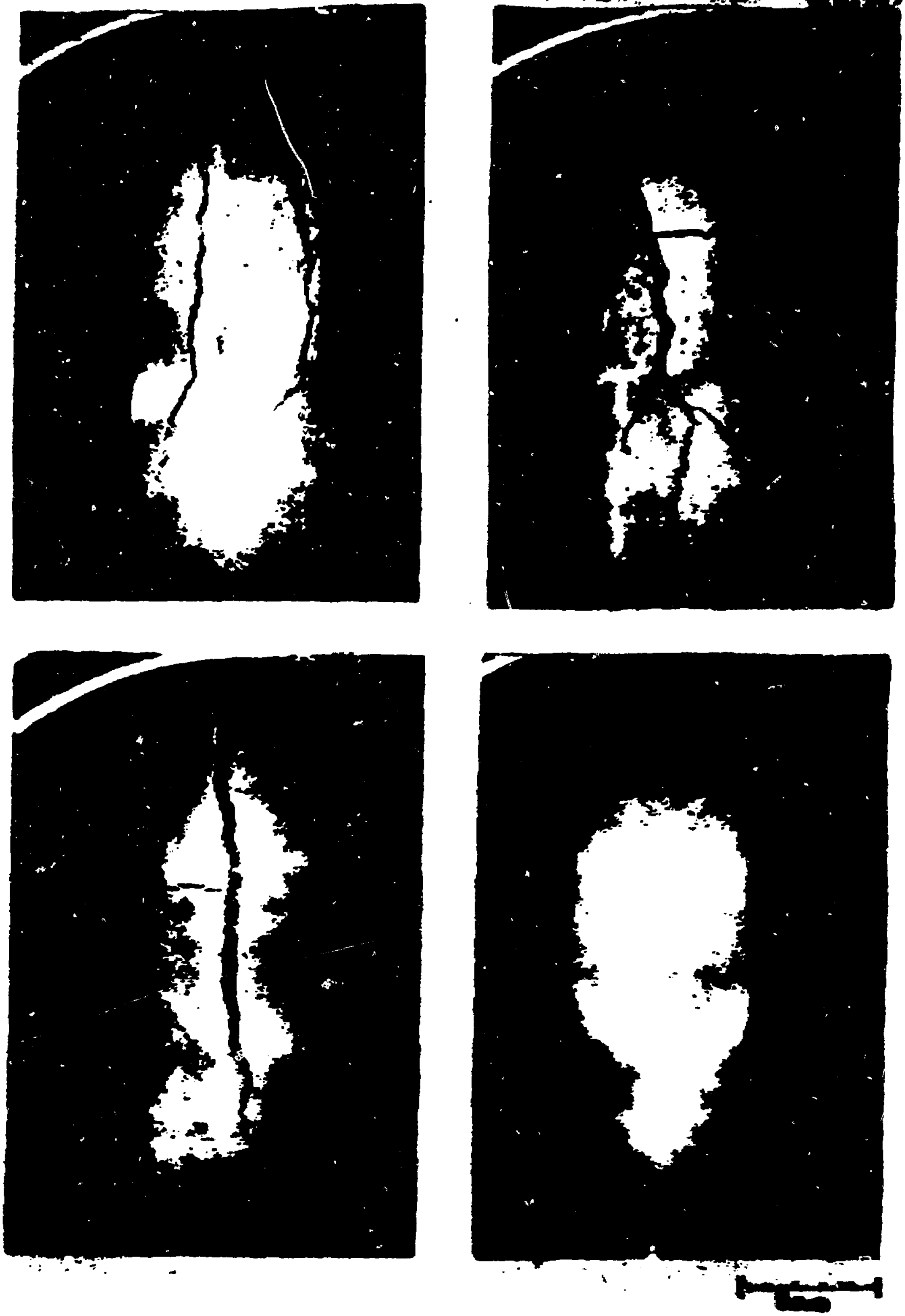
Rate of increase in porosity for HA501/A5 beryllium at 1125 °C

Arrhenius plot for HA501 type Beryllium





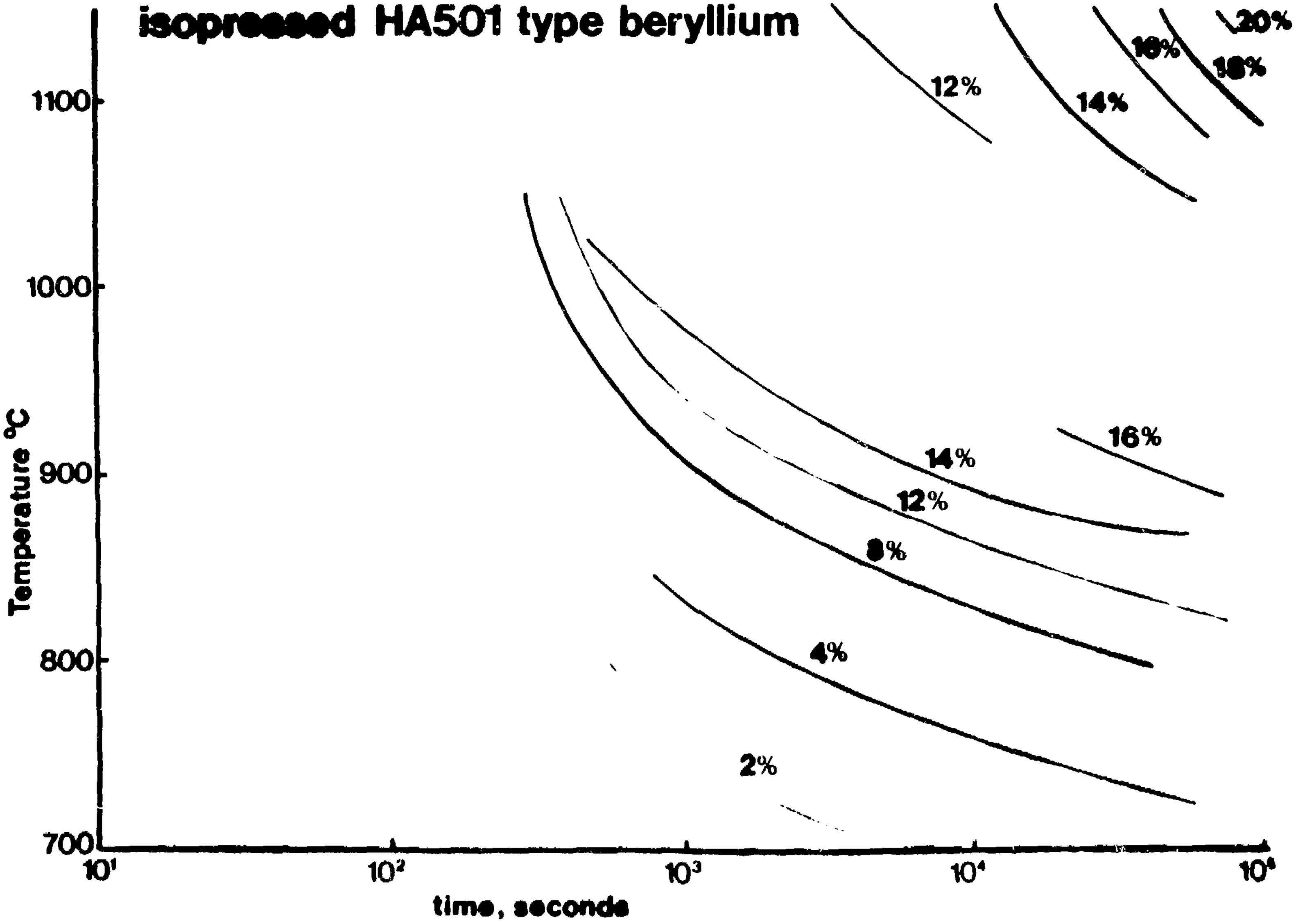
Isopressed Beryllium Specimen after Heat Treating at 1125°C for 24 hours. (74·26% Dense). Top View.

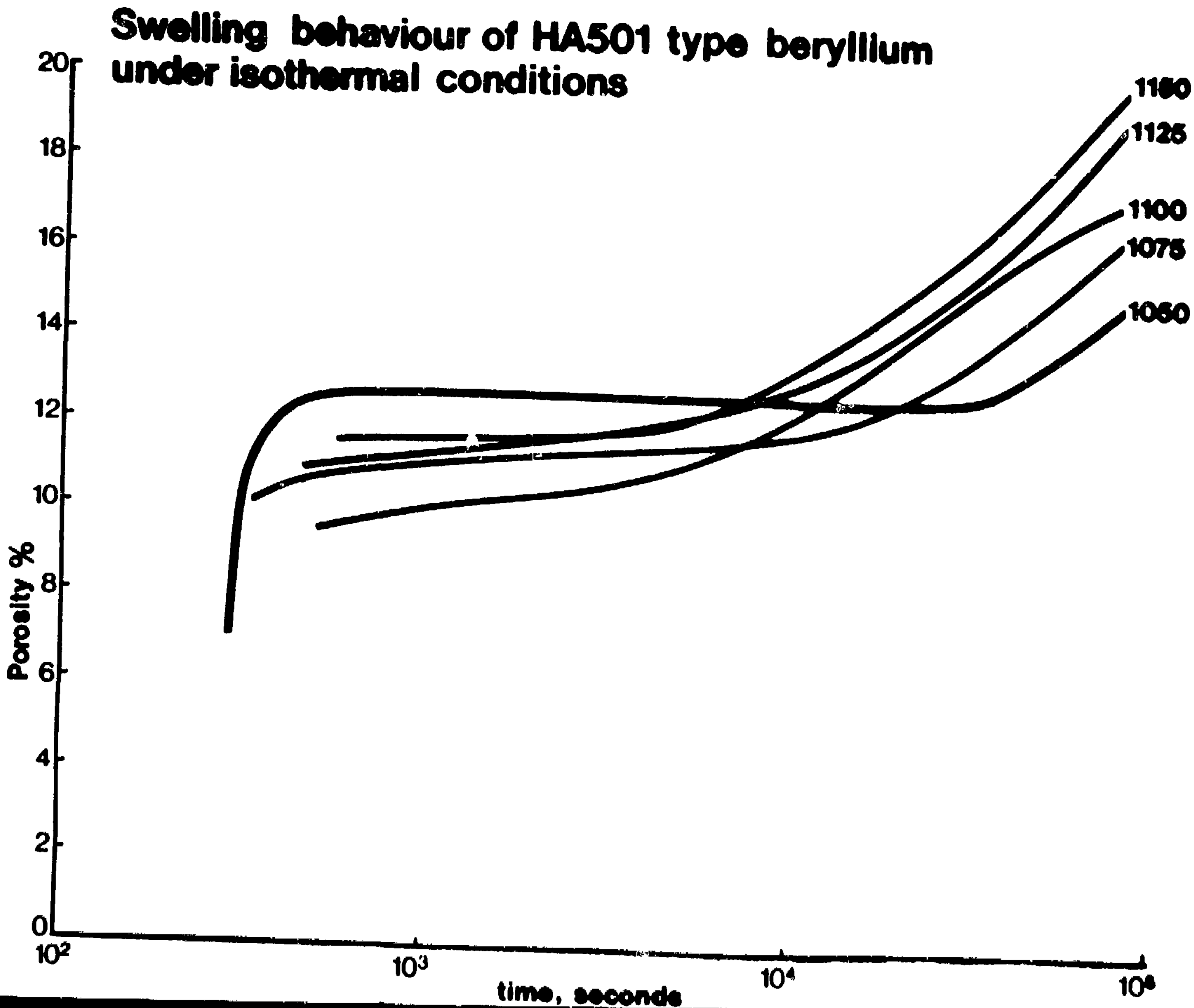


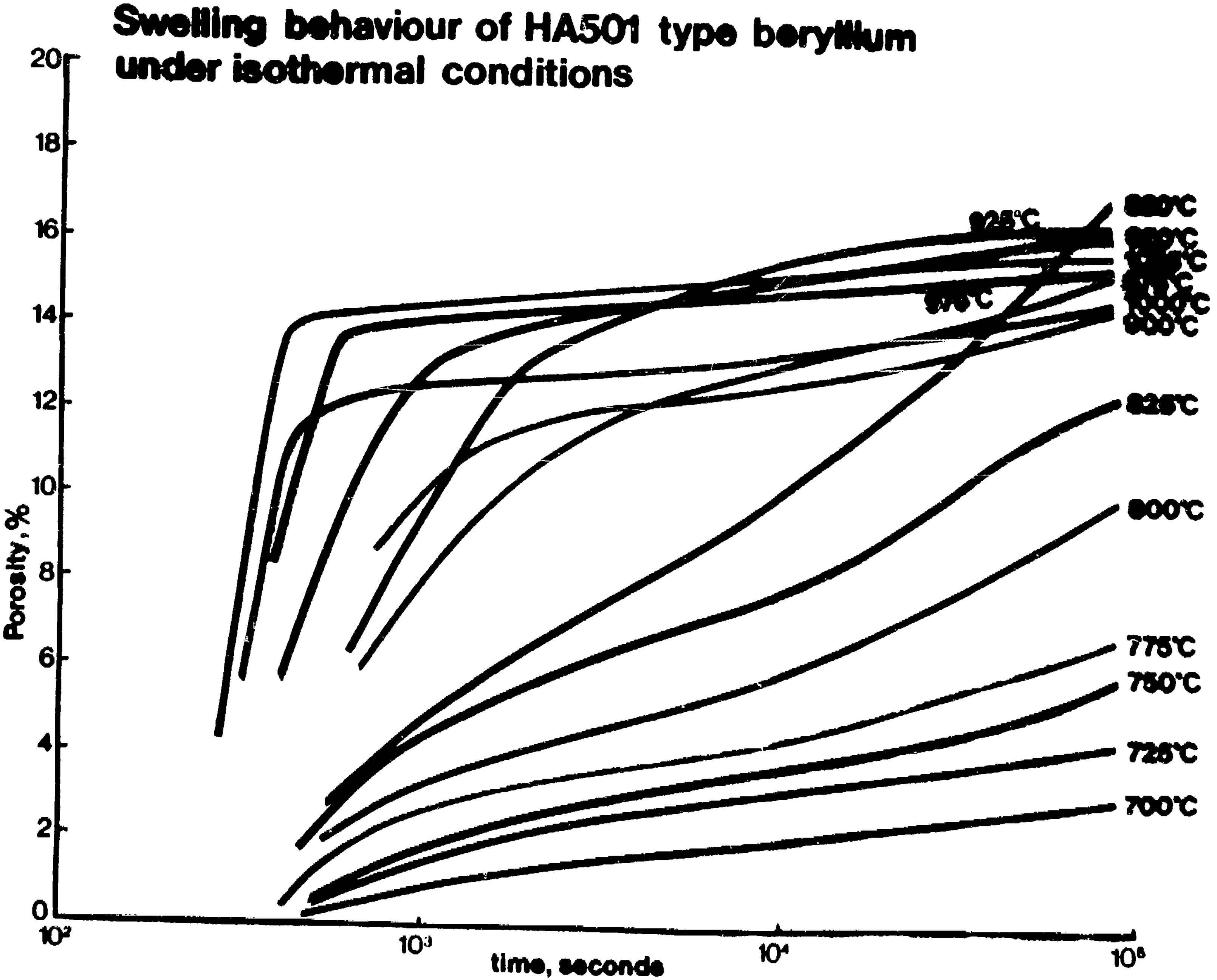
[illegible] Beryllium Specimen after Heat Treating at 1125°C for 24 hours.

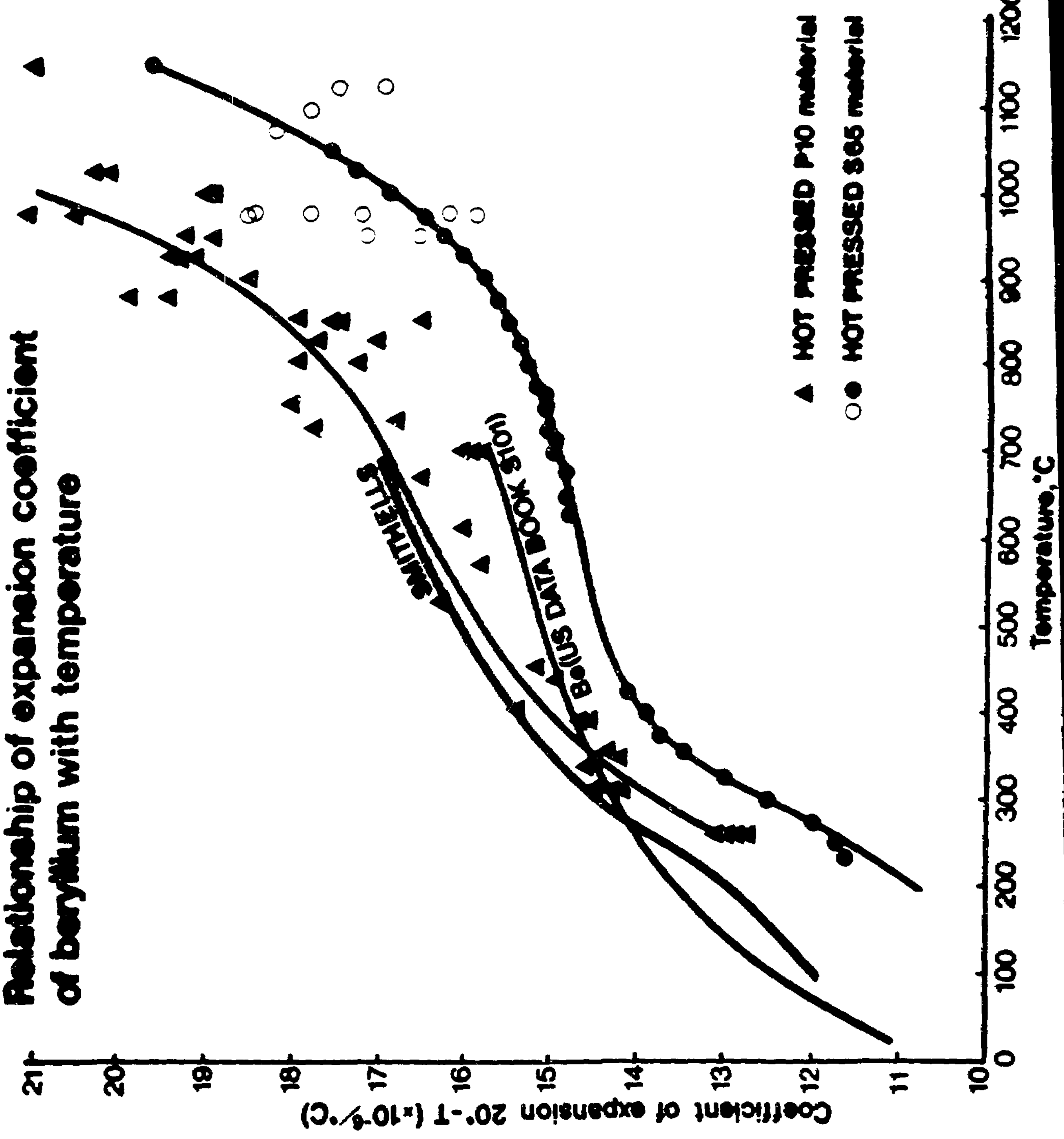
[illegible] [illegible] [illegible] Side Elevation.

Time, temperature, porosity relationship during the swelling of isopressed HA501 type beryllium

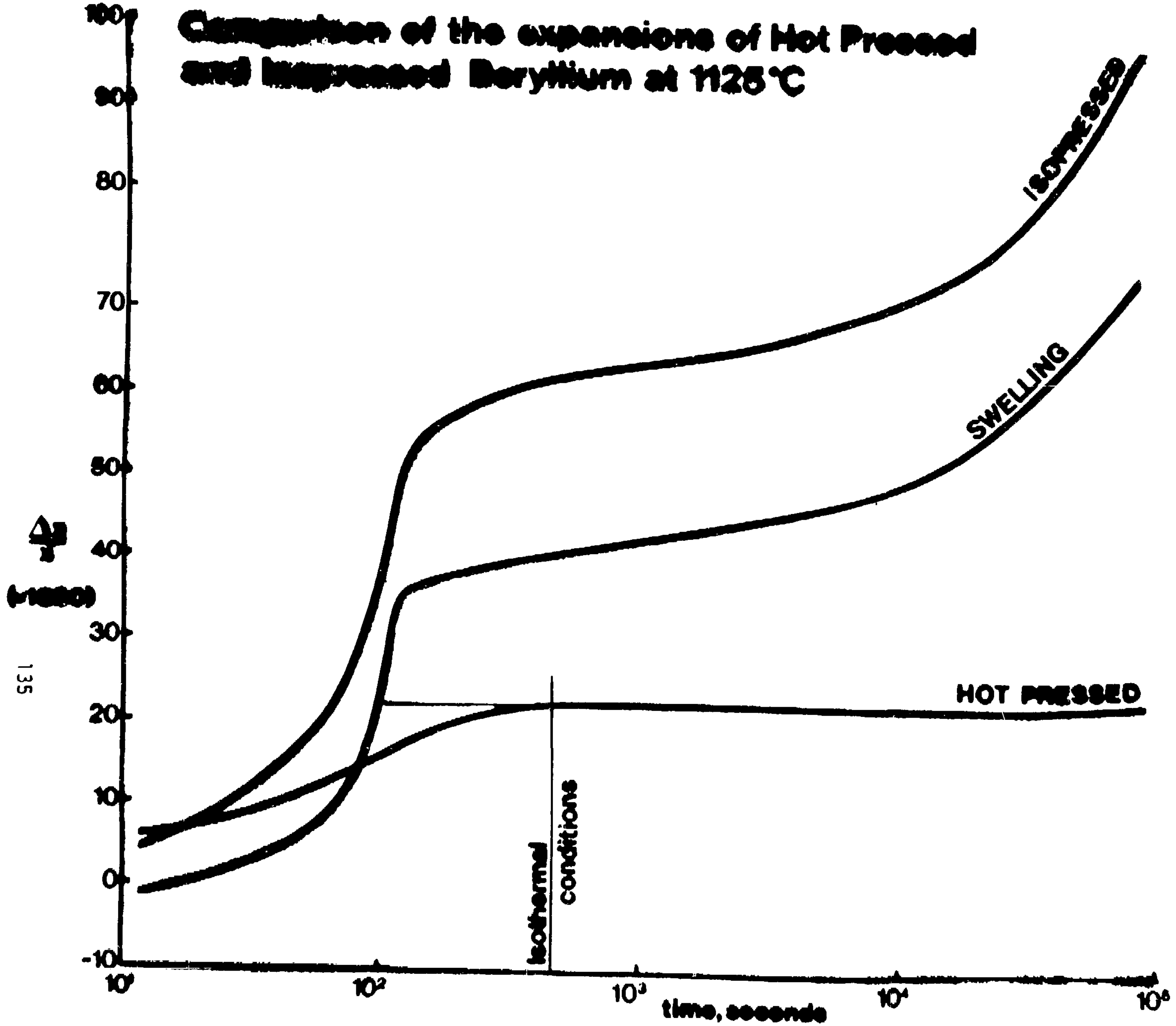



Swelling behaviour of HA501 type beryllium under isothermal conditions


Swelling behaviour of HA501 type beryllium under isothermal conditions

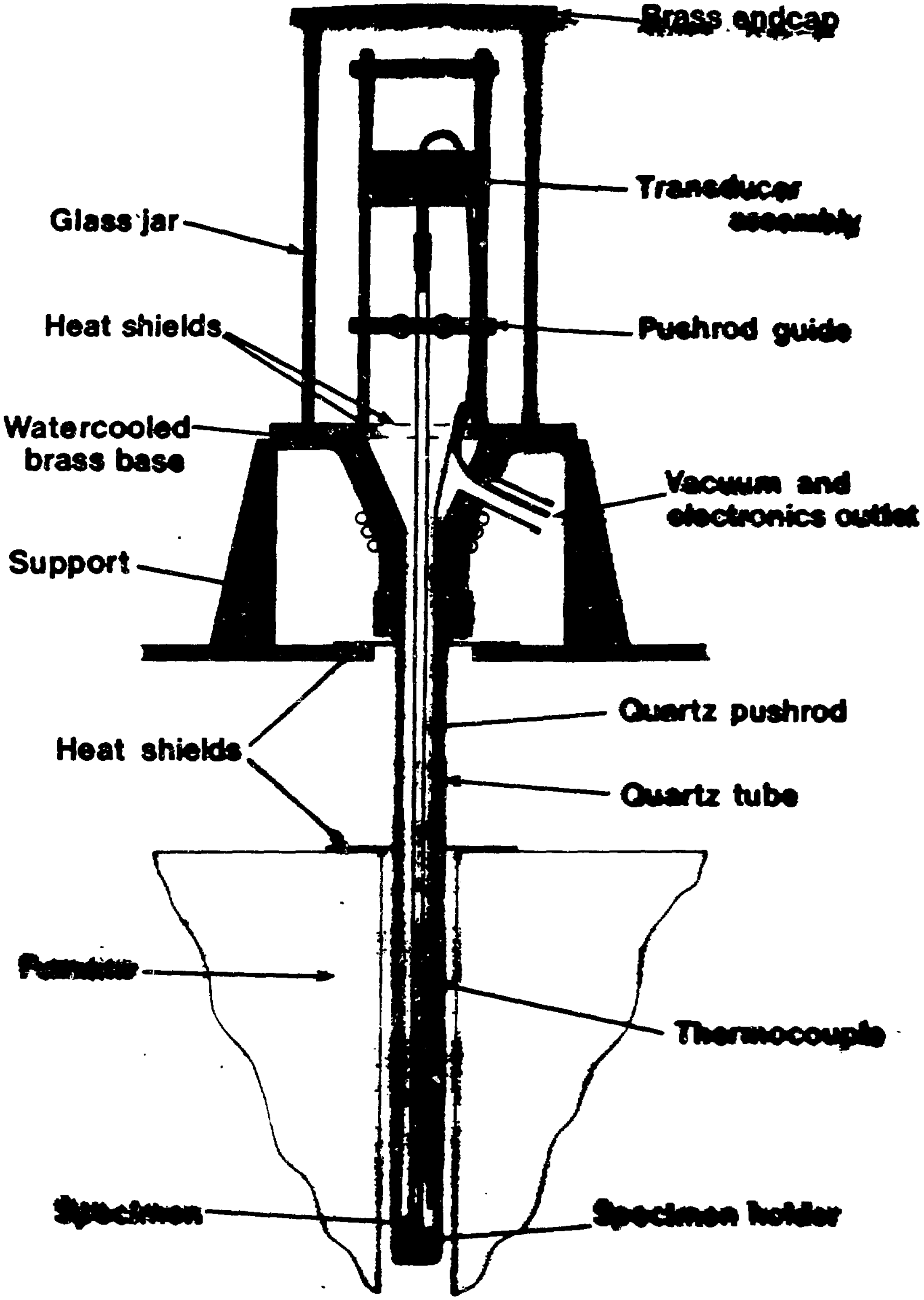


Relationship of expansion coefficient of beryllium with temperature



Comparison of the expansions of Hot Pressed and Isopressed Beryllium at 1125°C

Schematic of the Dilatometer



BERYLLIUM SPIN FORMING AT ROCKY FLATS

S. P. Abeln and M. P. Riendeau, RFP

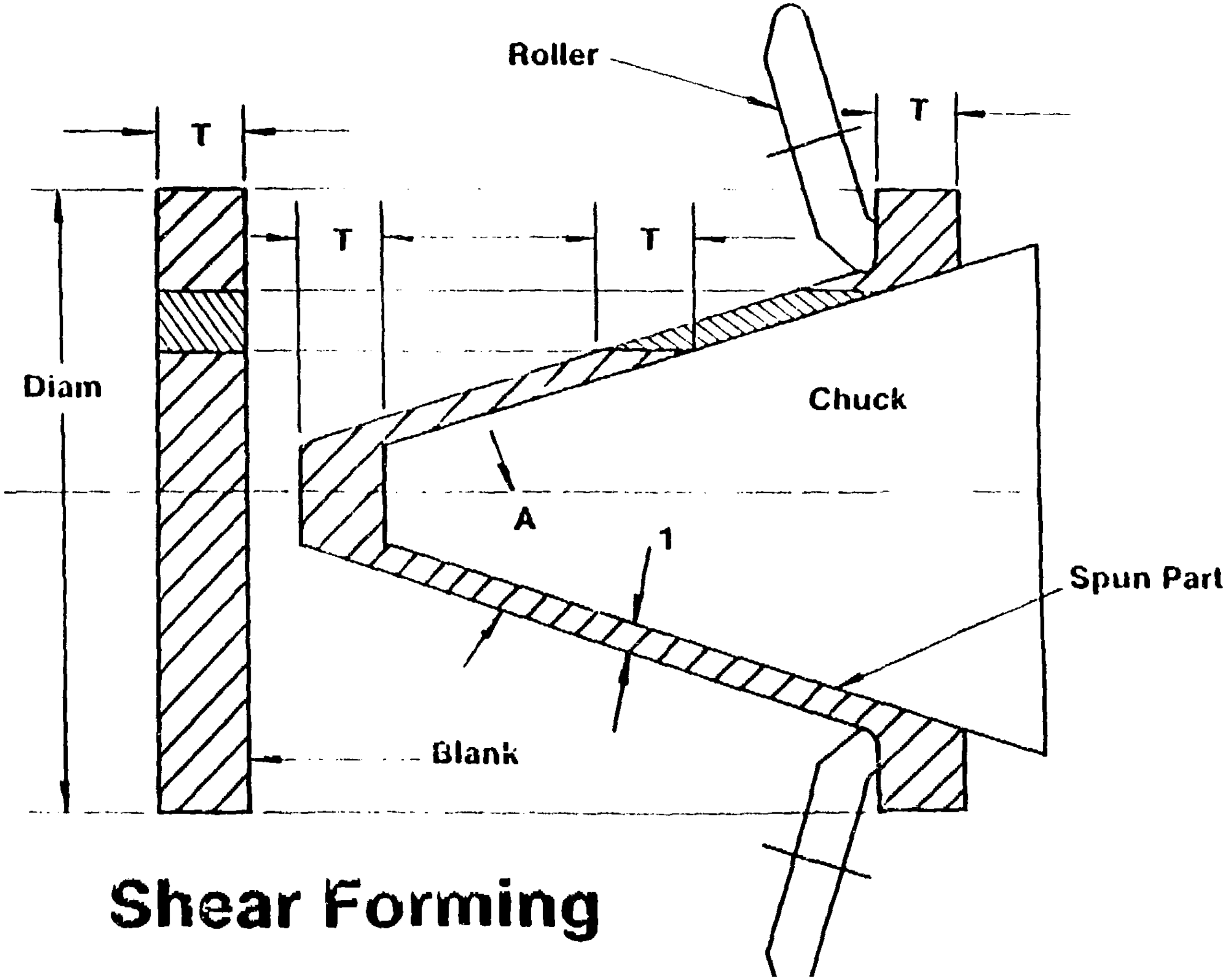
Abstract

Spinnability of pressed powder beryllium is investigated. Beryllium disks cut from vacuum hot pressed (VHP) logs are form spun over a mandrel and. total strains measured. The design matrix includes spinning of a bare beryllium disk, a disk with a cover-plate, and a completely encapsulated (canned) disk. Metallurgical evaluation includes grain structure, texture, and tensile properties before and after spinning. Material spinnability is evaluated at 400°C, 600°C and 750°C.

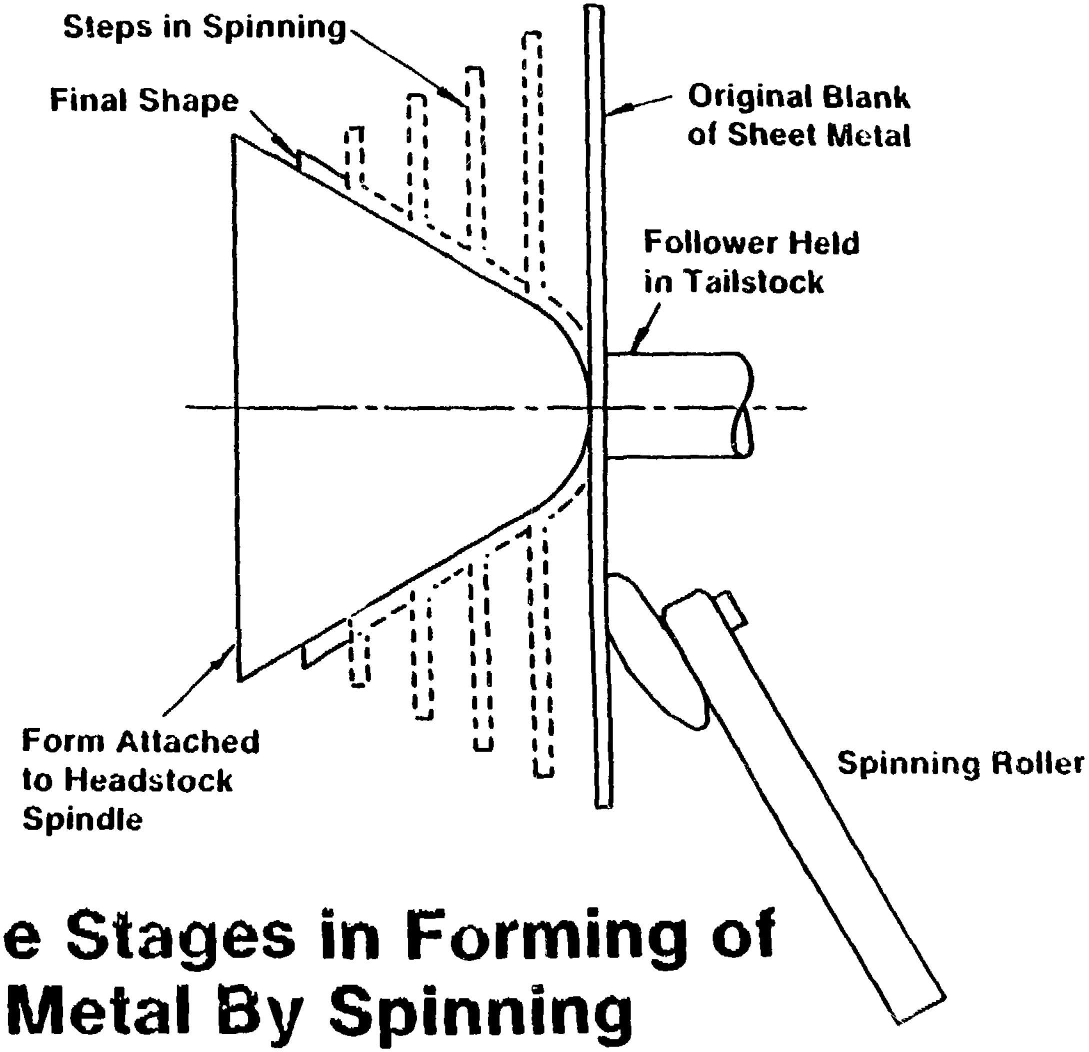
- Introduction
  - Objective
  - Form and Shear Spinning
  - Be Formability
- Experimental Approach
  - Material Characterization
  - Experimental Matrix
  - Hydrospin
- Results and Discussion
  - 600° C Spinning
  - Fractography
  - Metallography
- Conclusions

Objective

Determine environmental impact and feasibility of form spinning beryllium



Shear Forming



Progressive Stages in Forming of Sheet Metal By Spinning

Be Formability

Author(s)	Forming Method	Temp. (° C)	Material Thickness (inches)
R. R. Corle S. H. Gelles (1981)	Fukui Cup Test	400	0.060
P. B Lindsay R. P. Sernka (1972)	Shear Spinning	600	0.225
J. J. Blakeslee (1973)	Deep Drawing	600→750	0.325

Rockwell International

Material: RM 255116-A

Element	Wt%
Be	99.19
BeO	0.82
Fe	0.072
C	0.076
Al	0.036
Mg	0.022
Si	0.028
N	0.017
U	0.004
Other	-0.04

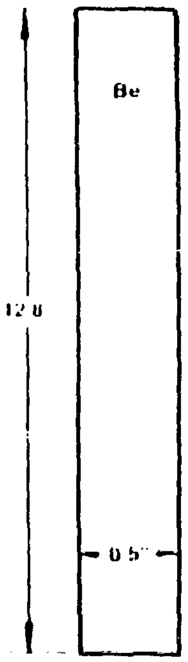
Rockwell International

Experimental Design

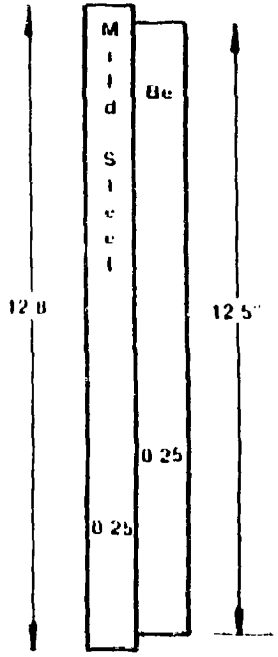
Temperatures

400° C 600° C 750° C

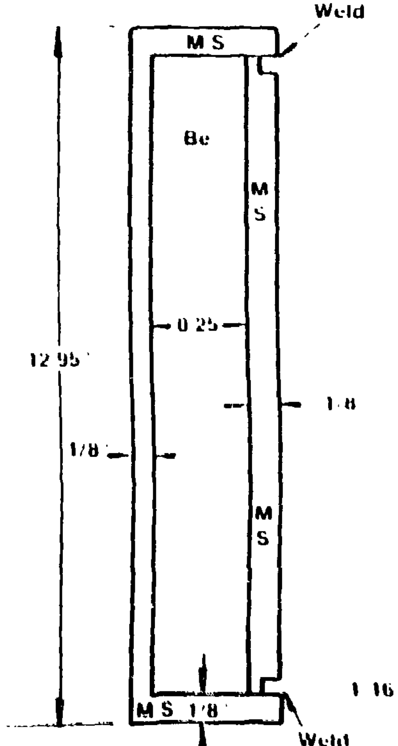
Configurations



Bare

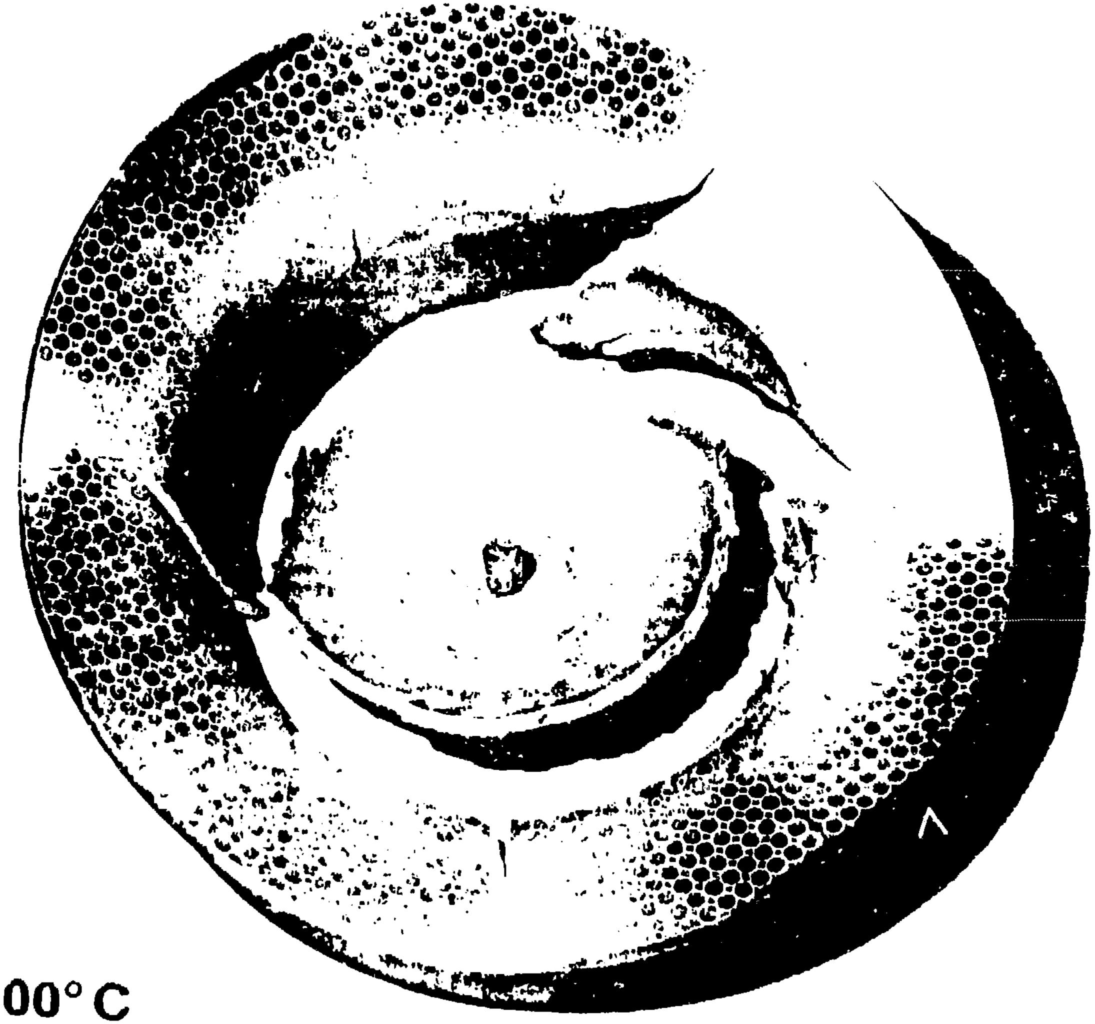


Coverplated

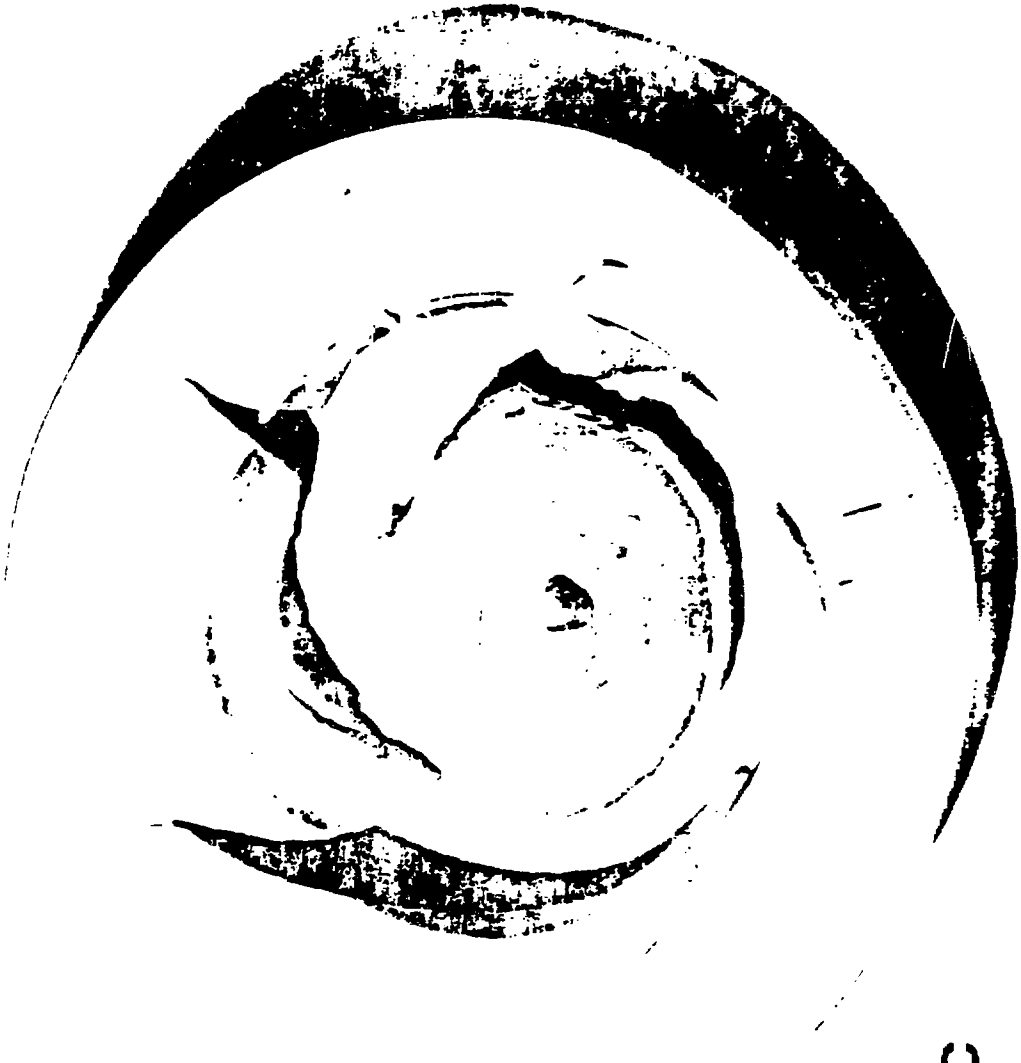


Canned

400°C

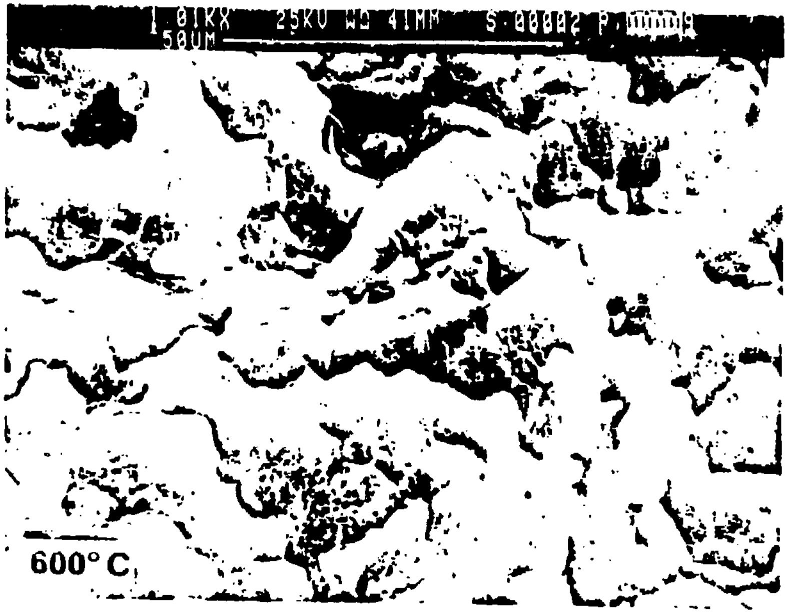


600° C

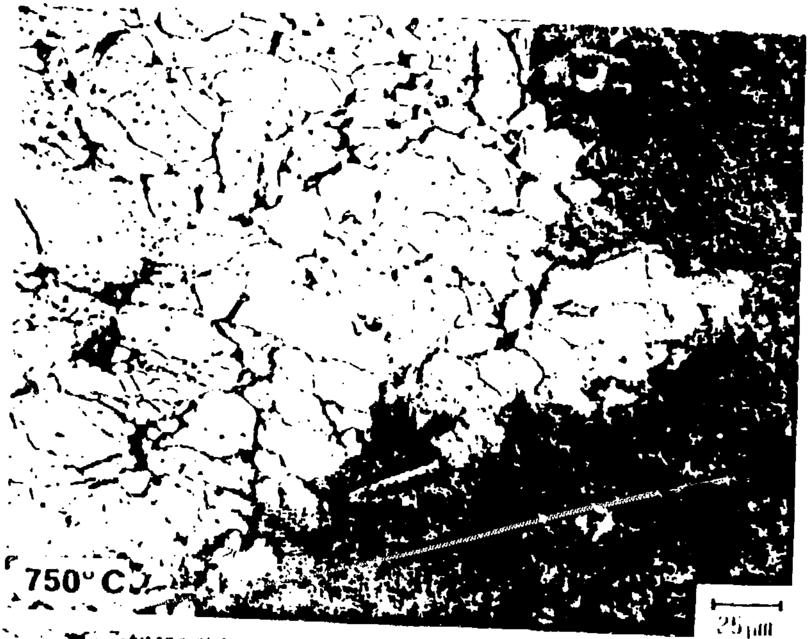


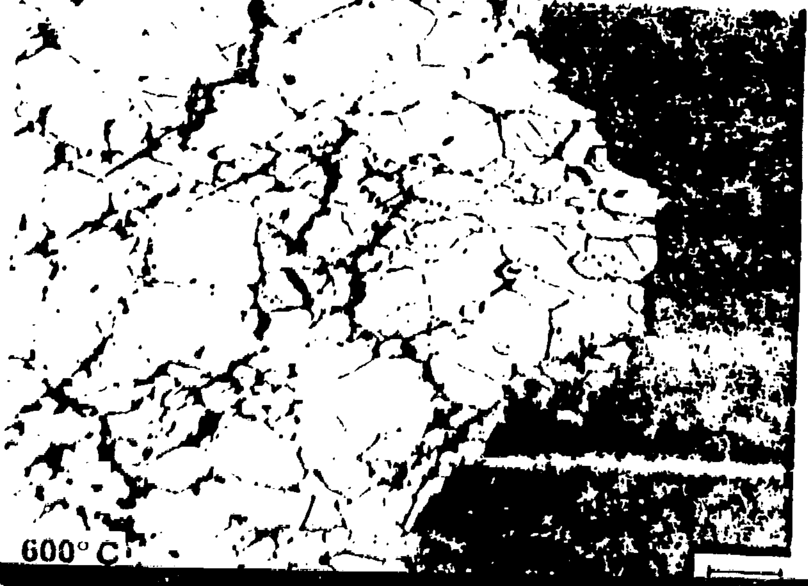
750° C

Fractography



Metallography





Conclusions

- Buckling prevents form spinning in one operation
- Several intermediate anneals required
- Respirators required by HS&E
- Not a cost-effective technology at this time

BERYLLIUM FILMS FORMED BY PHYSICAL VAPOR DEPOSITION

C. W. Nordin and R. O. Adams
Rockwell International
Rocky Flats Plant
Golden, CO 80401

ABSTRACT

Films of beryllium formed by physical vapor deposition are tenacious and non-porous. When deposited at the proper rate and substrate temperature, they have a fine grain structure. At a substrate temperature of 400°C, the grains in the film are columnar and are 10 to 20 microns in diameter. The columns typically extend from the substrate through the film. Increasing the substrate temperature increases the diameter of the columnar grains. In addition, the strength and ductility of the film increases as the substrate temperature during deposition increases. The fracture strength increases from 20,000 psi at about 400°C to about 45,000 psi at 650°C. Films up to 0.020 inch thick, have been formed by electron beam evaporation on a variety of substrates with these kinds of physical properties.

Editor's Note: The visuals for this presentation are not shown here because of reproduction constraints.

Slides are
1 number
off due to addition of a title
slide prior to slide 1.

INTRODUCTION

For a number of years we have deposited beryllium films in a variety of configurations for a variety of different applications. To date all of this work has been done by electron beam evaporation. The equipment in which this is done is shown in SLIDE 1. This is a typical deposition system that could be found in any kind of installation which does film deposition. This particular vacuum chamber is a stainless steel cylinder 3 feet in diameter and 3 feet high. The attached vacuum system consists of a mechanical pump and an 10 inch diffusion pump with a liquid nitrogen cold trap. The system is capable of pressures in the low 10^{-6} torr range on a routine basis; on a good day it will go into the 10^{-7} torr range. The beryllium is heated to evaporation temperatures with an electron beam gun operated at 10,000 volts. The beam current is variable and controls the deposition rate. As you might expect, beryllium evaporates readily. In fact it sublimes with a significant rate at temperatures a few hundred degrees below its melting point. At normal operating temperatures of about 1400 C, we can get deposition rates of over 1 mil/hr depending on the configuration of the system.

As mentioned earlier, we have deposited beryllium on a variety of parts of different sizes and shapes. SLIDE 2 shows some parts at one end of our size spectrum. These are about 2500 microns in diameter coated with beryllium to the thickness shown on the slide. These part were to be free standing spheres of beryllium. They were coated as hemispheres on copper mandrels, they were then machined and the the mandrel etched away. The beryllium film is fine grained which allowed for a mirror finish on the finish machined part.

SLIDE 3 shows a part at the other extreme in size . This is a beryllium mirror blank that is 24 inches in diameter and 3 inches thick. We coated it with 12 mils of beryllium. The coating was done in a single 4 hour run.

With the proper deposition parameters, particularly the deposition rate and the substrate temperature, films with very fine grains can be deposited. SLIDE 4 shows a typical film deposited at about 1 mil/hr on a substrate that is at 400 C during deposition. The grains are columnar and quite fine with diameters of 5 to 20 microns. The film is dense which is noticeable by the lack of voids and porosity between the grains. The angle at which the grains appear to be oriented is caused by the placement of the substrate in relation to the beryllium vapor source. The film grows toward the vapor source, hence the grains are oriented toward the vapor source. This slide shows two photomicrographs taken 90° to each other. This can give us some idea of the cross section of the grains.

The substrate on which the film is deposited has little effect on the structure of the film. SLIDE 5 shows a film grown on a polished surface. The film has a dense columnar growth pattern. SLIDE 6 shows a film grown an a machined surface. The film has the same appearance as the film grown on the polished surface.

The temperature of the substrate during deposition has probably the greatest effect on the grain growth of the deposited film. This is true of any material, metal semi-conductor or insulator. SLIDE 7 is a well known, to vacuum coaters anyway, diagram of the effect of temperature on the grain size of a deposited film. This slide also shows the effect of the pressure of a sputtering gas on the grain size but we can disregard that axis. You can see that there are three zones divided according to temperature measured in terms of percentage of melting point. As the temperature increases the size of the grains increase; a fact that is not too surprising.

Delete
Slide
7

Two examples of this are shown in the next two slides. SLIDE 8 is the cross section of a beryllium film deposited at a substrate temperature of 140 C. The grains are very fine but there are a large number of voids between the grains. Films such as these are quite brittle and highly stressed. They typically do not adhere well to the substrate. SLIDE 9 is a photomicrograph of a beryllium film deposited at 400 C substrate temperature. Here is the dense structure with still fairly small grains. Most of the beryllium films that we have deposited were done in the range of this temperature, 300 C to 500 C.

Some of the films we have made have been too thick to deposit in one deposition run because of the limited size of the evaporation source. In these cases, the chamber has to be opened and a new source put in place. This means that the film is exposed to the atmosphere and thus a layer of oxide forms on the surface of the film. SLIDE 10 is a photomicrograph of a cross section of a multilayer beryllium film. You can see that the oxide layer has little effect on the growth of the film. In most cases seen on this slide the oxide layer is so thin that it is not easily seen. In these cases the grains appear to continue to grow through the oxide layer. In one case a thicker oxide layer is present but the grains still appear to continue their growth.

The vapor deposition process also appears to refine the beryllium as it deposits. SLIDE 11 shows the composition of the film compared to the composition of the beryllium evaporation source. You can see the significant change in the oxygen content.

Since the characteristics of the grain growth change with temperature of the substrate, on might expect the physical properties of the film to also change with substrate temperature. And indeed they do as in shown in SLIDE 12. The electrical resistivity decreases with increasing substrate temperature. The hardness decreases as do the fracture strength and the total elongation.

Films and free standing shapes made from films of beryllium can be made by electron beam evaporation. The properties of these films can be tailored by controlling the substrate temperature.

Rockwell International

Chemical Composition of Beryllium

Element	Weight Percents		
	Source	As-Deposited	Changes in Composition (ΔC)
Beryllium Oxide (BeO)	0.05	—	—
Iron (Fe)	0.0135	0.001	−0.0125
Aluminum (Al)	0.010	0.018	+0.008
Calcium (Ca)	<0.003	<0.003	—
Cadmium (Cd)	<0.001	0.001	—
Cobalt (Co)	<0.001	0.001	—
Chromium (Cr)	0.002	0.002	—
Copper (Cu)	0.003	*—	—
Magnesium (Mg)	<0.001	0.004	+0.003
Manganese (Mn)	0.008	0.016	+0.008
Molybdenum (Mo)	<0.003	0.005	+0.002
Nickel (Ni)	0.013	0.003	−0.010
Lead (Pb)	<0.001	<0.001	—
Silicon (Si)	0.010	<0.001	−0.009
Titanium (Ti)	0.029	<0.001	−0.028
Tungsten (W)	<0.020	—	—
Zinc (Zn)	<0.010	<0.010	—

*Findings showed that the weight percent of copper in the foils was dependent on the substrate temperature.

Properties of Beryllium Films Versus Substrate Temperature

Substrate Temp. During Evaporation (°C)	Electrical Resistivity (micro-ohm centimeters)	Knoop Hardness No. (100-percent gram load)	Fracture Strength (1000 lbs. per sq. in.)	Total Elongation (percent)
200 to 270		488 to 490		
300 to 345	9.62 to 12.9	267 to 326		
350 to 425	8.85 to 9.22	250 to 230	19 to 21	0.1
480 to 550	4.2 to 4.9	204 to 233	39 to 43	0.3
625 to 685	4.5 to 4.8	168	40 to 52	0.45

SPEEDS AND FEEDS FOR CARBIDE ON BERYLLIUM

D. V. Gallagher

Abstract

Surface finish and tool wear in the machining of beryllium has long been a concern in the ability to machine accurate parts. This study covers the use of carbide tooling, (Carboloy 999 or equivalent grade), presently in use in the Be shop. The independent variables were speed in surface per minute (sfm) and feed rate in inches per revolution (ipr). The optimum values found for speed and feed are 220 sfm and .004 ipr respectively. These values were chosen so that if the machinist operated at 120% on feed and speed an acceptable product could still be made with good surface finish and minimum tool wear. The surface feet per minute was varied between 180 to 350 sfm, and feed rate was taken between .002 and .048 ipr. The dependent variables were surface finish of the part, and tool wear measured on the flank of the tool.

Objectives of tool study

Achieve surface finishes of 63 microinches AA or less so that polishing is not required and thus reduce aerosols produced.

Determination of optimum surface feet and feed rate was desired due to recent retrofitting of lathes in the Be shop permitting constant surface feet to be programmed.

Determination of wear on flank of tool so that tool radius wear may be estimated.

Parameters for tool study

Style of insert was DPGT 32X where X signifies radius of the tool. A .030" radius tool was used for straight cutting and a .015" radius tool was used for spherical cutting.

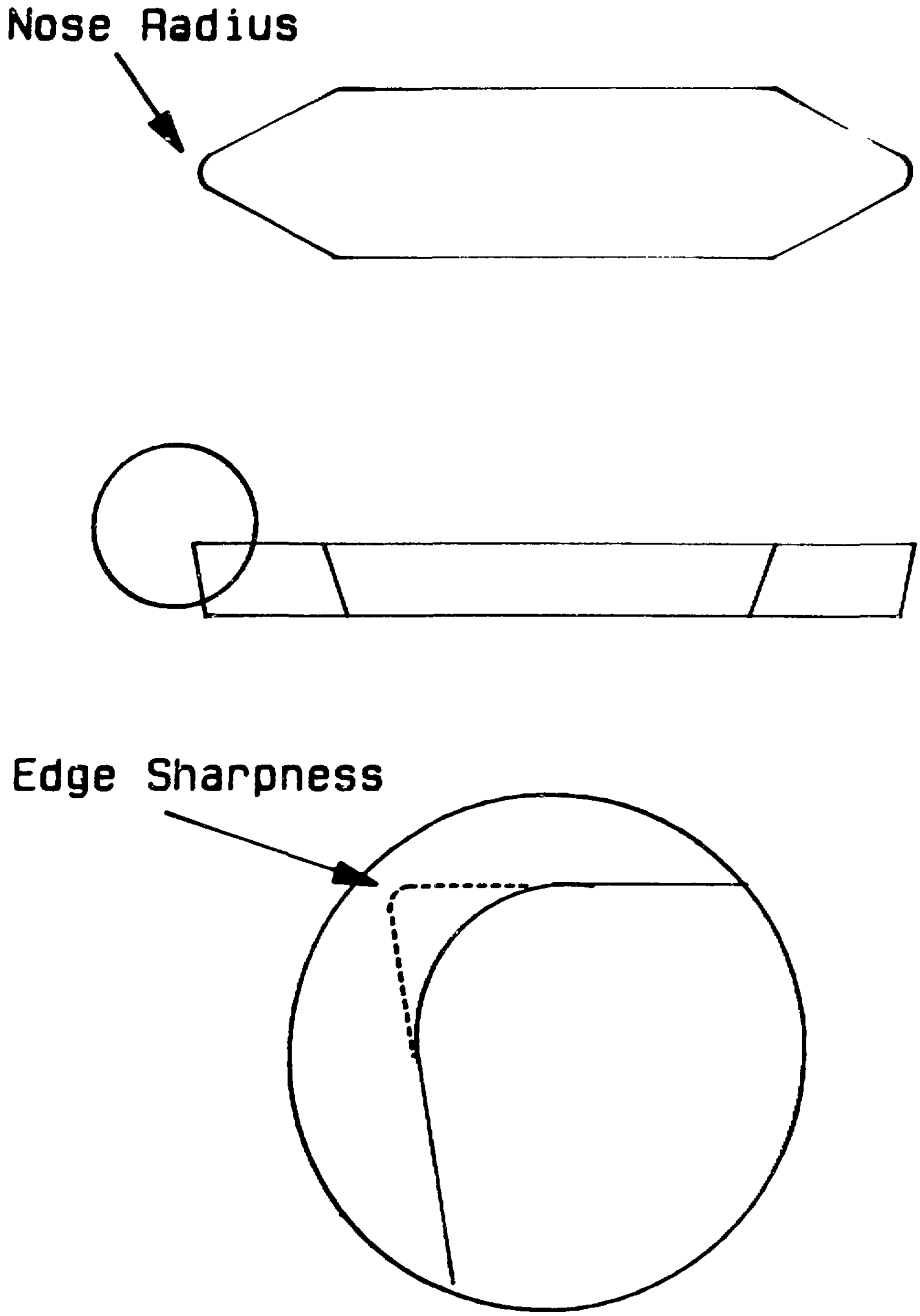
Grades: Teledyne HF
Kennametal K11

Nose radius tolerance: ± .0005

Neutral (0°) rake on tool

Tool holder was milled with a three degree positive rake.

Insert DPGT 32X



Parameters for tool study (cont.)

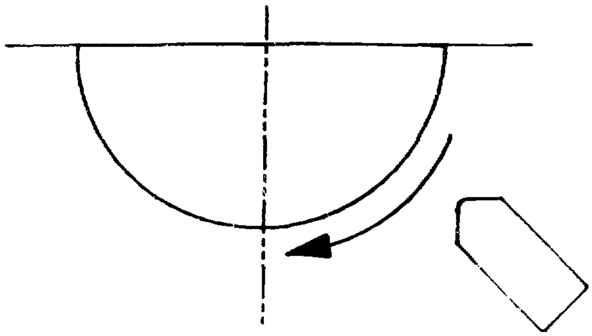
Feedrates: Given in inches per rev. (ipr)
.002, .004, .0048

Speeds: Given in surface feet per min. (sfm)
180, 220, 260, 300, 350

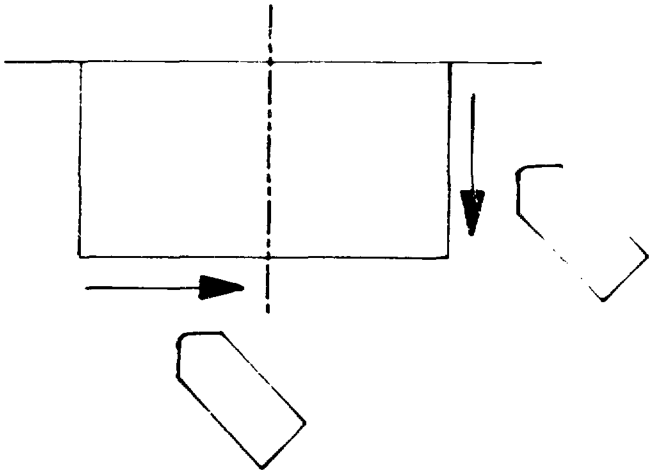
Depth of cut was .005" for all speeds and feeds used.

Machine: Excello T-Base lathe

Experimental setup for spherical and straight cuts



Spherical cutting



Straight cutting

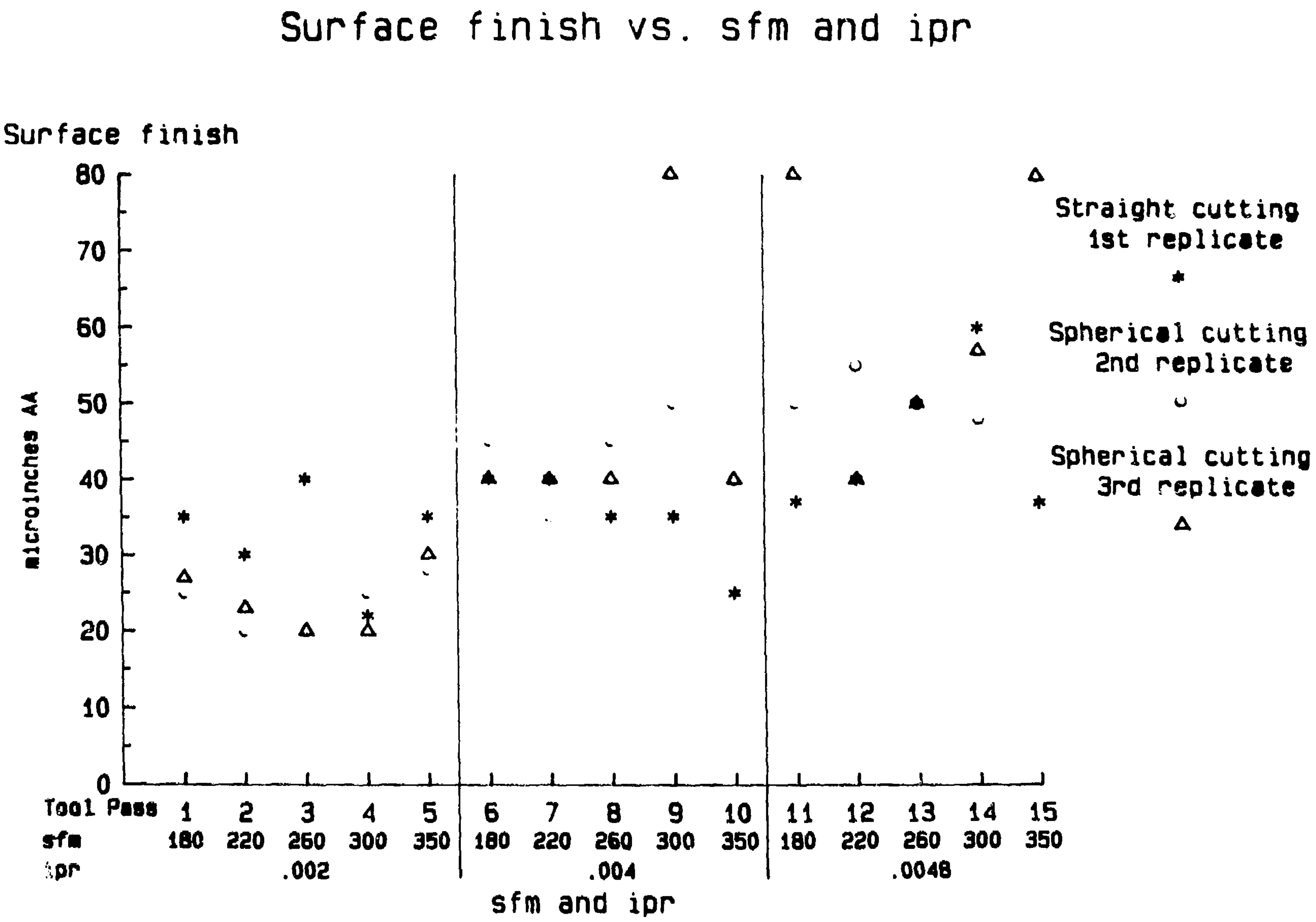
Results of tool study

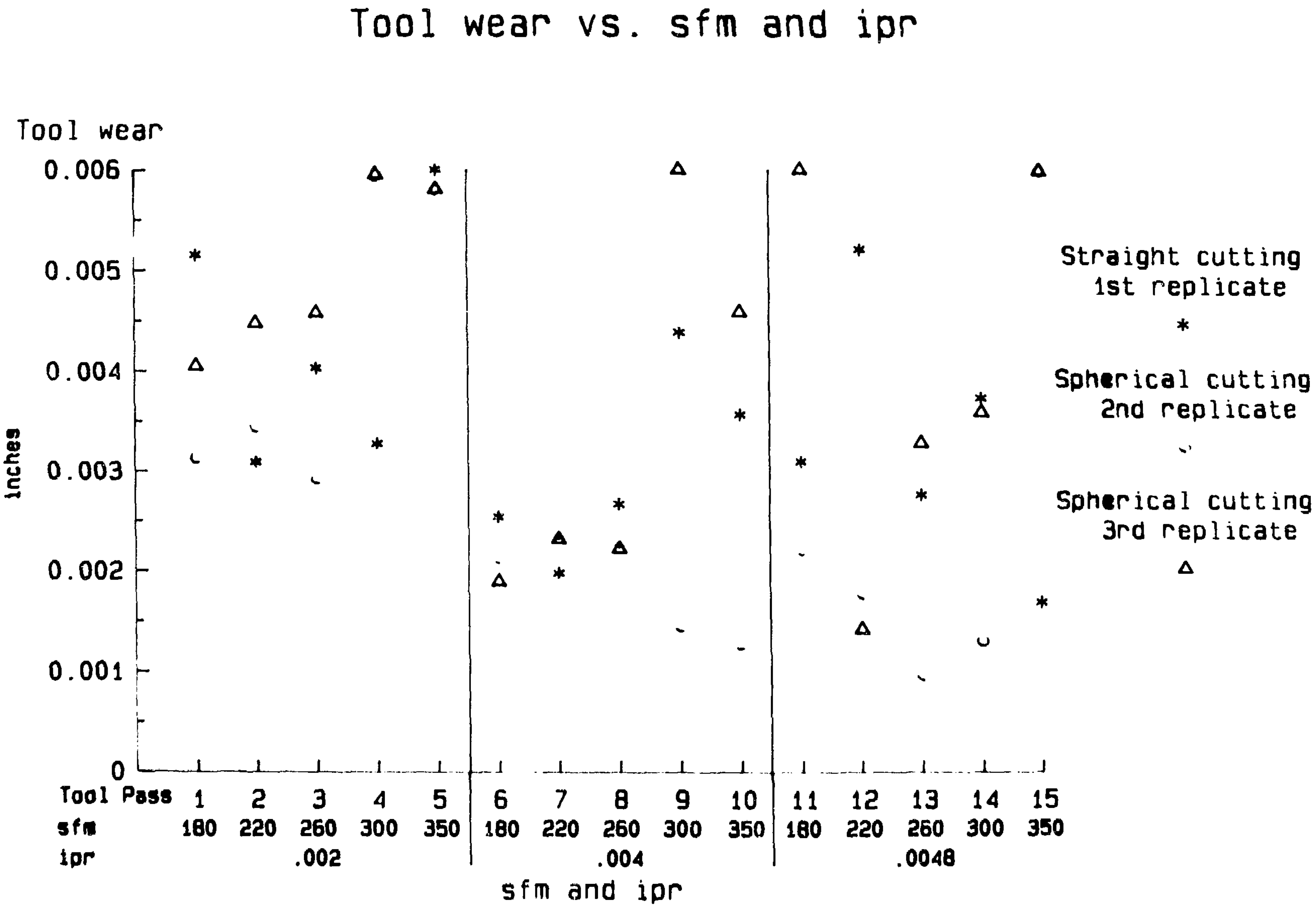
Tool flank wear varied from .0019" to .0055".

Surface finish was between 12 & 60 microinches AA excluding tool failure.

Optimum range for sfm & ipr was found to be 260 sfm. and .004 ipr.

A built up edge was found at all speeds and feeds tried. All cutting was done dry.


Surface finish vs. sfm and ipr
Surface finish
microinches AA
80
70
60
50
40
30
20
10
0
Tool Pass
sfm
ipr
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
180
220
260
300
350
180
220
260
300
350
180
220
260
300
350
.002
.004
.0048
sfm and ipr
Straight cutting
1st replicate
*
Spherical cutting
2nd replicate
○
Spherical cutting
3rd replicate
△


Tool wear vs. sfm and ipr
Tool wear
Inches
0.006
0.005
0.004
0.003
0.002
0.001
0
Tool Pass
sfm
ipr
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15
180 220 260 300 350 180 220 260 300 350 180 220 260 300 350
.002
.004
.0048
sfm and ipr
Straight cutting 1st replicate *
Spherical cutting 2nd replicate
Spherical cutting 3rd replicate Δ

Future work planned on Beryllium tooling

Ceramic and carbide tooling with controlled edge sharpness and 2 to 4 microinch AA finish on tooling.

Edge radii from .0002" to .0015" will be tried. First trial of tooling will have edge sharpness of .0005" to .0006".

Metallographic work on surface damage due to different cutting tools and machining parameters will be examined.

JOWOG 22B

BERYLLIUM

ABSTRACTS AND PRESENTATIONS

September 23, 1987

RECENT ADVANCES IN THE DIAGNOSIS OF CHRONIC BERYLLIUM DISEASE

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Acute and chronic beryllium diseases are lung diseases which have been identified in workers exposed to beryllium and beryllium-containing materials. Although scattered cases were reported in Europe earlier, these disesases were not recognized in the U.S. until the 1940's. Originally thought to be a continuum of the same disease, it is now clear they are separate entities with significantly different pathogeneses and prognoses. Acute beryllium disease was a chemical pneumonia, now only of historical interest, with the last cases occurring nearly 20 years ago. However, chronic beryllium disease, a granulomatous, interstitial, pneumonitis, can develop in 1-2% of workers exposed to a significant quantity of beryllium. This condition is considered an immunological disease and presents a difficult differential diagnostic dilemma between other granulomatous diseases most notably sarcoidosis. New specific diagnostic tests have recently become available which will be described, as well as their relationship to the more traditional use of X-rays and pulmonary function tests.

SPECIAL REVIEW OF EVALUATION AND CONTROL OF OCCUPATIONAL EXPOSURE TO BERYLLIUM AT DOE FACILITIES

Presented by

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for

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Office of Operational Safety
U.S. Department of Energy

September 23, 1987
JOWOG 22B, Beryllium

1.0 INTRODUCTION

This report presents the results of special reviews of beryllium operations to gather facts on the adequacy of the Department's programs for the evaluation and control of occupational exposure to beryllium. The special review was initiated by the Assistant Secretary for Environment, Safety, and Health.

This report is addressed to all DOE operating contractors that conduct beryllium operations. It is intended to describe the strengths and weaknesses of occupational health programs observed during this review and to provide guidance on acceptable practice for the evaluation and control of occupational exposure to beryllium. The beryllium operations visited during this review were at the Rocky Flats Plant, the Y-12 Plant, and Lawrence Livermore National Laboratory.

An Executive Summary of the conclusions and recommendations of EH is contained in Section 2 of the report. The findings and discussion obtained during facility visits are contained in sections 3, 4, and 5. Section 6 contains the overall conclusions about the strengths and deficiencies of DOE industrial hygiene and occupational medicine programs for the evaluation and control of occupational exposure to beryllium. Section 7 proposes a potential DOE-wide program for the evaluation and control of occupational exposure to beryllium.

2.0 EXECUTIVE SUMMARY

This report presents the results of special reviews of beryllium operations to gather facts on the adequacy of the Departments programs for the evaluation and control of occupational exposure to beryllium. This special review was initiated by the Assistant Secretary for Environment, Safety, and Health.

The review was undertaken by a team of occupational health specialists visiting the three major user facilities. The team was made up of industrial hygienists from: the Office of Operational Safety; the Albuquerque, Oak Ridge, and San Francisco Operations Offices; and Los Alamos National Laboratory and by occupational medicine physicians from the Office of The Deputy Assistant Secretary for Safety, Health, and Quality Assurance and Oak Ridge Associated Universities; and was led by the Director of the Office of Operational Safety. These visits were followed-up by industrial hygiene surveys to develop an evaluation of the degree of occupational exposure. Team members participated in an extensive survey being conducted at the Rocky Flats Plant and conducted brief independent surveys at the Y-12 Plant and at Lawrence Livermore National Laboratory.

SITE DESCRIPTION

Reviews were conducted at three of DOE's major beryllium user facilities: the Rocky Flats Plant, Golden, Colorado, (operated by Rockwell International); the Y-12 Plant, Oak Ridge, Tennessee, (operated by Martin Marietta Energy Systems); and Lawrence Livermore National Laboratory, Livermore, California, (operated by the University of California).

The Rocky Flats Plant fabricates beryllium metal components for nuclear weapons. The operations consist principally of machining metal pieces which have been purchased from a vendor. The finished pieces are handled by workers involved in quality assurance and assembly activities. The beryllium machine shop employs about 30 machinists and is located in building 444.

The Y-12 Plant fabricates beryllium oxide ceramic components for nuclear weapons. The principal operation consists of diamond grinding, high precision machining of ceramic pieces which are primarily purchased from a vendor. The ceramic hot press operation is a routinely functioning reserve manufacturing capability where beryllium oxide powder is mixed with other compounds, pressed into the desired shape and fired in a kiln. A third, remotely operated glove box production operation is also performed at Y-12. There are approximately 15 employees working in these beryllium shops which are located in building 9201-5E and the Butler annex.

The Lawrence Livermore National Laboratory fabricates beryllium metal components for experimental nuclear explosives devices. This is a typical laboratory facility where machinists work approximately half time at fabricating beryllium parts. There are approximately 4 employees who machine beryllium in building 321. There is also a destructive testing operation associated with development of new devices in which beryllium components are included in a non-nuclear package that is destroyed using high explosives. This testing takes place at site 300.

SUMMARY OF CONCLUSIONS

In general, state-of-the-art engineering controls are being applied to DOE operations that generate beryllium aerosols. However, the studies performed to date indicate that worker exposures near or above the permissible exposure limit are occurring at some locations as a result of short term excursions from hand work or opening hoods and gloveboxes. As a result, beryllium concentrations can be higher in the workers' breathing zone than anywhere else in the workroom. These workers are required to wear respirators.

Work places are being monitored using three types of sampling. Personal breathing zone sampling is being used to estimate exposure and assure compliance with exposure limits. Fixed head area sampling is used to assure that hoods and other control equipment are continuing to perform adequately. Since area samples are collected continuously, they also provide information in the event of an accidental release. Swipe sampling is being performed to assure that house keeping practices are adequate to prevent the spread of beryllium surface contamination.

While there is some variation in methods used by DOE contractors to analyze beryllium samples and in the quality control programs applied to those analyses, these variations are not creating any detrimental effect on the accuracy of exposure estimates. Inaccuracies inherent in collection of personal samples and the time varying nature of occupational exposure create much greater uncertainty than those associated with analytical procedures.

Operating procedures and safety rules have been established for beryllium work at all DOE facilities visited. These procedures require the use of protective clothing, limit access to authorized personnel, and require review of all non routine operations. The Rocky Flats Plant has authorized a large number of employees (2000 since 1984) to have access to beryllium shops mostly to perform maintenance and cleaning jobs.

Appropriate personal protective equipment is being provided at the DOE facilities visited when employees are potentially exposed to beryllium. The respiratory protection program reviewed meet DOE requirements for fitting, training, and medical clearance of respirator users and for selection and maintenance of equipment.

Effective programs are in place to minimize the potential for ingestion of beryllium and to control contamination of surfaces with beryllium. Eating, drinking, and smoking in controlled areas is prohibited or limited. All the facilities visited have swipe sampling programs to assure that work rooms and surfaces are kept clean, and to assure that tools and work pieces leaving controlled areas are clean.

The definition for the purpose of medical follow-up of a beryllium worker was found to have varied within and between plants over time. There are no protocols for the medical evaluation of workers being considered for employment in or transfer into beryllium operation areas. Although prior diagnosis of chronic lung disease precludes employment in a beryllium area, there are no specific standards of health or fitness which a beryllium worker must meet. Information on possible past exposure to beryllium is collected in a relatively casual fashion.

Annual medical examinations with pulmonary function testing, chest x-radiograph, and physical examinations are either required or "offered" to active beryllium workers; active employees who formerly were identified as beryllium workers are followed according to the routine periodic medical examination protocol for non-beryllium workers. Only recently has the requirement for a B reader chest x-radiograph interpretation been added at the Rocky Flats Plant. No location has any follow-up program on former beryllium workers who have retired or left employment. There is no agreement as to the definition of a beryllium worker so follow-up varies greatly from location to location.

The OSHA Hazard Communication Standard (29 CFR 1910.1200) creates standard requirements for an acceptable program and compliance with those requirements is policy at all facilities visited. Beryllium workers have always received on-the-job training but the requirement for formal, documented, specialized training has not been implemented at all locations. Where formal training has not yet been provided there are plans to provide the required training.

Signs and placards present at the Y-12 Plant carried the warning "suspect carcinogen" as required by OSHA standards. However, the principal hazard of beryllium is its ability to cause chronic lung disease. Employees interviewed found the warning confusing.

At the facilities visited, sample analysis results are routinely sent to line management and posted or distributed to make them available to the workers involved. The distribution and discussion of sample results is an effective method of identifying work practices that lead to excursions or the spread of contamination and developing methods of reducing exposures. While historical data is not always readily available, current monitoring data and medical records are being managed in a manner that makes the records easily searchable and retrievable. Monitoring data along with recommendations for correction of deficiencies are routinely reported to line management for action as appropriate. There is a lack of internal audits or other reporting to upper management on continuing effectiveness of programs to evaluate and control occupational exposure to beryllium.

OVERALL RECOMMENDATIONS

- Where practicable, efforts should be made to eliminate or mechanise hand work and operations that require hoods to be open.
- Increased emphasis should be placed on collecting personal samples and especially short-term personal samples to identify work practices that result in short-term excursions. Beryllium workers who are routinely exposed should be monitored at least quarterly.
- Steps should be taken to assure the credibility and quality of beryllium analyses. These include: the use of National Bureau of Standards traceable material to calibrate detectors; accreditation by the American Industrial Hygiene Association; and participation in a round-robin exchange of spiked samples among DOE contractors.
- At the Rocky Flats Plant there needs to be an administrative method developed to limit the number of maintenance personnel and janitors allowed access so that all potentially exposed personnel can be followed medically.

- In order to develop an adequate medical surveillance program, a precise definition of "beryllium worker" must be determined that can be applied DOE-wide, and former beryllium workers still employed must continue to be included in the medical surveillance program.

- A protocol must be developed describing the methods used to follow former or current beryllium workers which: detects the earliest signs of respiratory disease; calls attention to the importance of meticulous evaluation of respiratory symptoms; and calls for the performance and specific review of pulmonary function tests and chest x-radiograph examinations.

- Hazard labels and signs should carry the warning "causes lung disease" in addition to the warning "suspect carcinogen" to assure that the message that beryllium is highly toxic is communicated clearly.

- The continuing effectiveness of programs and procedures to evaluate and control occupational exposure to beryllium should be reviewed periodically in a manner that conforms to the requirements of DOE 5482.1B, paragraph 9.d. Internal Appraisals.

- The Department needs to implement a DOE-wide program for the evaluation and control of occupational exposure to beryllium.

INDUSTRIAL HYGIENE ASPECTS OF VARIOUS BERYLLIUM FORMING OPERATIONS

T. F. Lewis, RFP

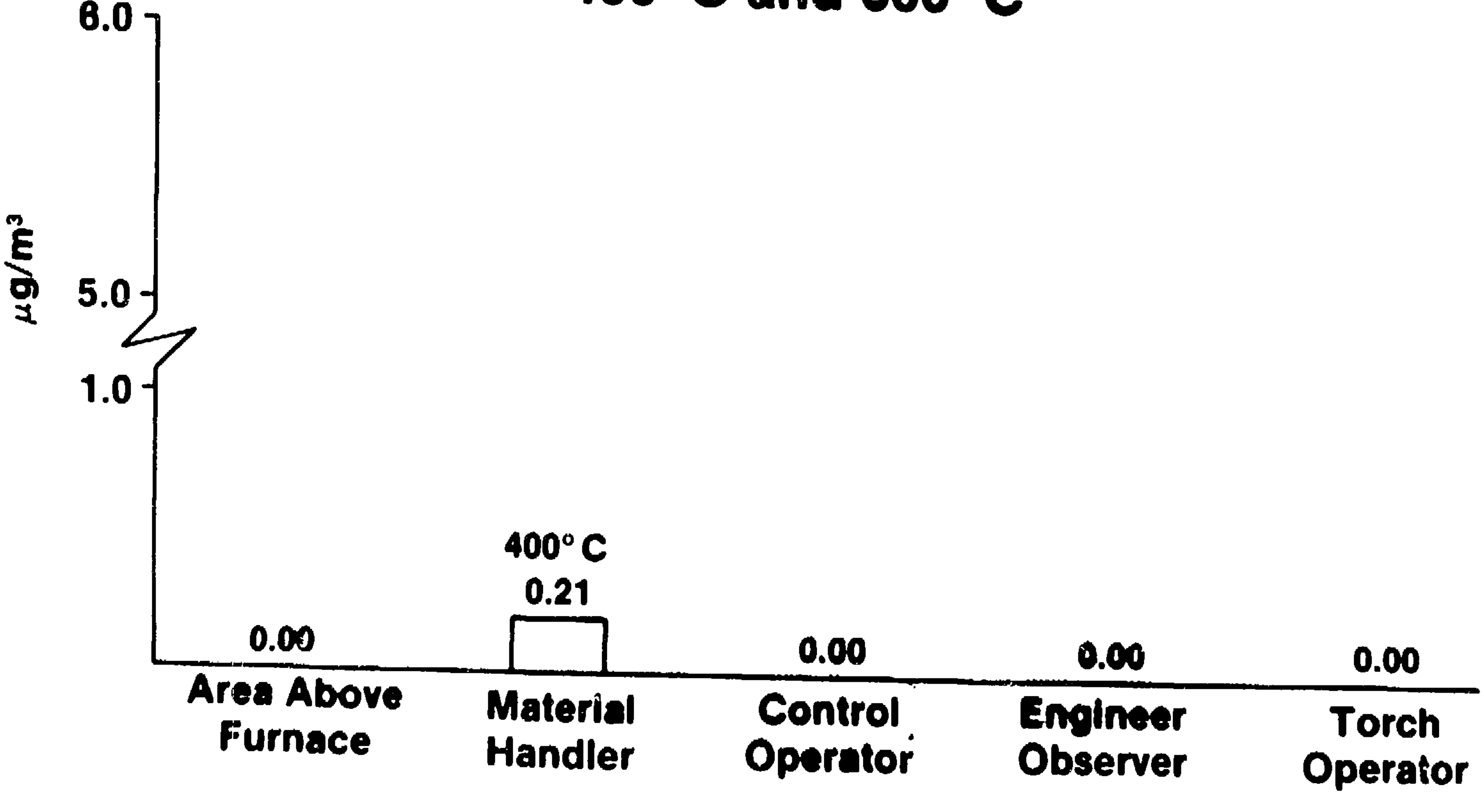
Abstract

An industrial hygiene study was conducted during casting, spinforming, and reshaping of beryllium metal. The goal of the study was to determine if these processes released any beryllium aerosol, and if so to what extent. Data was collected utilizing traditional personal air sampling in conjunction with high volume (17 LPM) diagnostic sampling. During casting operations 2 of the 4 major steps were identified as major generators of beryllium aerosol. Modified work procedures and engineering control will be incorporated to reduce beryllium aerosols. Preliminary data from limited reshaping operations indicate levels below 1 ug/m3. All available data from beryllium hydrospinning operations will also be presented.

Rockwell International

Spin Forming Project

400° C and 600° C

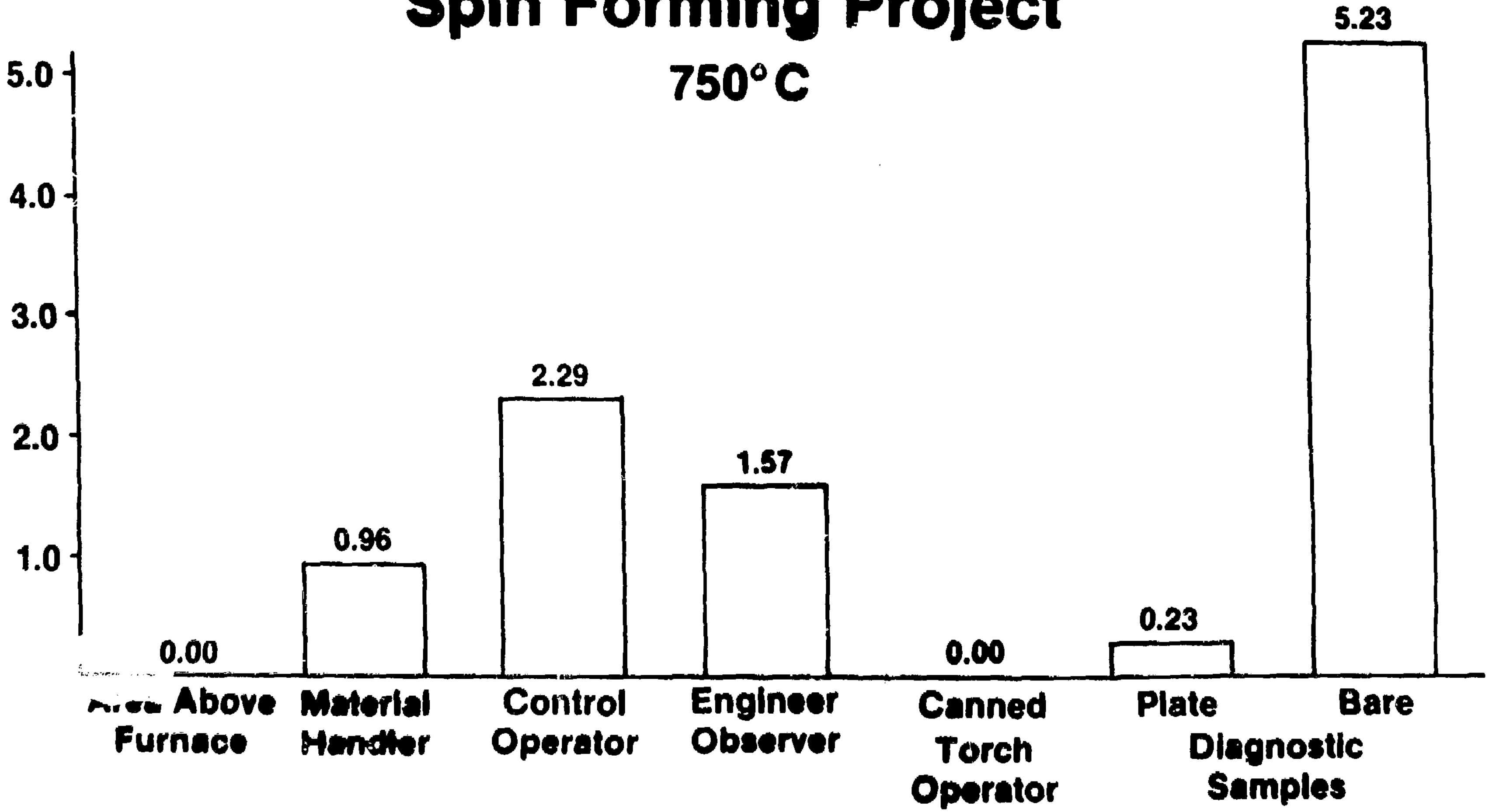


*Min. detectable amount at 95% confidence is 0.10 µg

Rockwell International

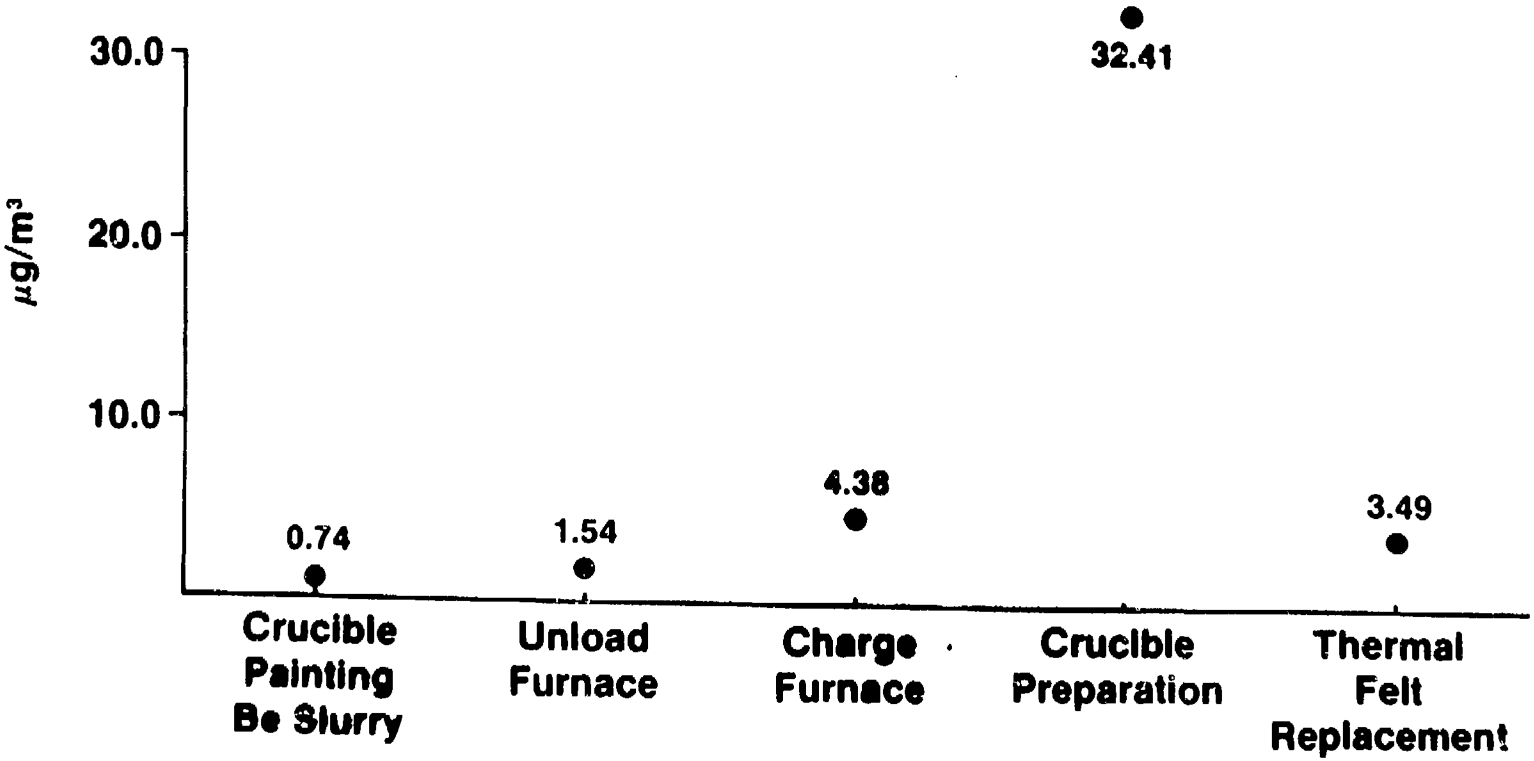
Spin Forming Project

750° C



*Min. detectable amount at 95% confidence is 0.10 µg

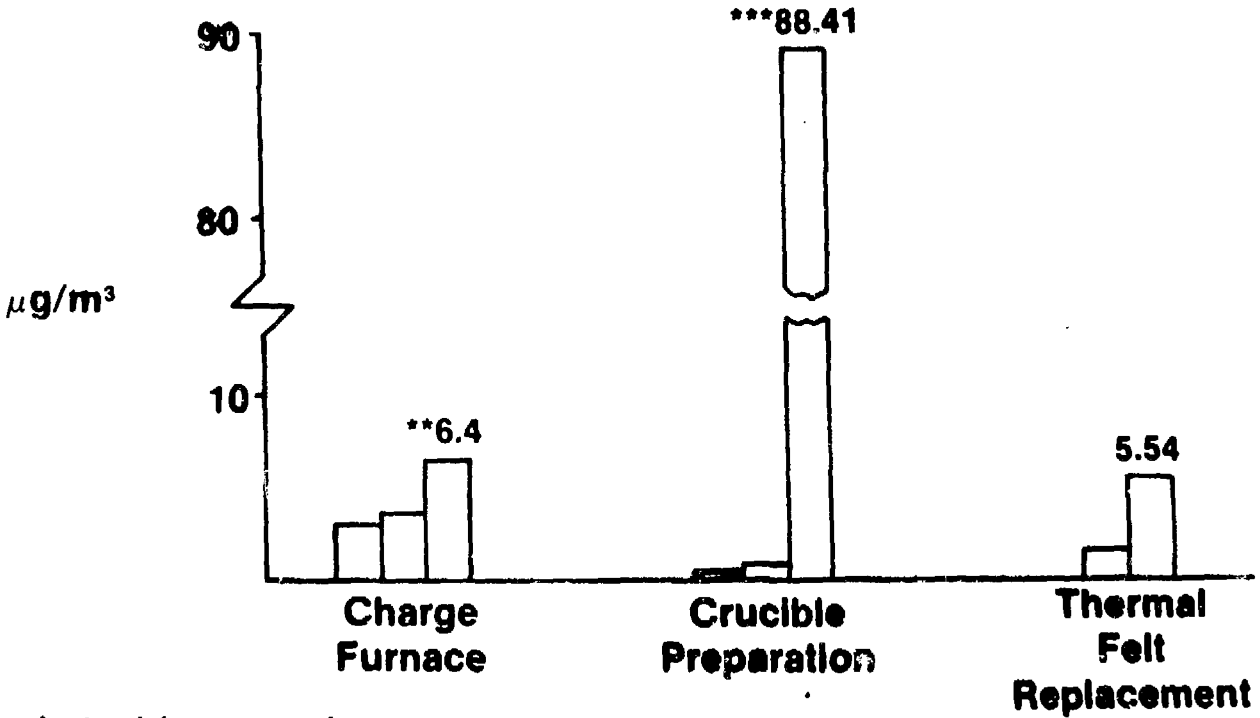
Beryllium Casting Project



*Min. detectable amount at 95% confidence is 0.10 µg

Rockwell International

Be Casting



***Hood at widest opening.

**Be contaminated thermal felt was reused.

*Min. detectable amount at 95% confidence is 0.10 µg

A REVIEW OF BERYLLIUM MONITORING DATA AT R.O.F. CARDIFF

by

G.Cogbill (ROF Cardiff).

Summary

An extensive sampling programme is run to ensure the safety of the staff employed in the facility. The work utilises a combination of static and personal air samplers together with smear testing for assessing surface contamination levels. Several years data are reviewed and the results from various work areas are compared.

Cardiff is the capital city of Wales and is situated approx 160 miles West of London.

The ROF or more correctly AWE(C) is to be found on the Northern outskirts of the city and occupies a site within a busy residential suburb.VU-FOIL(1)

The Beryllium facility within the establishment plays a very important role in the production of Be components in aid of the UK nuclear programme.

The facility is one of which we are duly proud, having opened in 1960 and subsequently extended in 1975 ____ hence during my talk I may refer to parts of the facility as "OLD" or "NEW".

During the last 25 years or so we have had only one serious medical incident. In 1964 an operator sustained a cut on a grinding wheel impregnated with BeO. The resultant wound necessitated a finger amputation.

It is by Brush Wellman standards a small facility employing about 100 people of mixed disciplines, although less than half of that number machine metal.The building itself is self-sufficient and is fully ventilated___during normal conditions the air is changed 15-20 times per hour, maintaining the shops at a slight negative pressure

The staffing arrangements covering Safety at the establishment are shown on the VU-FOIL(2)

Some of you will already have visited our work-shops but for those who haven't the next, VU-FOIL(3)shows the layout of the various activities.

I would now like to talk generally about our approach to monitoring ____ there are three specific areas of interest.

STATIC OR AREA SAMPLING

PERSONAL AIR SAMPLING

SMEAR OR WIPE TESTS VU-FOIL(4)

STATIC

For static monitoring we use 30 l/min. pumps which are run for the duration of the shift. They are strategically positioned VU-FOIL(5) in order to give representative results of conditions throughout the shops.

Be analysis is not unfortunately instantaneous, though much work has been done on this topic so consequently in selected areas we do mid morning or core samples. The purpose being to minimise the period of time the workforce are exposed to unknown conditions. The core samples and a similar batch taken at the end of the shift are immediately analysed, normal samples are analysed the next day. Currently we are running about 75 static samplers daily. The method of analysis for all our samples is Atomic Absorption.

PERSONAL AIR SAMPLING

Turning to personal air sampling,our approach has changed somewhat in that from the beginning of 1986 we made the wearing of PAS mandatory for all regular workers in the facility,covering all but infrequent visitors.The effect of this change has influenced some of our PAS results as I will show later.It is worth mentioning at this stage that the PAS results are those obtained by personnel during their daily work-------some of the time including periods spent whilst wearing respiratory protection.Therefore the results reflect the worst case situation not necessarily the true exposure.We have done some limited work involving the use of two samplers per man and also instructing operators to switch off samplers whilst masked but the results were inconclusive----maybe your thoughts on this anomaly could be discussed later.

Supplementing our air monitoring programme. we conduct an extensive wipe test programme--- routinely taking some 2000 samples per month

We set ourselves the following targets VU-FOIL(6)

I would now like to talk about detailed monitoring results that we have obtained over the last 5years or so:-

STATICS

Looking first at some annual data VU-FOIL(7)

Powder stages refers to VACUUM HOT PRESSING.IMPACT MILLING.CASTING.CIP/HIP.

Similarly for 1985 VU-FOIL(8) and 1986 VU-FOIL(9)

Further back, data in a slightly different format reveals similar trends VU-FOILS(10.11.12.13)

One specific area we have looked at is the Vacuum press shop.and some data is shown on the next VU-FOIL(14)

PERSONAL AIR SAMPLING

The following information covers the last 5 years and is divided to differentiate those personnel working in specific areas.You may recall that I mentioned that the results do not take into consideration periods of time when personnel may have been wearing a respirator VU-FOILS(15.16.17)

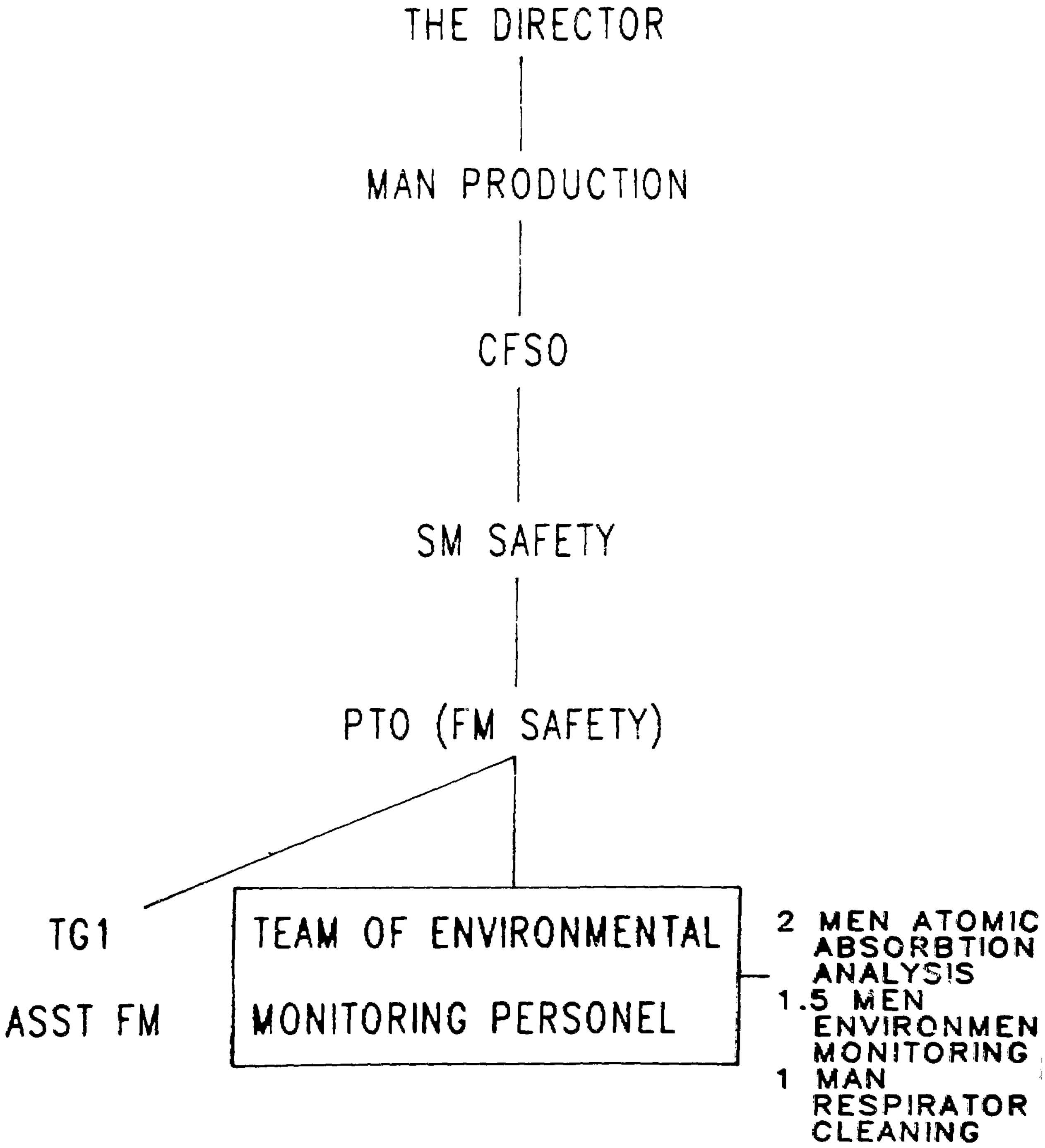
The mandatory introduction in 1986 of PAS has affected the FOUNDRY data.

SMEAR/WIPE TEST DATA

VU-FOILS 18 OMS/NMS ABOVE TARGET COMPARISON
19 OMS FREQUENCY BREAKDOWN
20 NMS FREQUENCY BREAKDOWN
21 CASTING FREQUENCY BREAKDOWN
22 PRESS SHOP FREQU. BREAKDOWN
23 COMPARISON CASTING/PRESSING
24 ABOVE TARGET PLASMA SPRAY
25 PLASMA SPRAY FREQU BREAKDOWN
26 IMPACT FREQUENCY BREAKDOWN

To date we have not done much work on correlation of the data I have presented.However,we have confidence in our programmes since without exception,each time a high static reading has been obtained we have been able to establish,on investigation,that something abnormal had happened,and that subsequent wipe tests would be high.Probably because of the relatively low flow rate of PAS(21/min) the effect of the abnormality would be quite dependent on the location of personnel within the affected area.

AWE(C) SAFETY DEPARTMENT STRUCTURE (Be)



THE DIRECTOR

MAN PRODUCTION

CFSO

SM SAFETY

PTO (FM SAFETY)

TG1

ASST FM

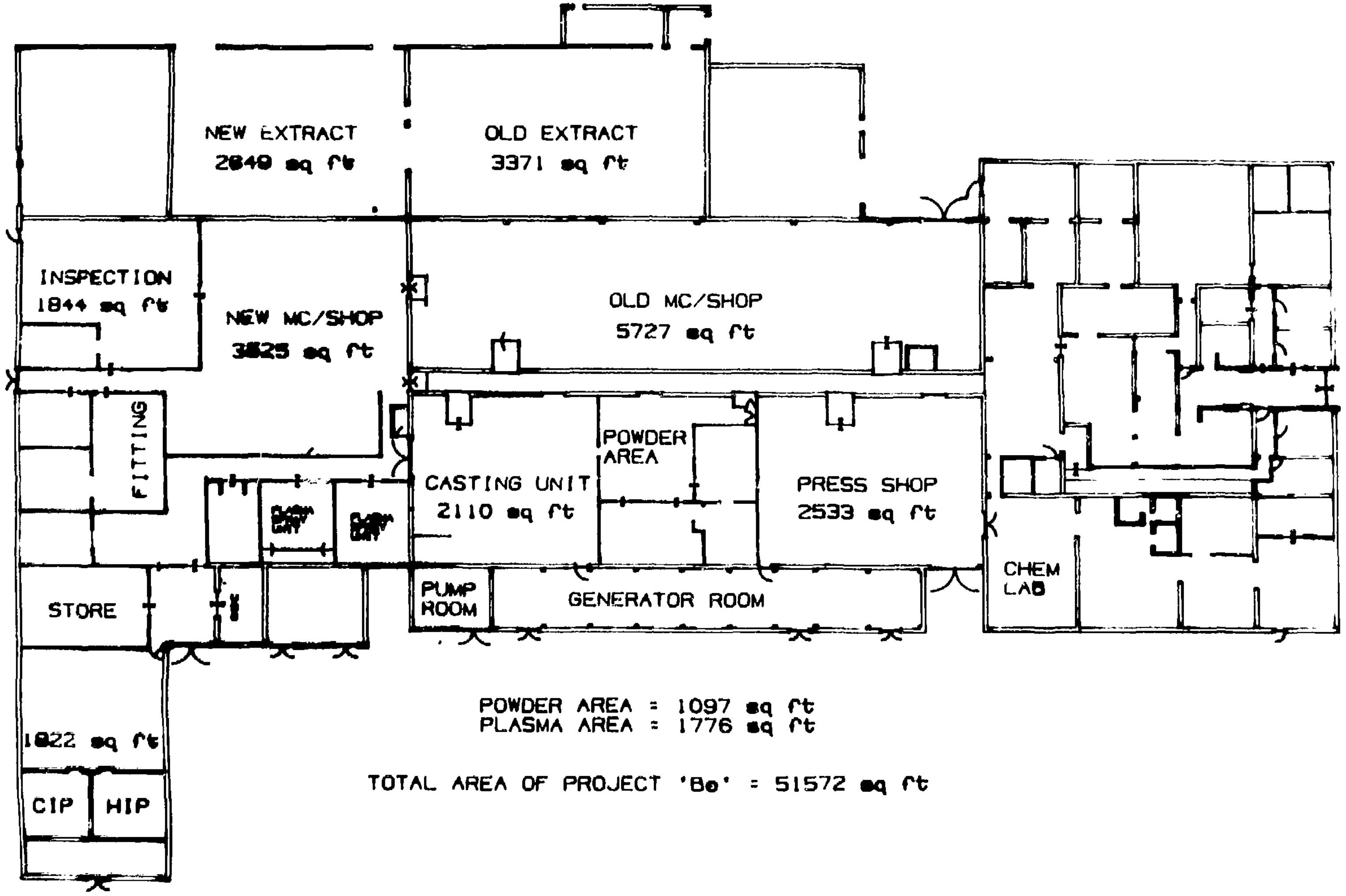
TEAM OF ENVIRONMENTAL MONITORING PERSONEL

2 MEN ATOMIC ABSORBTION ANALYSIS

1.5 MEN ENVIRONMEN MONITORING

1 MAN RESPIRATOR CLEANING

PLAN VIEW
PROJECT 'Be'



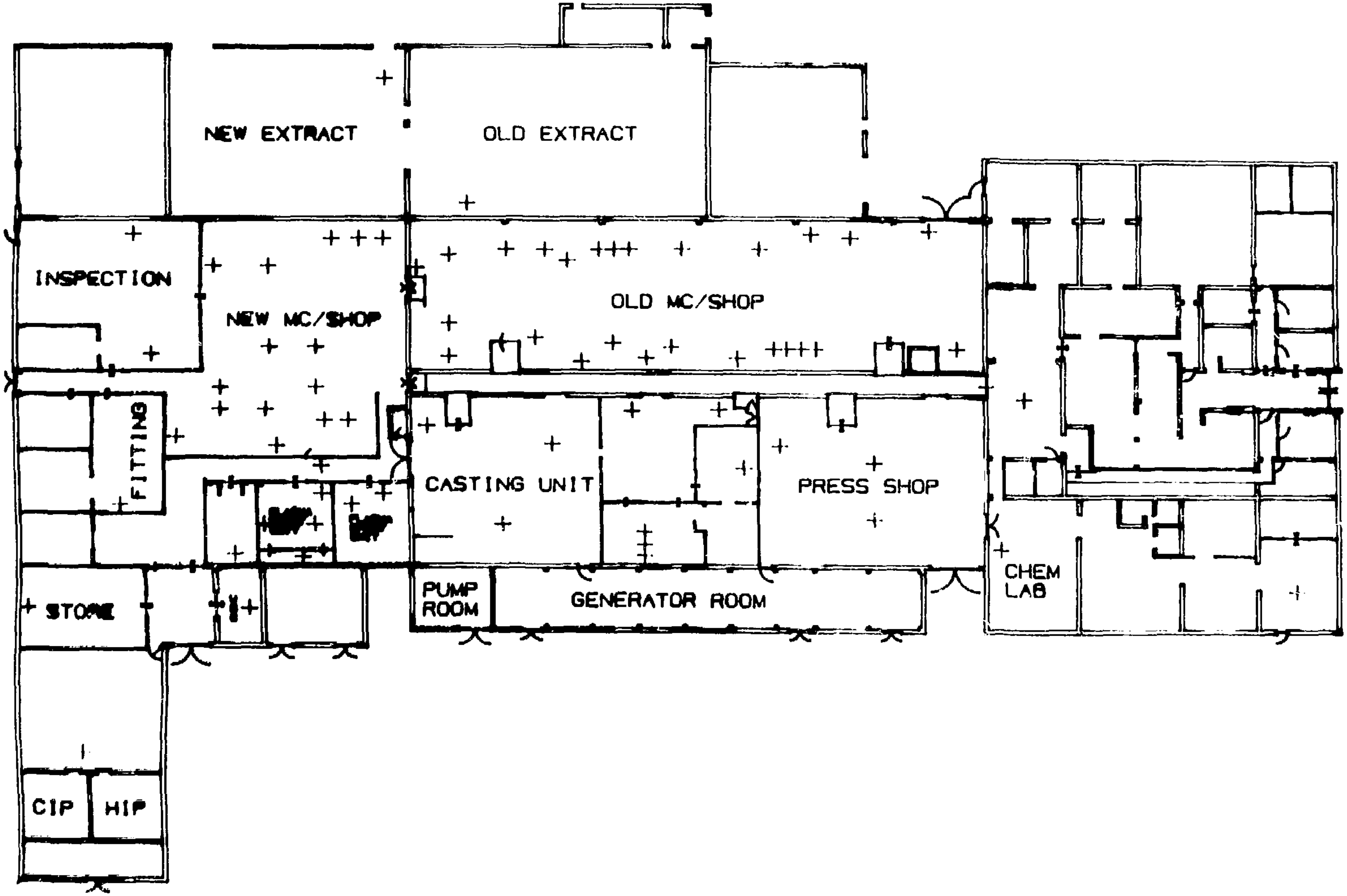
POWDER AREA = 1097 sq ft
PLASMA AREA = 1776 sq ft

TOTAL AREA OF PROJECT 'Be' = 51572 sq ft

MONITORING

1. STATIC OR AREA SAMPLING
2. PERSONAL AIR SAMPLING
3. SMEAR OR WIPE TESTS

PLAN VIEW
PROJECT 'Be'



TARGET LEVELS (MICRO GMS./SQ.FT).

MACHINE SHOPS, FOUNDRY ETC. < 25

LABORATORIES, METROLOGY ETC. < 10

ITEMS LEAVING AREA < 1

ANNUAL SUMMARY OF BERYLLIUM AIR SAMPLES (STATIC) - 1984 RESULTS

	µg Per Metre Cubed			
	< 0.2	0.2-1.0	1.0-2.0	> 2.0
Old Machine Shop	98%	1.79%	0.09%	0.12%
New Machine Shop	99%	0.89%	0.19%	0.01%
Powder Stages	74%	22.0%	2.50%	1.50%
Plasma Spray	80.5%	16.0%	1.30%	2.20%

Annual Summary of Beryllium Air Samples (Static) 1985 Results

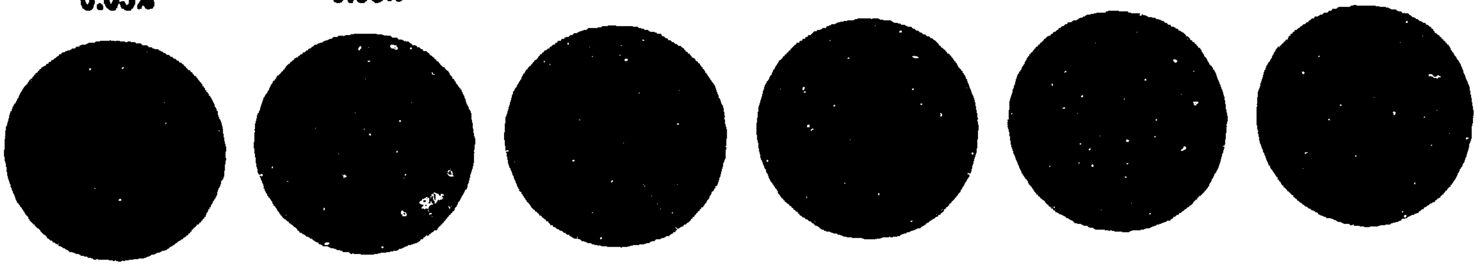
	ug Per Metre Cubed			
	<0.2	0.2–1.0	1.0–2.0	>2.0
Old Machine Shop	97.6%	2.13%	0.22%	0.05%
New Machine Shop	99.3%	0.44%	0.03%	0.23%
Powder Stages	71.2%	24.3%	3.0%	1.5%
Plasma Spray	81.8%	15.17%	1.54%	1.49%

Annual Summary of Beryllium Air Samples (Static) 19[illegible] Results

	µg Per Metre Cubed			
	<0.2	0.2–1.0	1.0–2.0	[illegible]
Old Machine Shop "7565"	99.24%	0.73%	0.02%	[illegible]
New Machine Shop "5874"	99.69%	0.28%	0.03%	[illegible]
Powder Stages "4428"	75.13%	18.4%	4.01%	[illegible]
Plasma Spray "3189"	96.29%	2.66%	0.21%	[illegible]

Be STATIC AIR SAMPLES

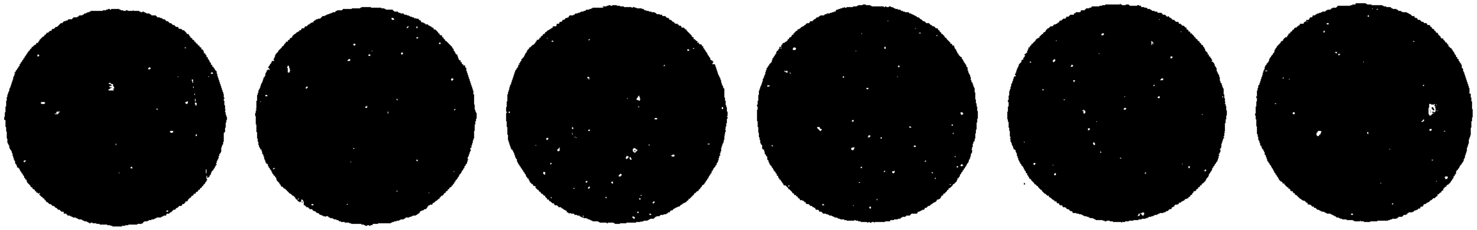
	(>=2.0)	(>=2.0)	(>=2.0)	(>=2.0)	(>=2.0)	(>=2.0)
	0.05%	0.03%	0.1%	0.12%	0.05%	0.01%



YR	1981	1982	1983	1984	1985	1986
NO. OF SAMPLES	5270	5700	6330	6550	6310	7565

OLD M/C SHOP

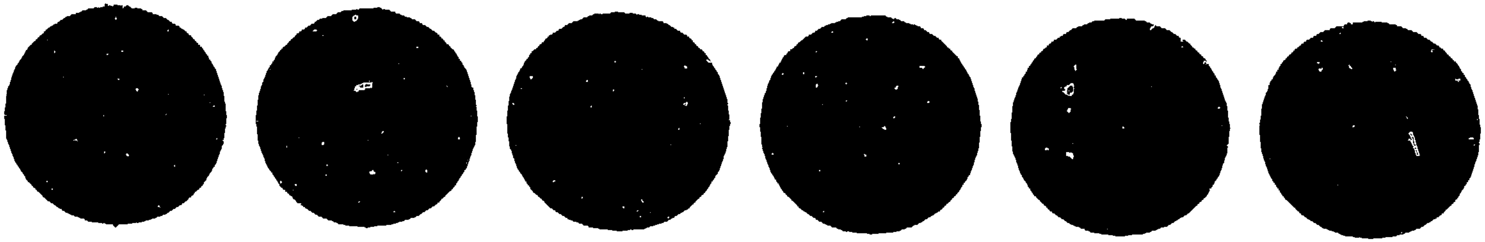
Be STATIC AIR SAMPLES



	(>=2.0)	(>=2.0)	(>=2.0)	(>=2.0)	(>=2.0)	(>=2.0)
	0.00%	0.02%	0.02%	0.01%	0.3%	0.%
YR	1981	1982	1983	1984	1985	1986
NO. OF SAMPLES	3175	3570	3840	4750	5580	5874

NEW M/C SHOP

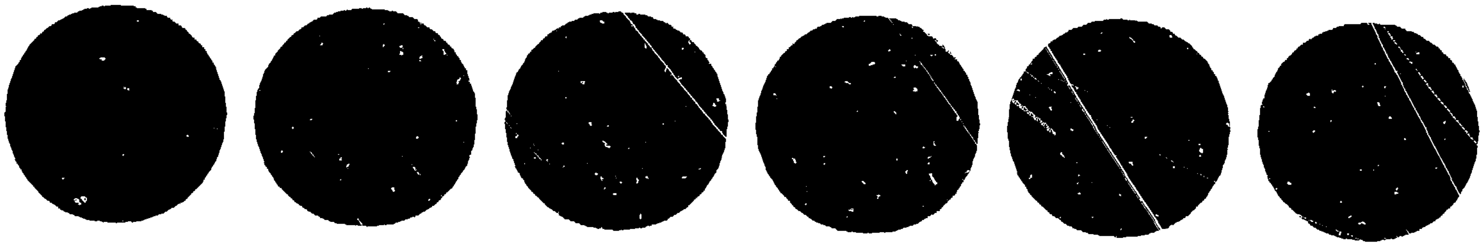
Be STATIC AIR SAMPLES



	(>=2.0)	(>=2.0)	(>=2.0)	(>=2.0)	(>=2.0)	(>=2.0)
	1.6%	2.5%	2.5%	1.5%	1.5	2.37%
YR	1981	1982	1983	1984	1985	1986
NO. OF SAMPLES	3300	3750	3770	3908	3460	4428

POWDER STAGES

Be STATIC AIR SAMPLES



	(>=2.0)	(>=2.0)	(>=2.0)	(>=2.0)	(>=2.0)	(>=2.0)
	1.0%	3.2%	0.9%	2.2%	1.5%	0.84%
YR	1981	1982	1983	1984	1985	1986
NO. OF SAMPLES	1340	1480	1710	2340	4020	3189

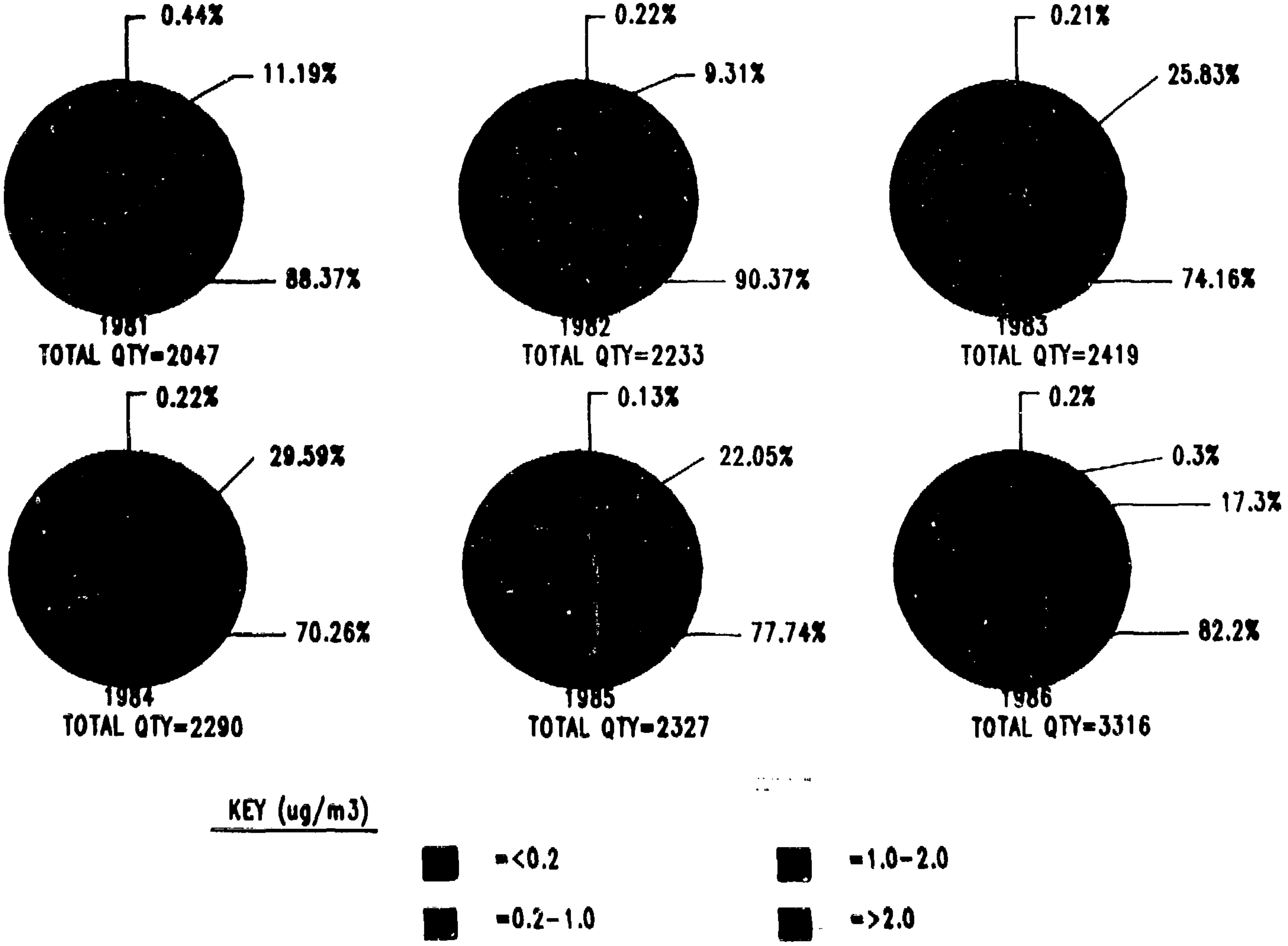
PLASMA SPRAY

Be PRESS SHOP AIR [illegible] 1981-1986
(ALL POTENTIAL [illegible] VALUES)

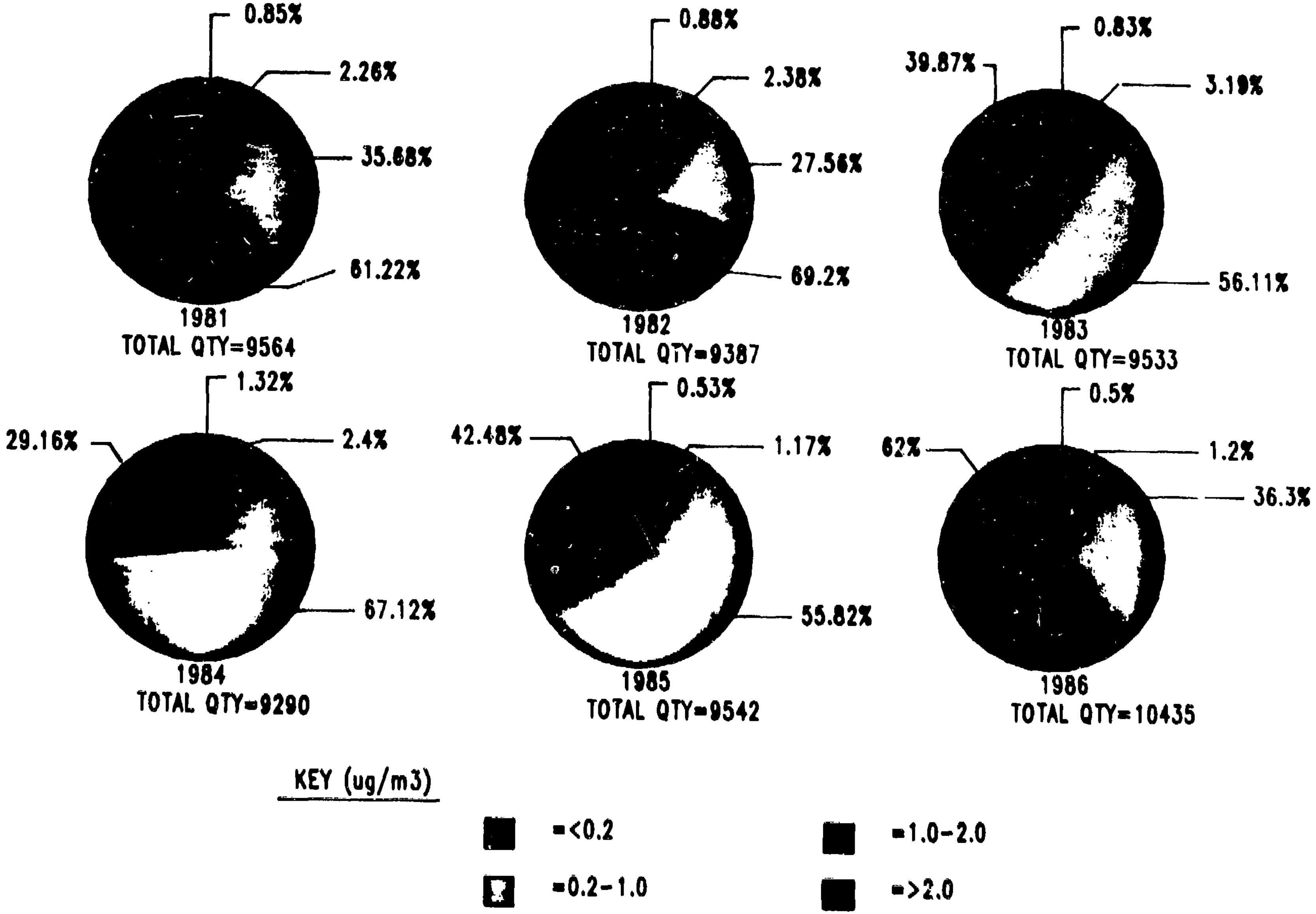
COMBINATION
POS 210,211 & 220

YEAR	TOTAL	<0.2	0.2 - 1.0	1.0 - 2.0	= >2.0
1981	821	32	53	10.7	4.3
1982	897	27.2	51.6	13.7	7.5
1983	880	27.4	48.5	17.3	6.8
1984	978	45.1	46.3	5.3	3.3
1985	961	36	55	7.1	1.9
1986	1114	36.1	44.4	13.4	6.1

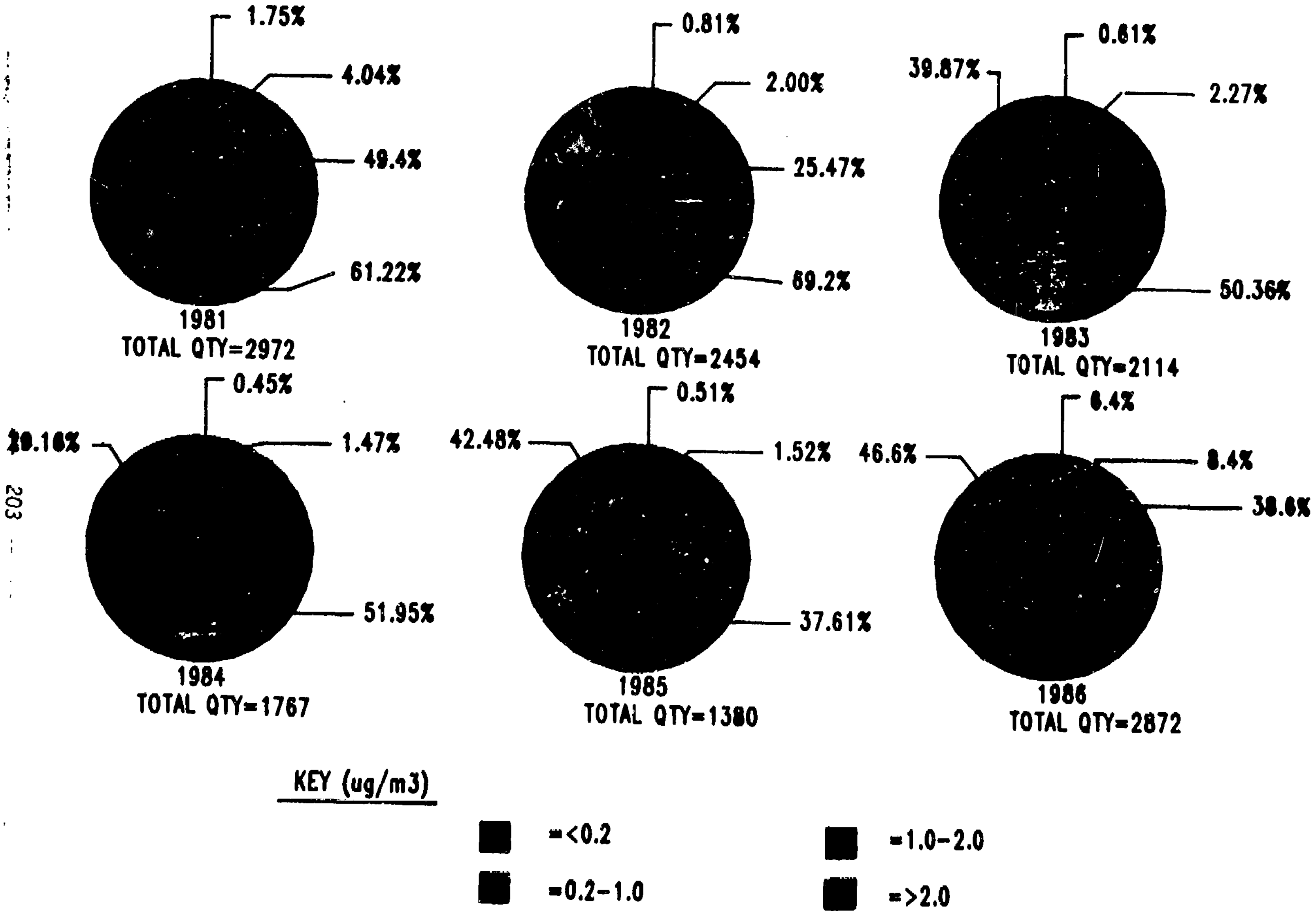
Be INSPECTION



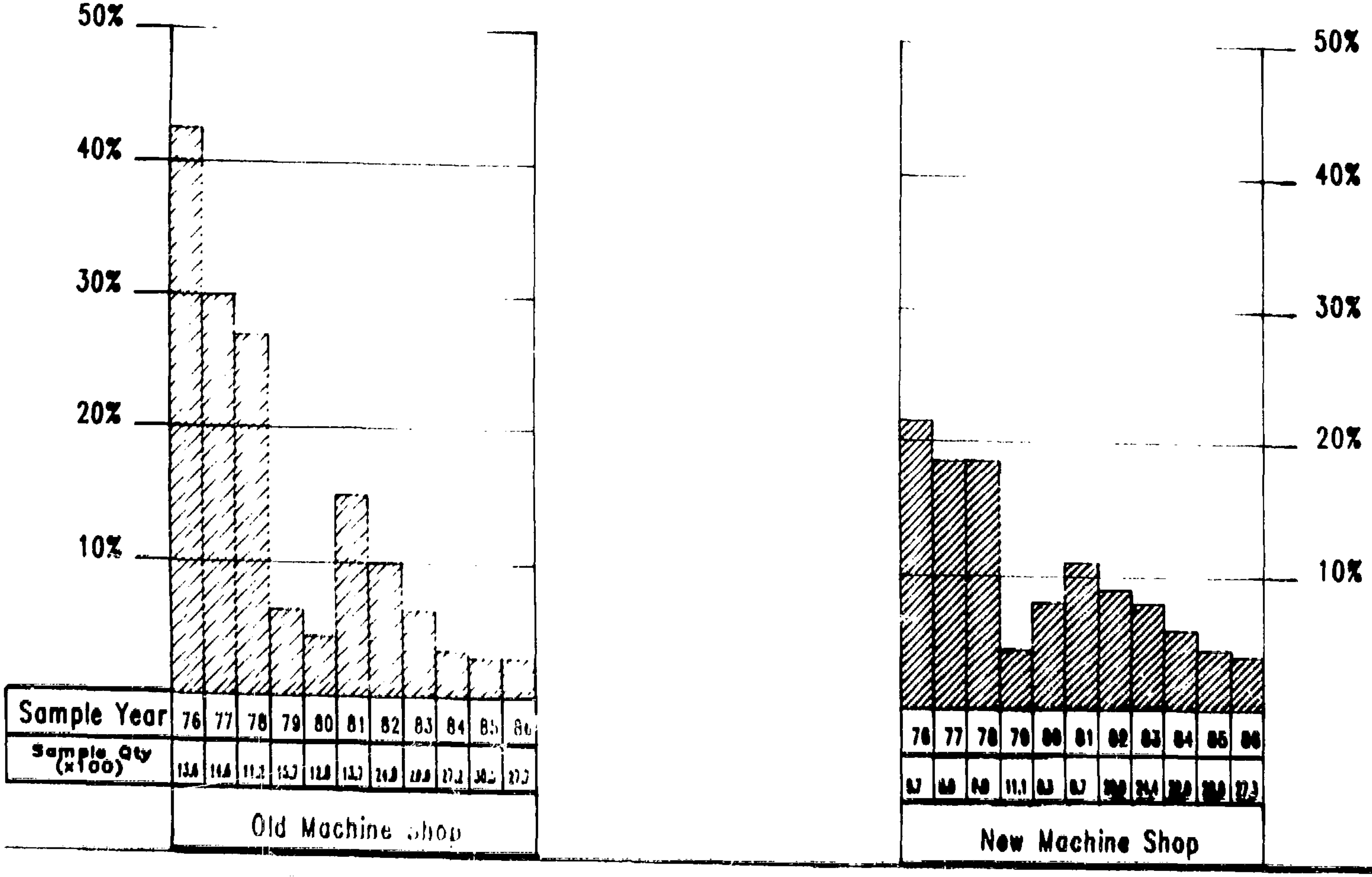
Be M/C SHOPS


0.85%
2.26%
35.68%
61.22%
1981
TOTAL QTY=9564
0.88%
2.38%
27.56%
69.2%
1982
TOTAL QTY=9387
39.87%
0.83%
3.19%
56.11%
1983
TOTAL QTY=9533
1.32%
29.16%
2.4%
67.12%
1984
TOTAL QTY=9290
0.53%
42.48%
1.17%
55.82%
1985
TOTAL QTY=9542
0.5%
62%
1.2%
36.3%
1986
TOTAL QTY=10435
KEY (ug/m3)
■ =<0.2
▨ =0.2-1.0
■ =1.0-2.0
■ =>2.0

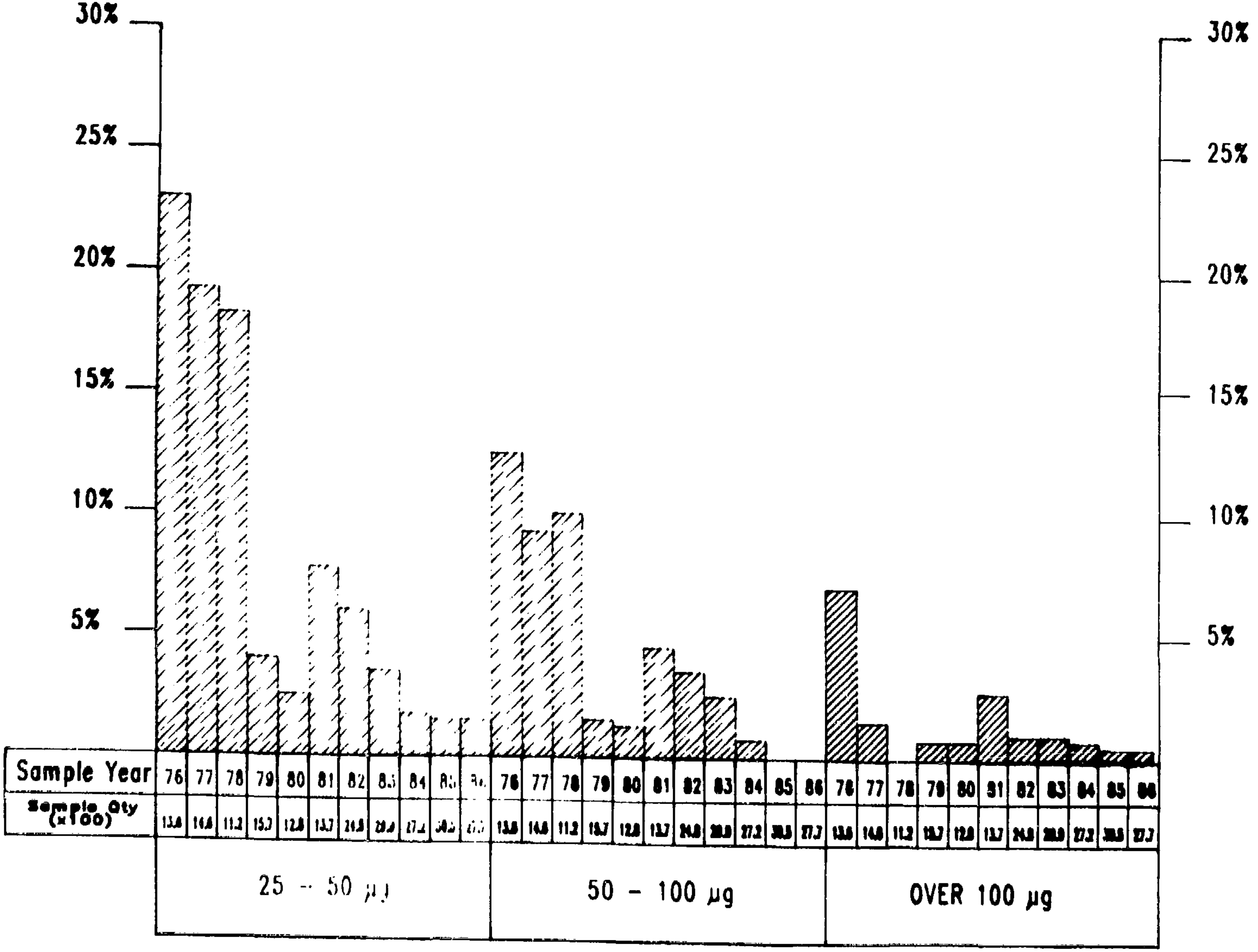
Be FOUNDRY


1.75%
4.04%
49.4%
61.22%
1981
TOTAL QTY=2972
0.81%
2.00%
25.47%
69.2%
1982
TOTAL QTY=2454
39.87%
0.61%
2.27%
50.36%
1983
TOTAL QTY=2114
20.16%
0.45%
1.47%
51.95%
1984
TOTAL QTY=1767
42.48%
0.51%
1.52%
37.61%
1985
TOTAL QTY=1380
46.6%
6.4%
8.4%
38.6%
1986
TOTAL QTY=2872
KEY (ug/m3)
=<0.2
=0.2-1.0
=1.0-2.0
=>2.0

Be SMEAR SUMMARY 1976 to 1986
(Readings greater than 25 $\mu g/ft^2$)

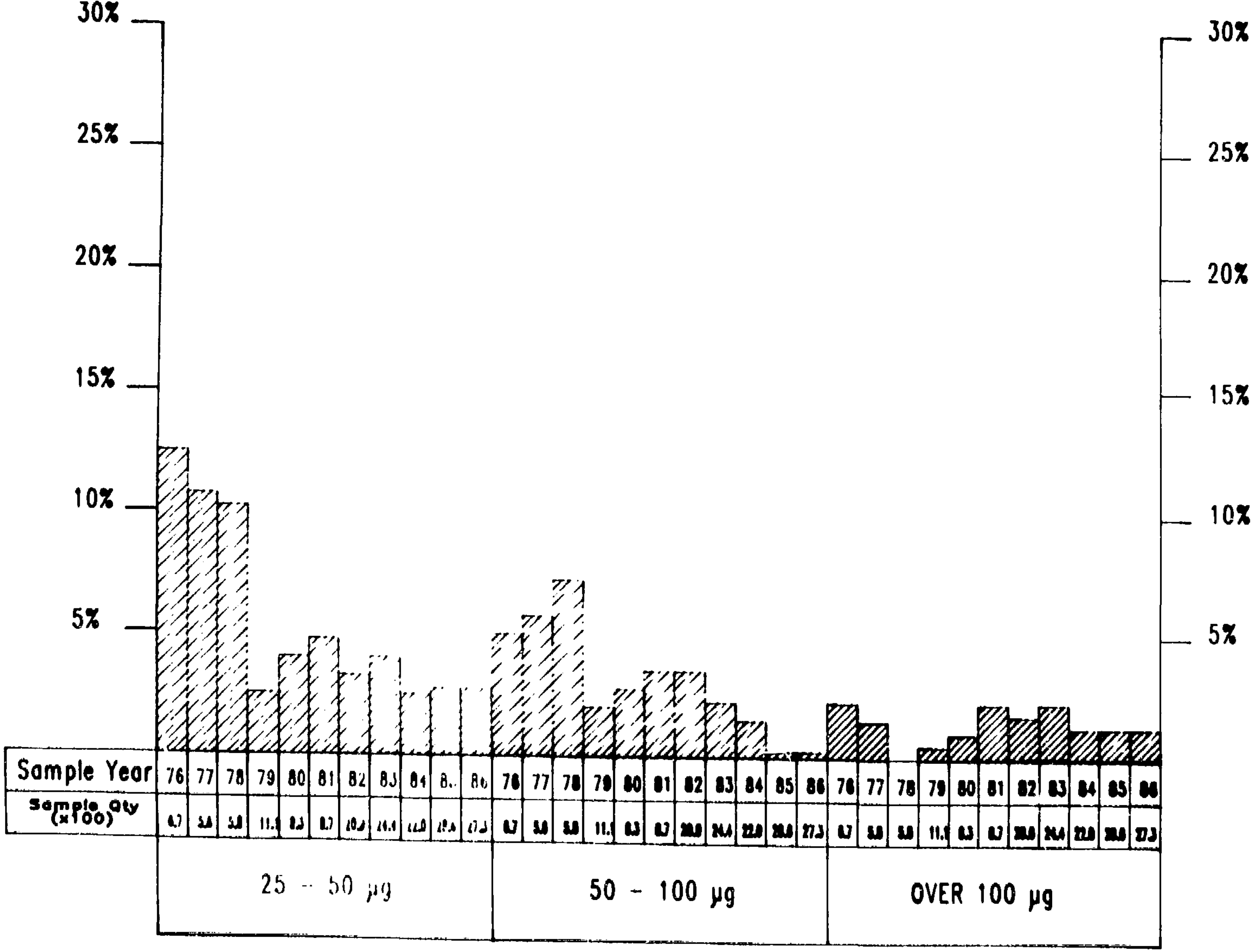


Be SMEAR SUMMARY 1976 to 1986



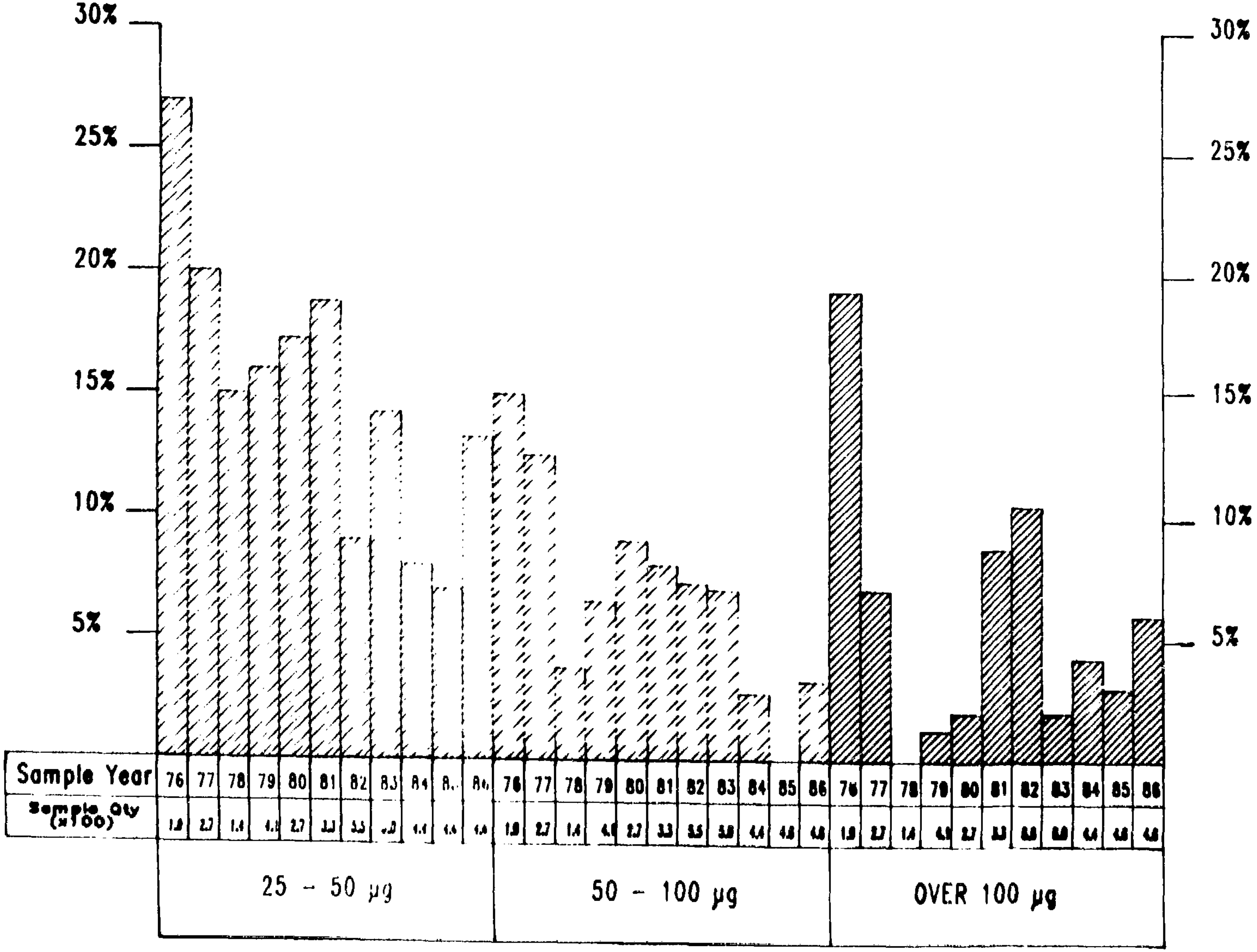
OLD MACHINE SHOP

Be SMEAR SUMMARY 1976 to 1986



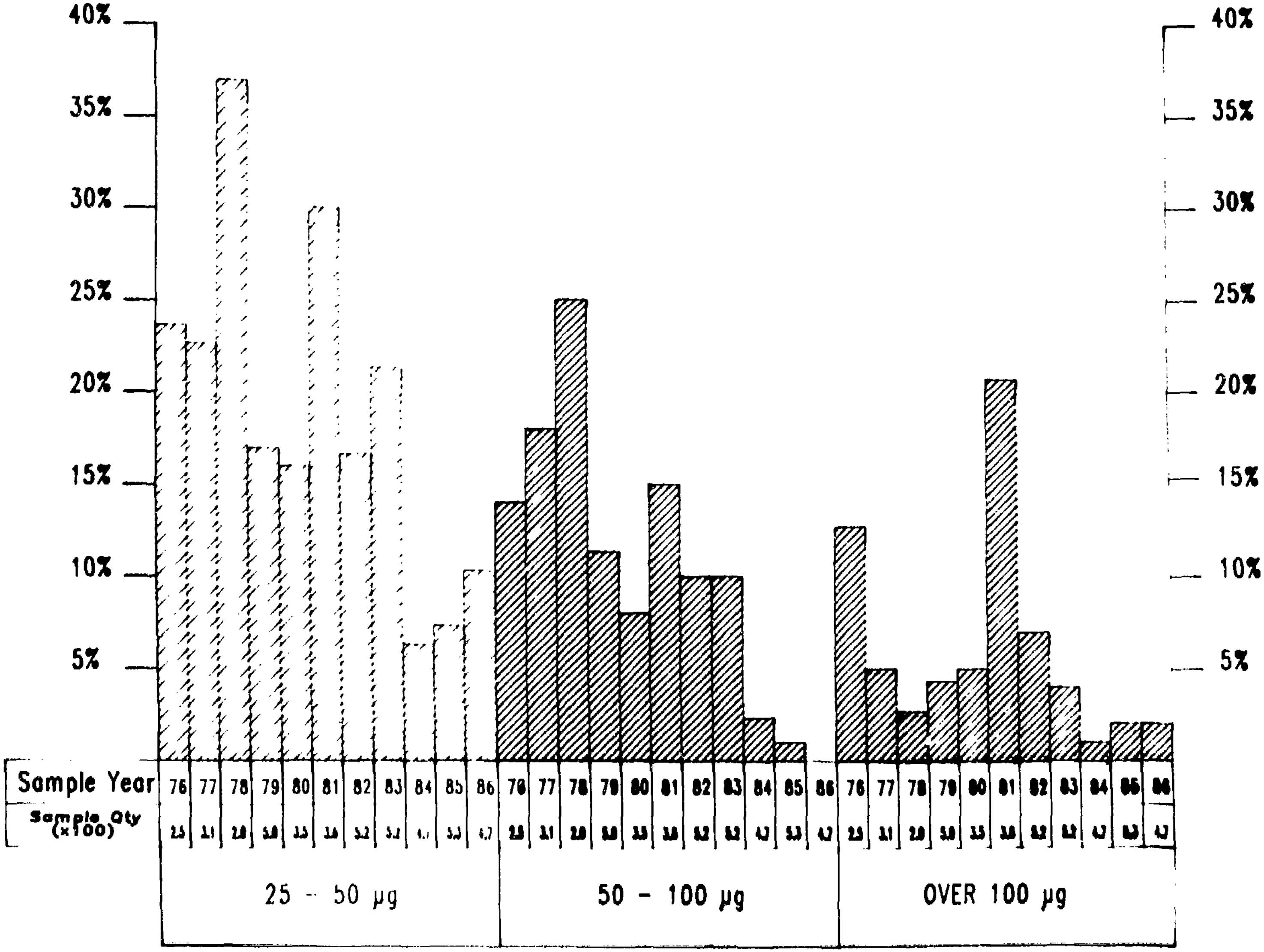
NEW MACHINE SHOP

Be SMEAR SUMMARY 1976 to 1986



CASTING SHOP

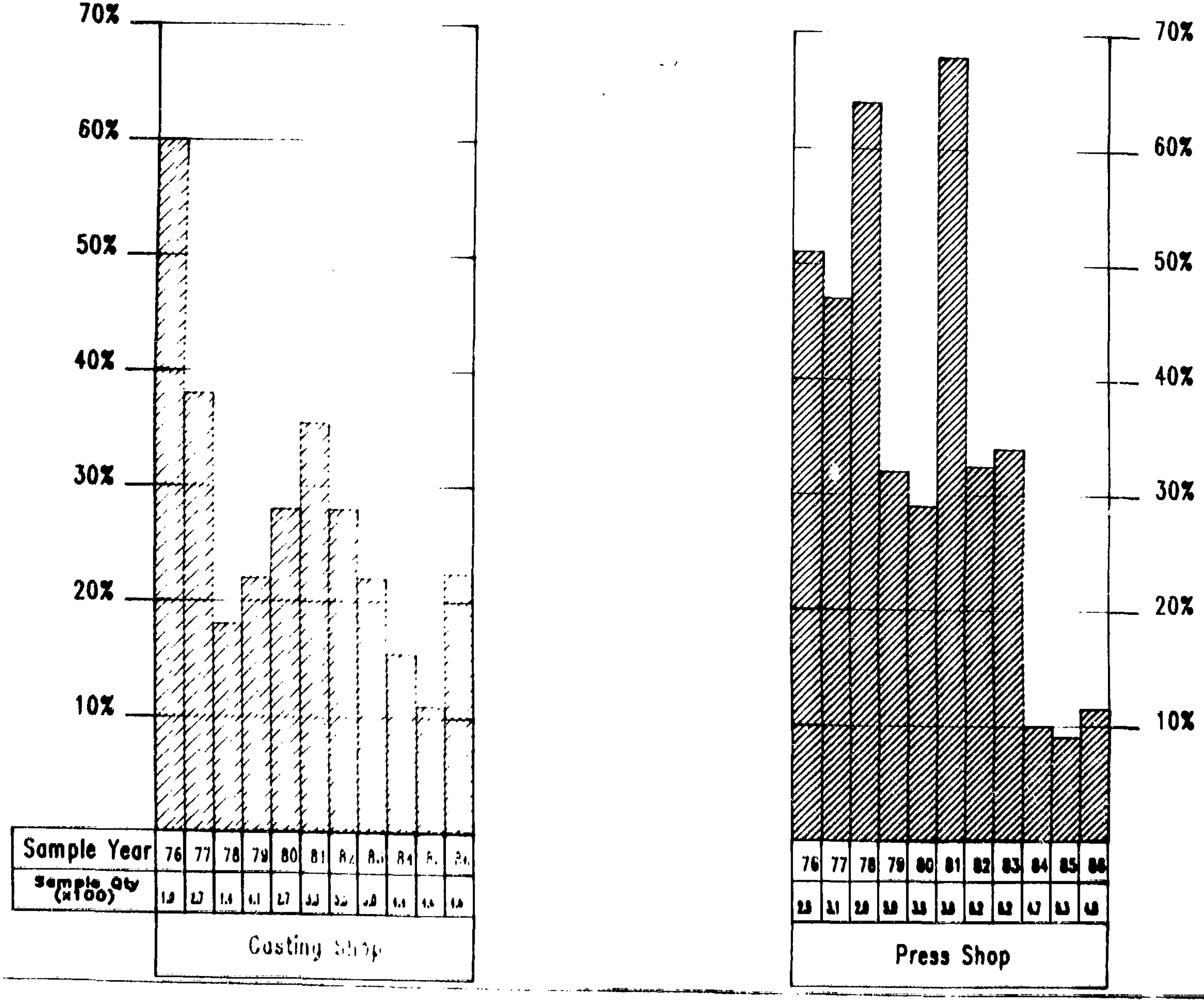
Be SMEAR SUMMARY 1976 to 1986



PRESS SHOP

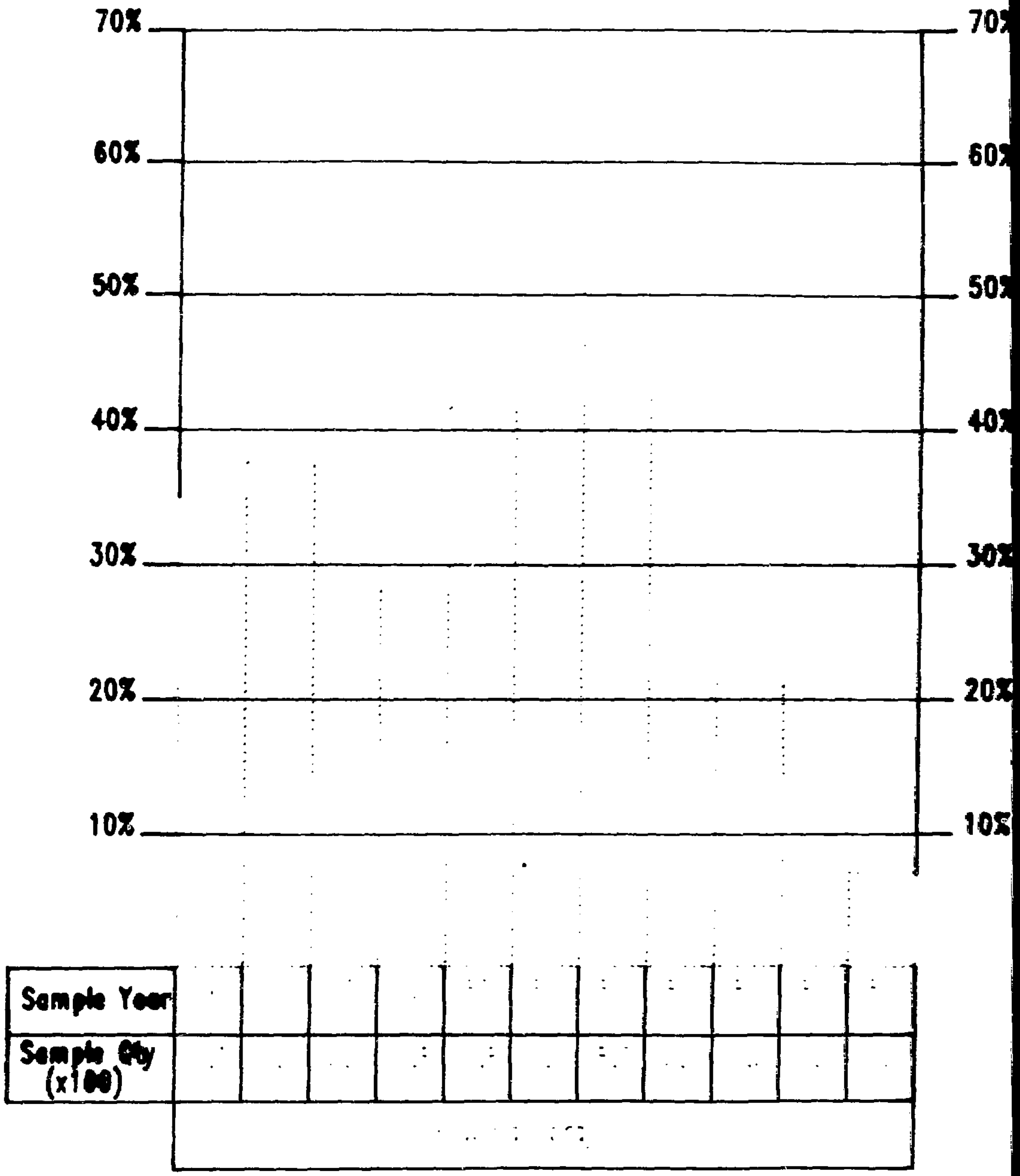
Be SMEAR SUMMARY 1976 to 1986

(Readings greater than 25 $\mu g/ft^2$)

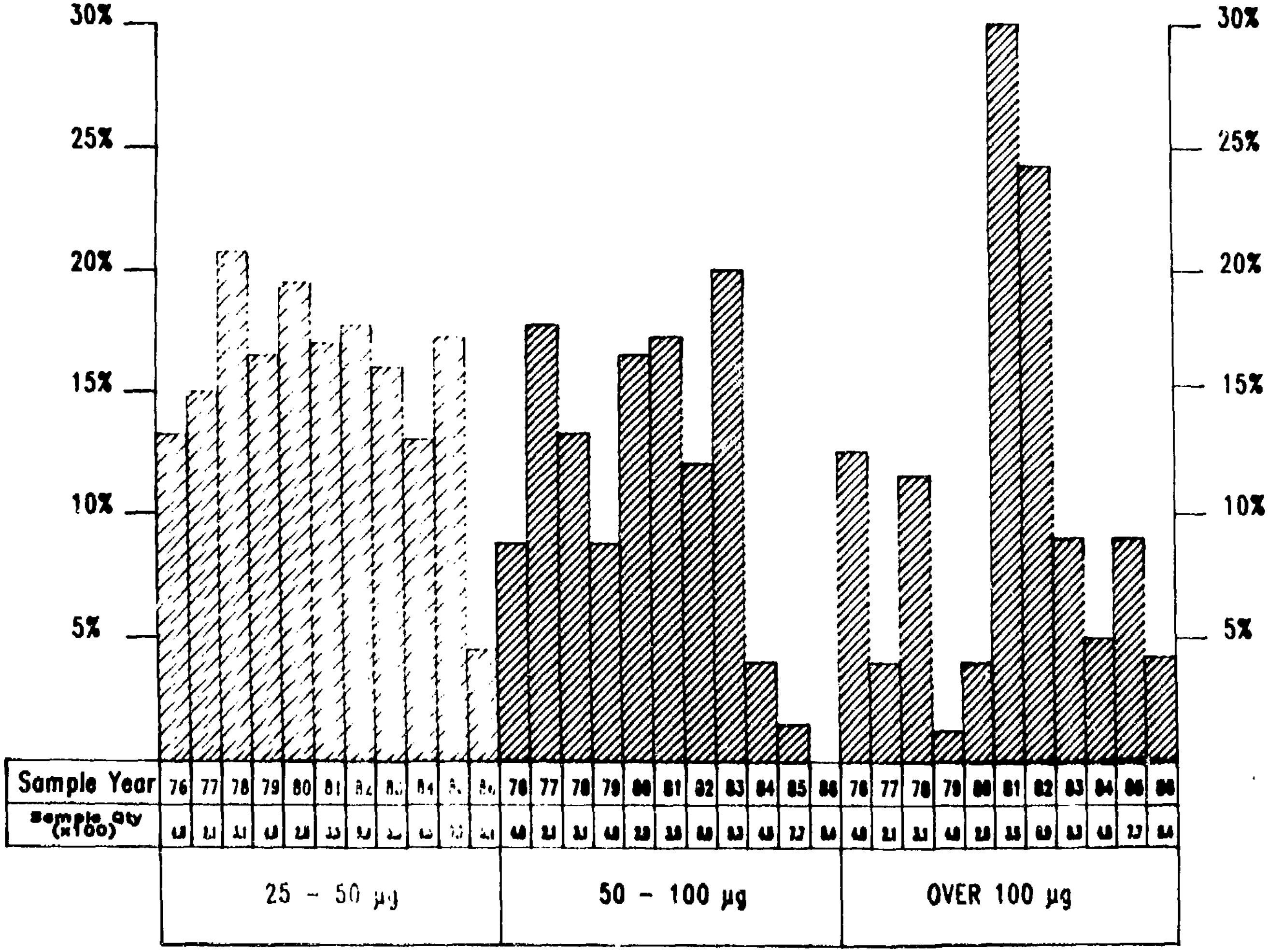


Be Smear Results 1976 to 1986

(Readings greater than 25 μg/ft²)

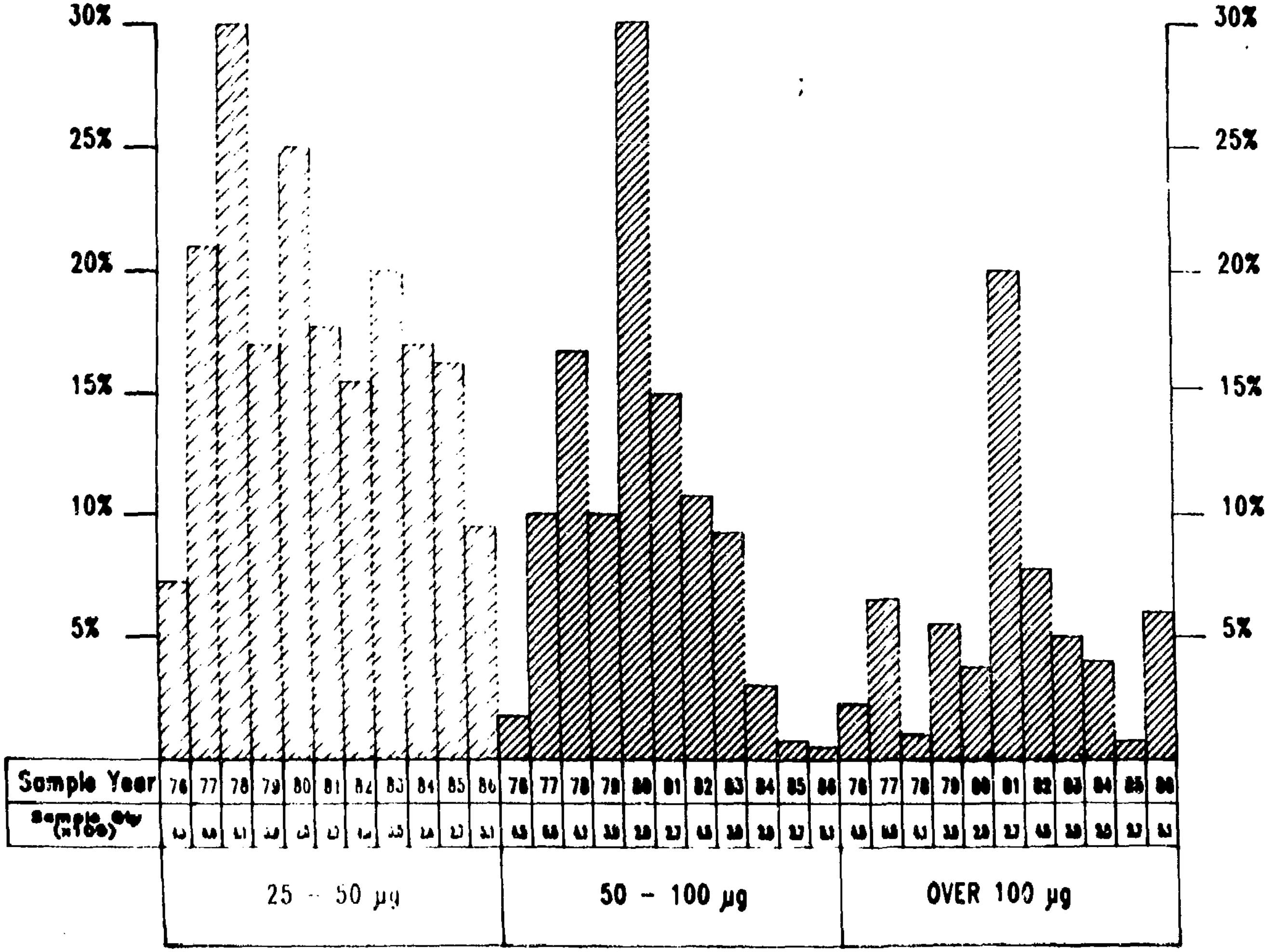


Be SMEAR SUMMARY 1976 to 1986



PLASMA SPRAY

Be SMEAR SUMMARY 1976 to 1986



ROTEX (IMPACT ROOM)

EXPERIENCE WITH ULTRA-LOW LEVEL Be ANALYSIS

Marcel W. Nathans

Lawrence Livermore National Laboratory

Emphasis will be placed on the analysis of large filters used for sampling of environmental Be around Livermore and Site-300. After digestion and filtration, the samples are brought to 100 mL and analyzed in an AAS with electrothermal atomization in precoated graphite tubes. Concentrations are mostly in the 0.5 - 10 ng/mL range. Spike recoveries (2.0 ppb) are erratic, but recoveries of spikes of Be-7 tracer are about 100%. Standard additions usually yield more satisfactory results. It was also found that the residues contained quantities of Be of the same order of magnitude as the filtrate. Results by standard additions again were quite different. An attempt was made to correlate this phenomenon with the presence of other environmental elements present in the solutions.

Slide 1

Sample types / Analysis procedures

sample type	digestion	measurement	rep't limit
swipes	$HNO_3 - HClO_4$	flame - AA	0.15 μg
area samples	$HNO_3 - HClO_4$	flame - AA	0.15 μg
personal samples	HNO_3	HGA	.3 ng
well waters, run-off	HNO_3	HGA	0.3 ng/mL
env. filters	$HNO_3 - HClO_4$	HGA	{ 20 - 50 ng
— dig. res.	$HF - HNO_3$	HGA	$(\sim 2\times10^{-6}\ \mu g/m^3$

Slide 2

Experimental Conditions

1. **AAS**

Perkin-Elmer-5000
λ = 239.4 nm, background corrected
slit L0.7
read: peak absorbance, 5 sec

2 **HGA**

HGA-500 with AS-40
aliquot: 10 or 20 μL
Program:

Step:	1	2	3
temp. (°C)	120	1200	2700
ramp (sec)	10	10	1
hold (sec)	15	15	6
read			0
flow (mL/min)	300	300	50

tube: pyrolytically coated

Slide 3

Response of Standards

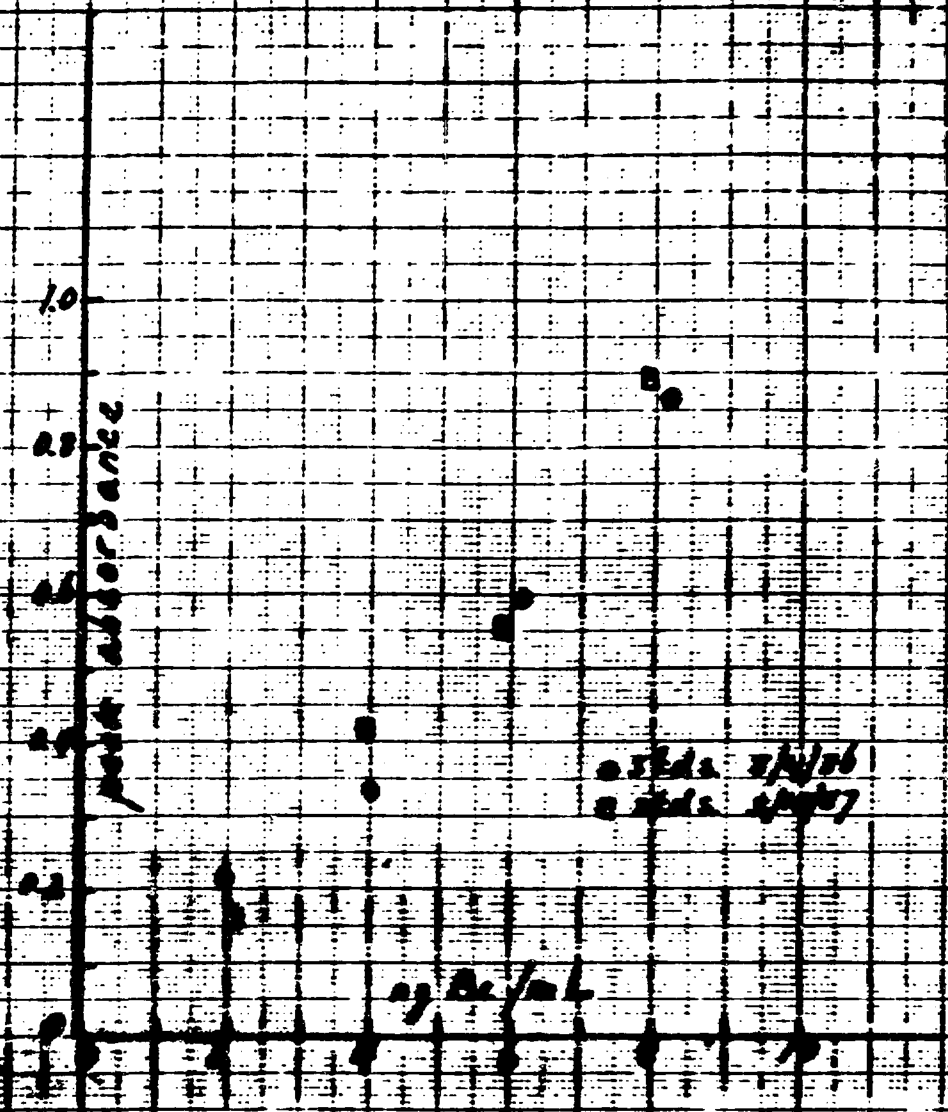
old tube	peak absorbance			
nominal conc.	2 ppb	4 ppb	6 ppb	8 ppb
stds. 8/4/86	0.120	0.258	0.434	0.509
stds. 3/18/87	0.025	0.068	0.105	0.085
new tube				
stds 8/4/86	0.212	0.333	0.595	0.862
stds 3/18/87	0.164	0.419	0.554	0.894

Slide 4

Standards Check by ICP

concentration in ppb

nominal	2	4	6	8
stds 8/4/86	1.96	4.01	6.18	8.19
stds 3/18/87	2.09	3.89	5.83	7.89
stds 3/25/87		3.89		7.86


1.0
0.8
0.6
0.4
0.2
0
peak absorbance
µg Be/mL
● stds
■ stds

Slide 5

Recoveries

	200 ng	^{7}Be
set 4461-4465	155 %	98 %
set 5558-5573	180 %	106 %

Slide 6

Standard Additions

sample no.	direct	by MOSA	
	ng	ng	slope
4461	445	143	10.6
4463	166	147	21.0
4465	462	276	7.93
4469	225	370	21.5
spike(ng)	310	184	29.6

Slide 7

Procedure Comparison-1

	Total nanograms	
sample no	filtrate	residue
4461	445	—
4463	166	—
4465	462	673
4469	225	147
4472	468	1260
4475	431	1440

Slide 8

Procedure Comparison - 2

Total nanograms

station no	filtrate (HNO_3 - $HClO_4$)	eluates (HF)
01	186	764
02	425	1251
12	275	1138
13	322	990
14	267	880
15	317	932

Slide 9

Response of Standards
Use of Platform

nom. conc'n	Absorbance direct	 platform
2*	0.145	0.104
4	0.200	0.178
6	0.216	0.230
8	0.420	0.401

* 2.4 by ICP

Slide 10

Platform - Standard Additions

	total nanograms		
sample no	direct*	MOSA	MOSA slop
8772538	472	660	11.9
8772540	225	288	11.3
8772542	326	458	12.1
8772544	322	400	11.4

* diluted 1:1

BERYLLIUM AEROSOL CONTAINMENT DURING MACHINING OPERATIONS

S. C. Sadler, RFP

Abstract

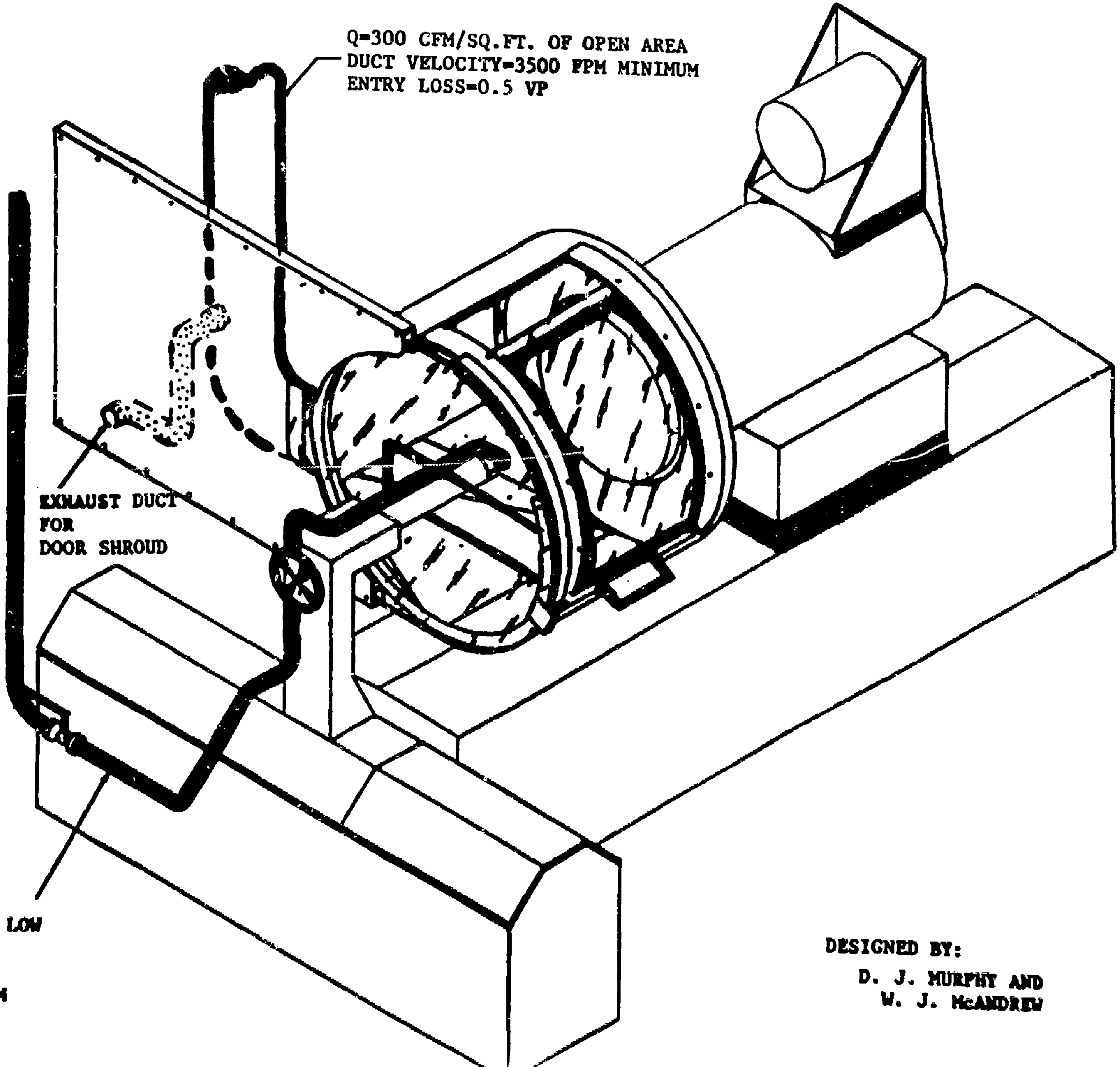
A study was conducted to determine the effectiveness of a High Velocity Low Volume ventilation system in controlling beryllium aerosols. Five hundred fifty personal samples were collected to assess ventilation containment efficiency. The sample data was examined to determine what effect the employee, machine, operation, part type, part size, shift, room and work practice had on beryllium aerosol generation. The geometric mean airborne concentration of beryllium prior to installation of the HVLV system was 0.4 micrograms per cubic meter. Following installation of the HVLV system the geometric mean airborne concentration of beryllium was 0.02 micrograms per cubic meter. Operations and adherence to work practices were found to be most significant factors in controlling beryllium aerosols. Modifications to operational procedures and adherence to safe work practices have further reduced airborne beryllium concentrations.

Rockwell International

DESIGN COMPARISON

ENCLOSURE	"HV/LV"
o Dry Machining	o Dry Machining
o Single Exhaust 300 FPM	o Multiple–Exhaust 300 FPM 10,000 FPM
o Single Access Point Tool & Employee	o Dedicated Access Point Tool Employee
o Exposed Surfaces	o Enclosed Surfaces
o Square Interior	o Round Interior
o Clean–Up–Shared Portable HEPA Vacuum	o Clean–Up–Individual Pick–Up At Each Machine
o Dampers	o No Dampers

HV/LV BERYLLIUM VENTILATION SYSTEM

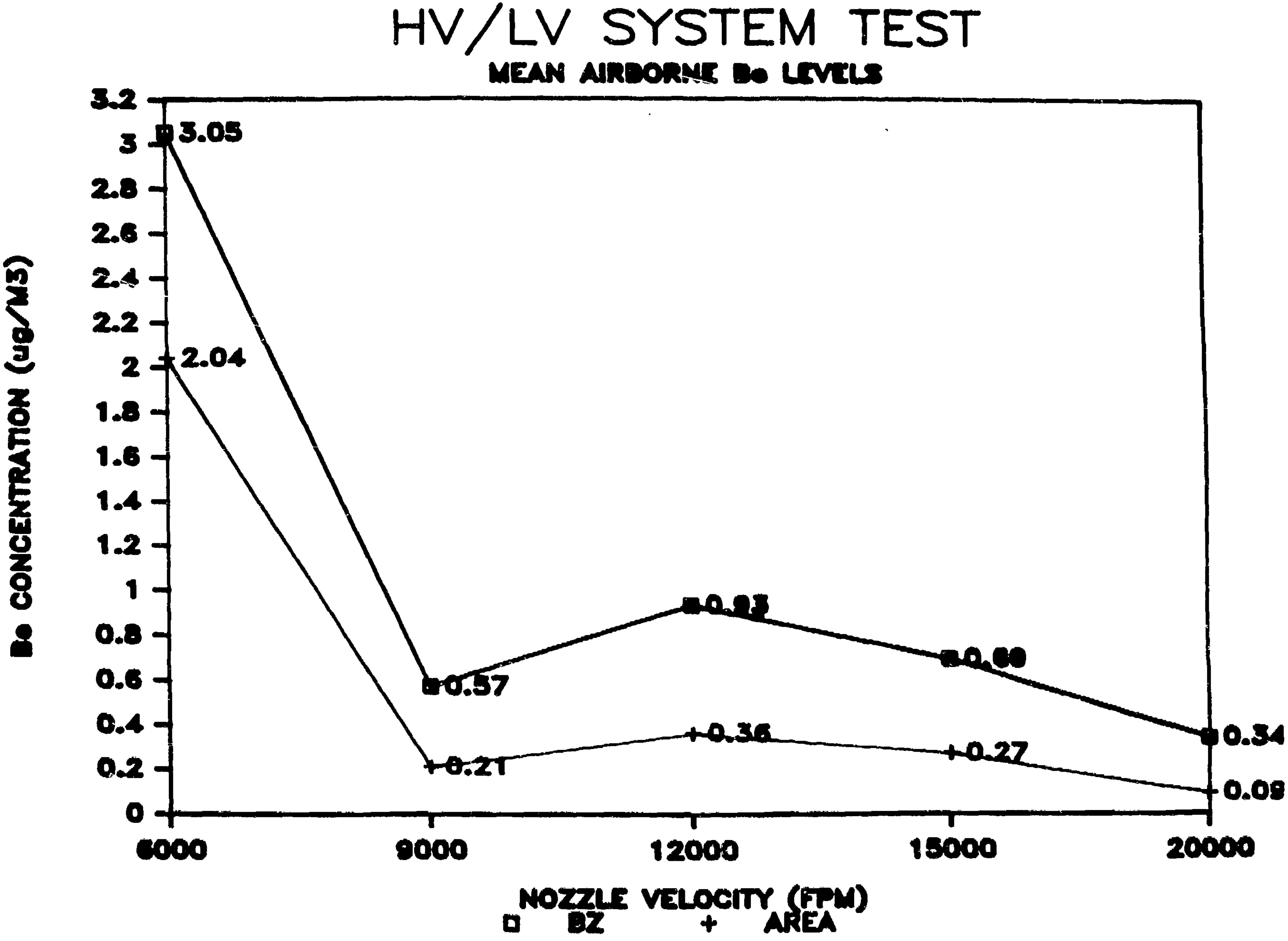


Q=300 CFM/SQ.FT. OF OPEN AREA
DUCT VELOCITY=3500 FPM MINIMUM
ENTRY LOSS=0.5 VP

EXHAUST DUCT
FOR
DOOR SHROUD

HIGH VELOCITY LOW
VOLUM EXHAUST
Q=55 CFM
VEL=10,000 FPM
1" DIA NOZZLE

DESIGNED BY:
D. J. MURPHY AND
W. J. McANDREW


HV/LV SYSTEM TEST
MEAN AIRBORNE Be LEVELS
Be CONCENTRATION (ug/M3)
3.2
3
2.8
2.6
2.4
2.2
2
1.8
1.6
1.4
1.2
1
0.8
0.6
0.4
0.2
0
3.05
2.04
0.57
0.21
0.93
0.36
0.69
0.27
0.34
0.09
6000
9000
12000
15000
20000
NOZZLE VELOCITY (FPM)
□ BZ + AREA

SAMPLING STRATEGY

- Full Shift — 8 Hours
- All Shifts — 3
- All Operations
    - Machinists
    - Tool Crib
    - Maintenance
    - Custodial
    - Supervision
    - Industrial Hygienists
- Hygienists In Shop — 24 Hours Per Day
- 6 Weeks Duration / 500—600 Samples

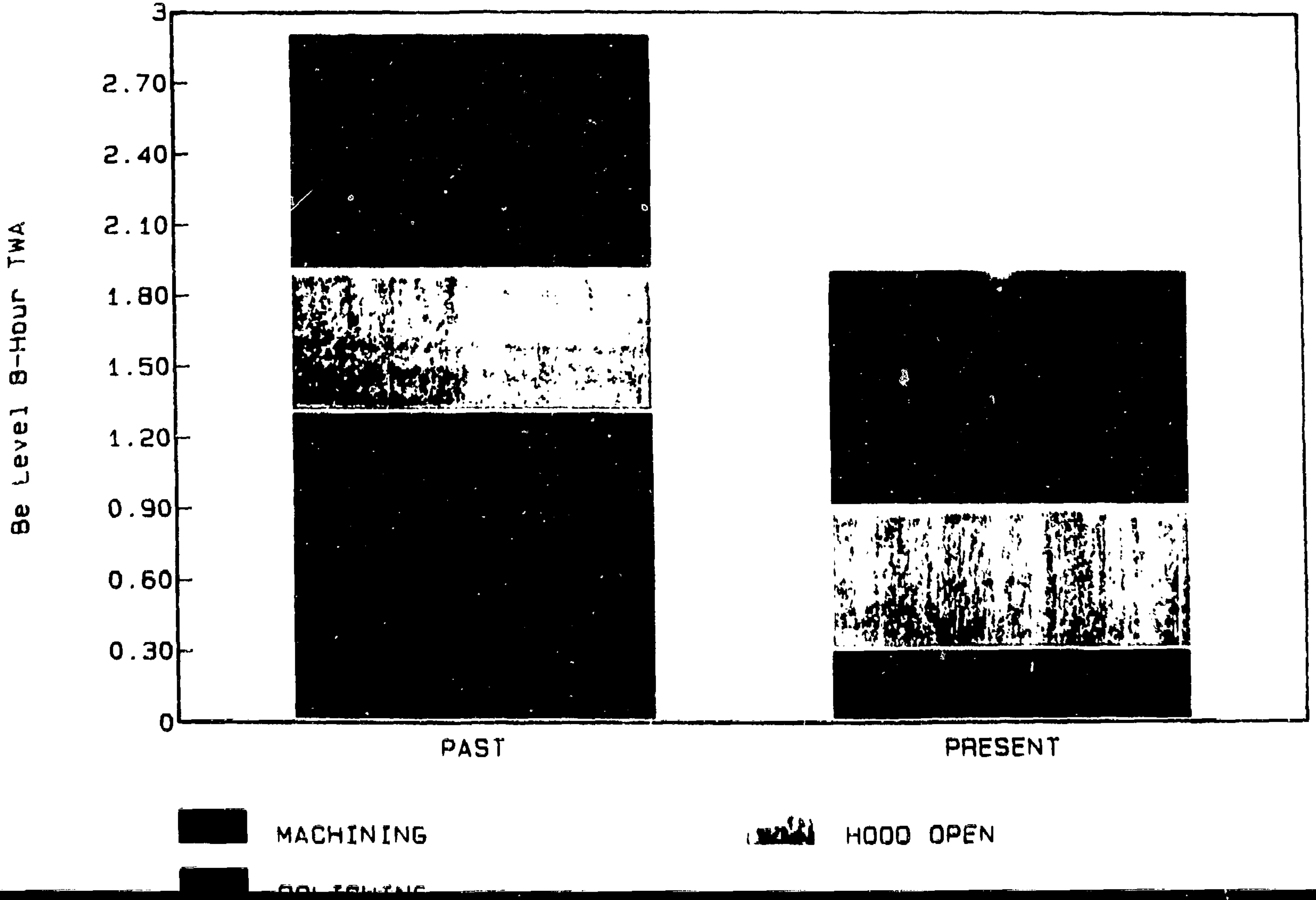
ANALYSIS

- Average Concentration — Before vs After
- Excursions — Types
- Day
- Room
- Shift — Days, Pm's, Mids's
- Right Sholder vs Left
- Person
- Machine
- Machine Type
- Part Type
- Operation
- Size
- Combination of Above

RESULTS

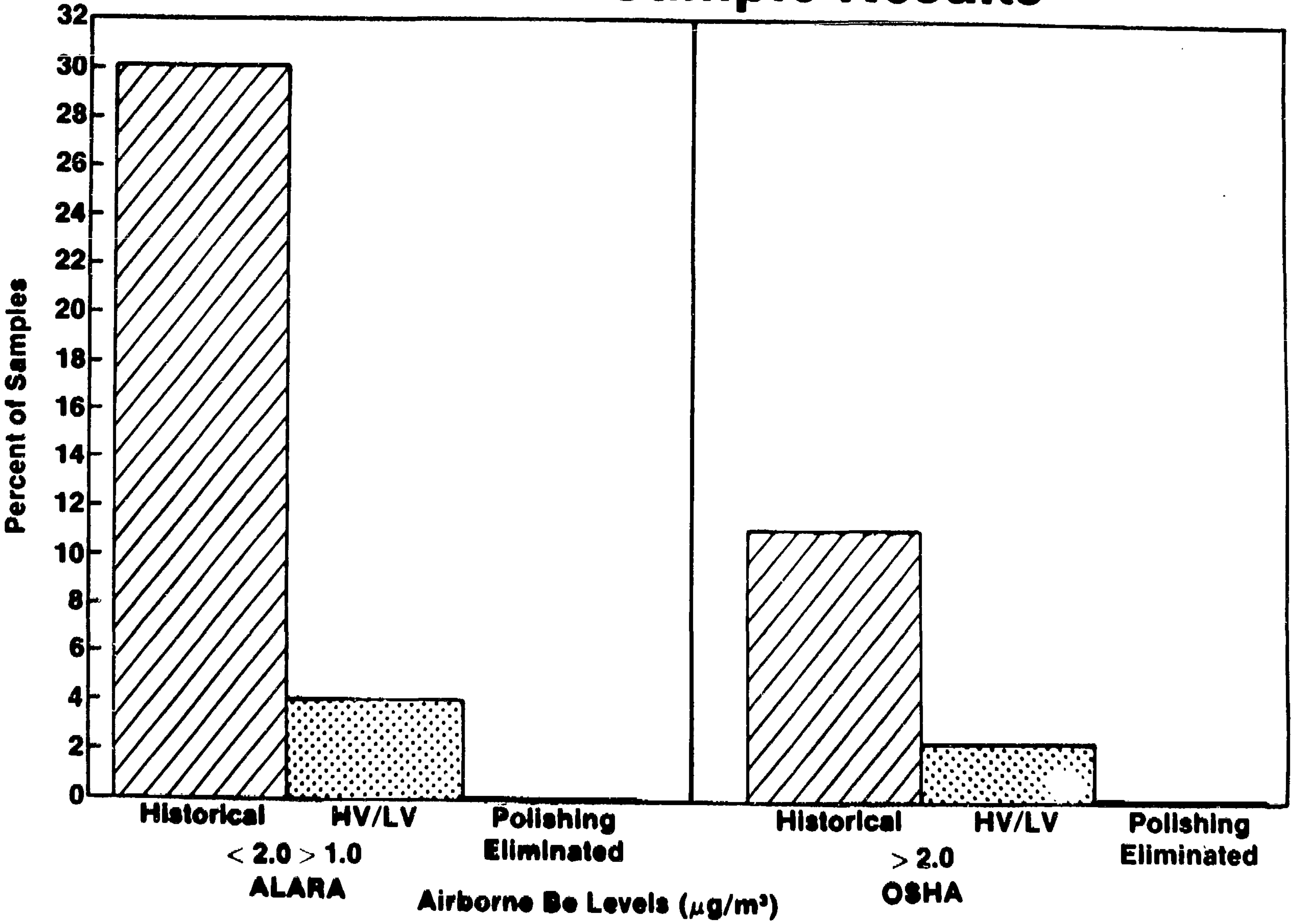
- Significant Reductions Achieved
- Excursions above 2.0 ug/M3 Still Occurred
- Polishing Identified As Major Source

EFFECT OF POLISHING



Rockwell International

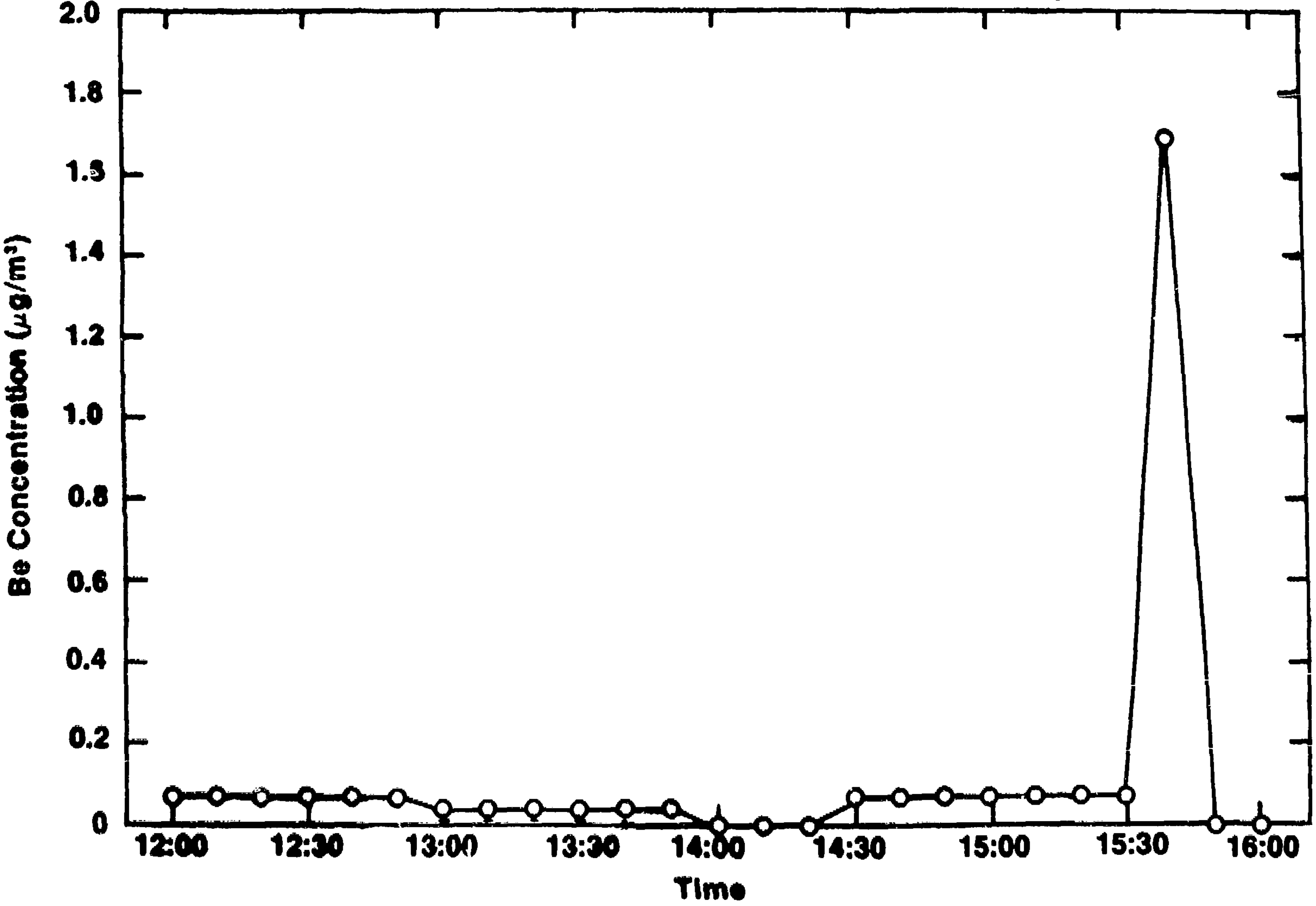
Personal Air Sample Results



Rockwell International

Diagnostic Sampling

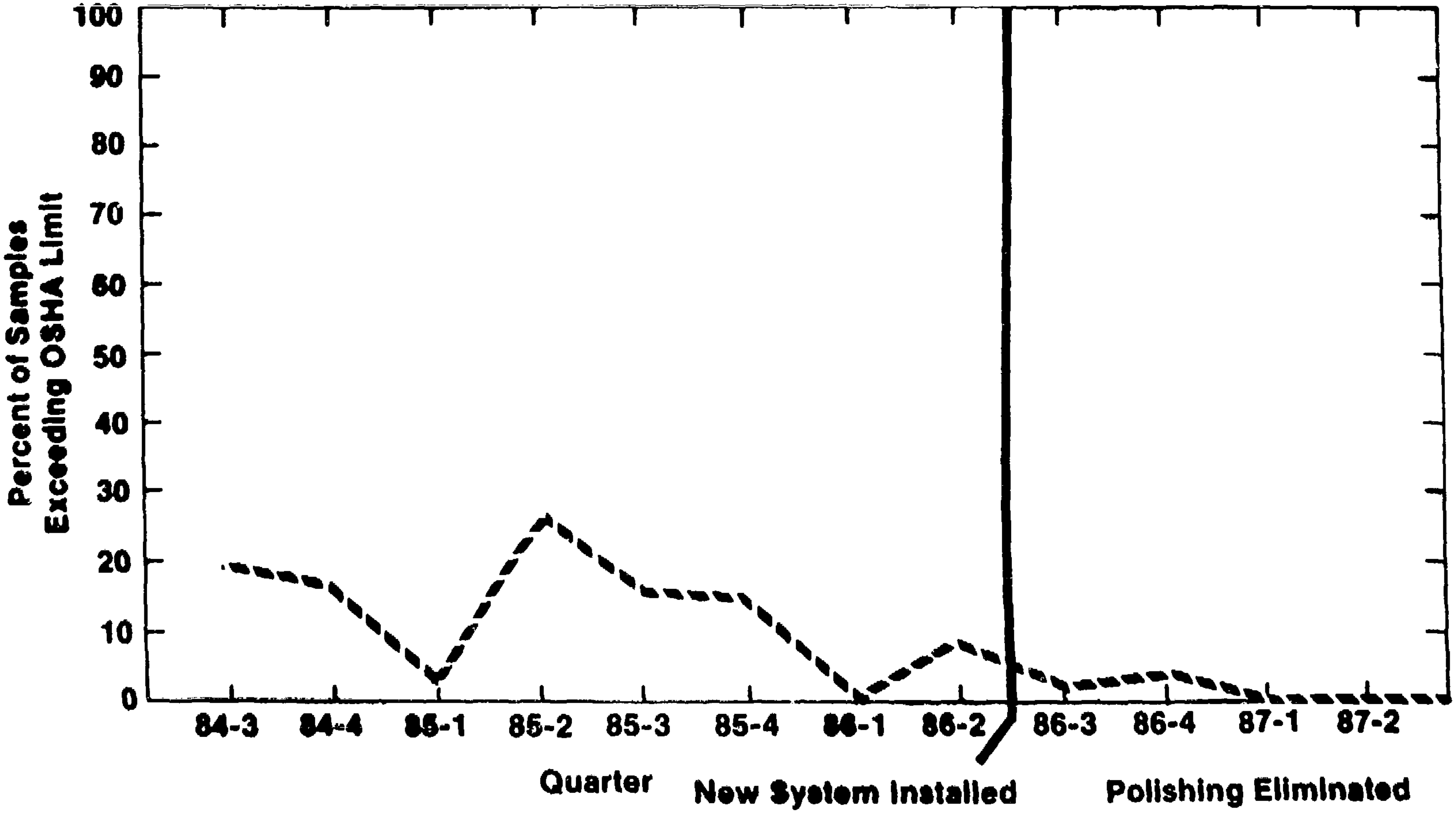
Round Hood; Machining vs. Polishing



Rockwell International

Historical Air Sample Analysis
444 Be Shop

1984 → Present



Analysis of Oxygen in Beryllium and Beryllium Foils by Ion Microanalysis

C. W. Price, J. C. Norberg, R. G. Musket, F. J. Fulton
Chemistry and Materials Science Department
Lawrence Livermore National Laboratories
P. O. Box 5508, Livermore, CA 94550

Ion micoranalysis using a primary $^{133}Cs^+$ ion beam and secondary-ion mass spectroscopy (SIMS) is an effective technique for the analysis of oxygen in beryllium. The sensitivity of SIMS permits analyses of oxygen contents near the solid solubility limit of oxygen in beryllium, which is estimated to be of the order of 30 wppm or 17 appm. The excellent spatial resolution of the ion microanalyzer also permits characterization of the oxygen distribution among the various phases and particles that may exist in beryllium. However, exact quantitative analyses of oxygen in beryllium are complicated by the presence of oxygen in discreet BeO particles and by residual contamination in the specimen chamber of the ion microanalyzer. In an effort to obtain more quantitative data from the SIMS analysis, ion-implanted specimens were prepared with fluences from 1.28×10^{14} to 3.84×10^{15} O atoms/cm^2 and calculated peak concentrations of 119 to 3695 appm. These specimens were then used to establish sensitivity factors required to perform quantitative analysis using the SIMS data. This analysis is effective for determining the oxygen content in the bulk beryllium matrix. When inclusions and other particles are present in beryllium, ion imaging can be used to determine the spatial distribution of particles containing oxygen, and optical metallographic and stereologic techniques can then be used to determine the oxygen content in the particles based on the assumption that they are BeO. Use of the Cs^+ ion beam also permits the generation of depth profiles to determine the distribution of oxygen normal to the surface, and depth profiles have been particularly effective to characterize the oxygen distribution through thin beryllium foils. In addition, ion microanalysis permits the detection of low levels of other trace impurities in beryllium such as carbon and chlorine, and the spatial distribution of these elements also can be determined by ion imaging.

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ANALYSIS OF OXYGEN IN BERYLLIUM AND BERYLLIUM FOILS BY ION MICROANALYSIS

INTRODUCTION

EXPERIMENTAL PROCEDURE

RESULTS

EFFECT OF OXIDE PARTICLES

SUMMARY

MAJOR POINTS

ION MICROANALYSIS USING SIMS IS A POWERFUL AND VERSATILE TECHNIQUE TO ANALYZE BERYLLIUM FOR LOW LEVELS OF CONSTITUENT AND IMPURITY ELEMENTS AND TO DETERMINE SPATIAL DISTRIBUTIONS OF THE ELEMENTS ON A MICROSOCPIC SCALE.

QUANTITATIVE ANALYSES CAN BE PERFORMED USING ION-IMPLANTED BERYLLIUM SPECIMENS FOR STANDARDS.

COMPLETE QUANTITATIVE ANALYSES REQUIRE ACCURATE DETERMINATIONS OF THE OXIDE CONTENT RESIDING IN OXIDE PARTICLES.

SIMS DEPTH PROFILES PROVIDE EXCELLENT CHARATERIZATION OF OXYGEN AND CARBON DISTRIBUTIONS IN BERYLLIUM FOILS.

INTRODUCTION

- Ion microanalysis is effective for the analysis of oxygen in beryllium.
  - Depth profiling
  - Ion imaging
  - SIMS permits analyses of oxygen near the solid solubility limit

- Design and operation of an ion microanalyzer
  - Analyzes secondary ions produced by a primary ion beam
  - Microprobe mode using a mass spectrometer (SIMS)
    - sensitivity can be in the lower ppm or upper ppb
    - depth resolution of the order of 5 nm
  - Imaging mode
    - spatial resolution of about 1 μm
    - microchannel plate and fluorescent screen system -- 2-D image
    - resistive anode encoder (RAE) system -- digital image

- Quantitative analyses can be performed
  - Requires adequate standards to determine conversion factors
  - Beryllium ion implanted with 150 keV O^+ was used
  - Operational limitations must be properly recognized
  - Analysis of oxygen content in oxide particles

- Overlaps the lower analytic ranges of other techniques
  - Rutherford backscattering (RBS)
  - Auger electron spectroscopy (AES)
  - Electron microprobe analysis (EMX)

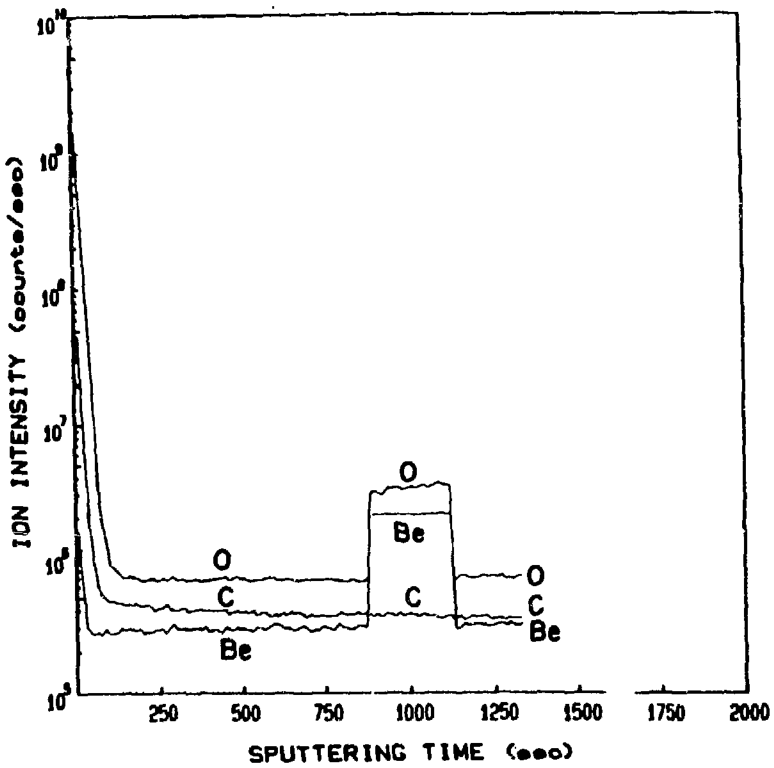
EXPERIMENTAL PROCEDURE

+ SIMS PERFORMED WITH A CAMECA IMS-3F ION MICROANALYZER USING A $^{133}Cs^{+}$ PRIMARY ION BEAM.
    - HIGH ION YIELDS FOR ELEMENTS WITH HIGH ELECTRON AFFINITIES
    - MAJOR CONCERNS FOR THE ANALYSIS OF LOW CONCENTRATIONS
        - DETECTION LIMITS IN THE HOST MATERIAL
        - PRESENCE OF EXTRANEOUS OR RESIDUAL CONTAMINATION
        - ACCELERATION POTENTIAL OF 10 KV TO GENERATE THE Cs^{+} BEAM
        - SPECIMEN WAS MAINTAINED AT -4.5 KV
        - PRIMARY-BEAM CURRENT DENSITY OF ABOUT 16 MA/CM^{2}

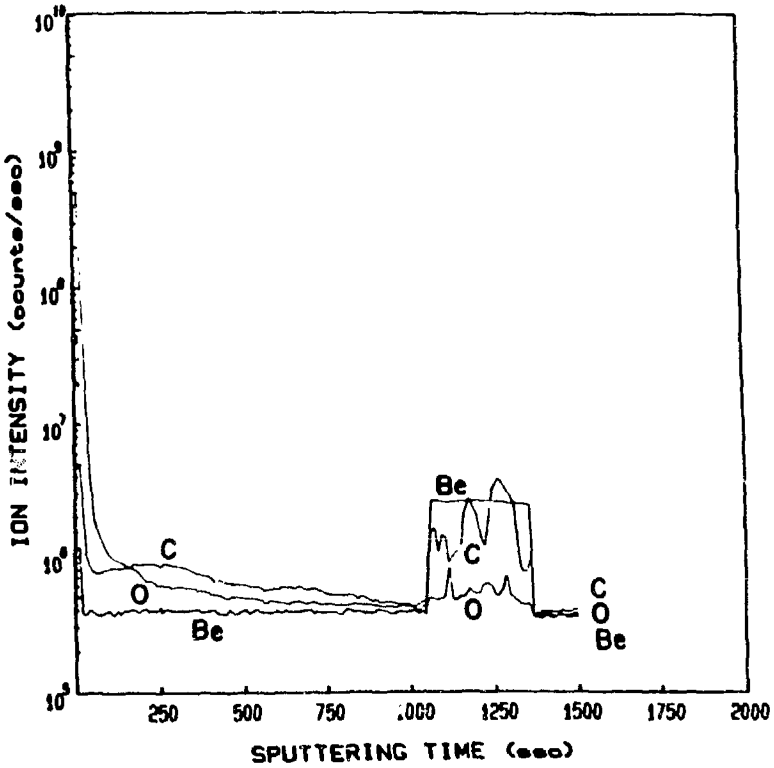
+ SOURCES OF RESIDUAL OXYGEN AND CARBON CONTAMINATION
    - SPECIMEN-BORNE CONTAMINATION
    - SPECIMEN-CHAMBER PRESSURE
    - MEMORY-TYPE EFFECTS -- REDEPOSITION FROM INTERNAL SURFACES

+ HIGH SPUTTERING RATES REQUIRED TO OVERCOME RESIDUAL CONTAMINATION
    - RASTER-SIZE TEST TO DETERMINE MINIMUM SPUTTERING RATES
    - SPUTTERING RATE OF 10 A/SEC. FOR LOW CONCENTRATIONS
    - SPUTTERING RATE OF 1 A/SEC. FOR SURFACE PROFILES

VACUUM DISTILLED MATERIAL

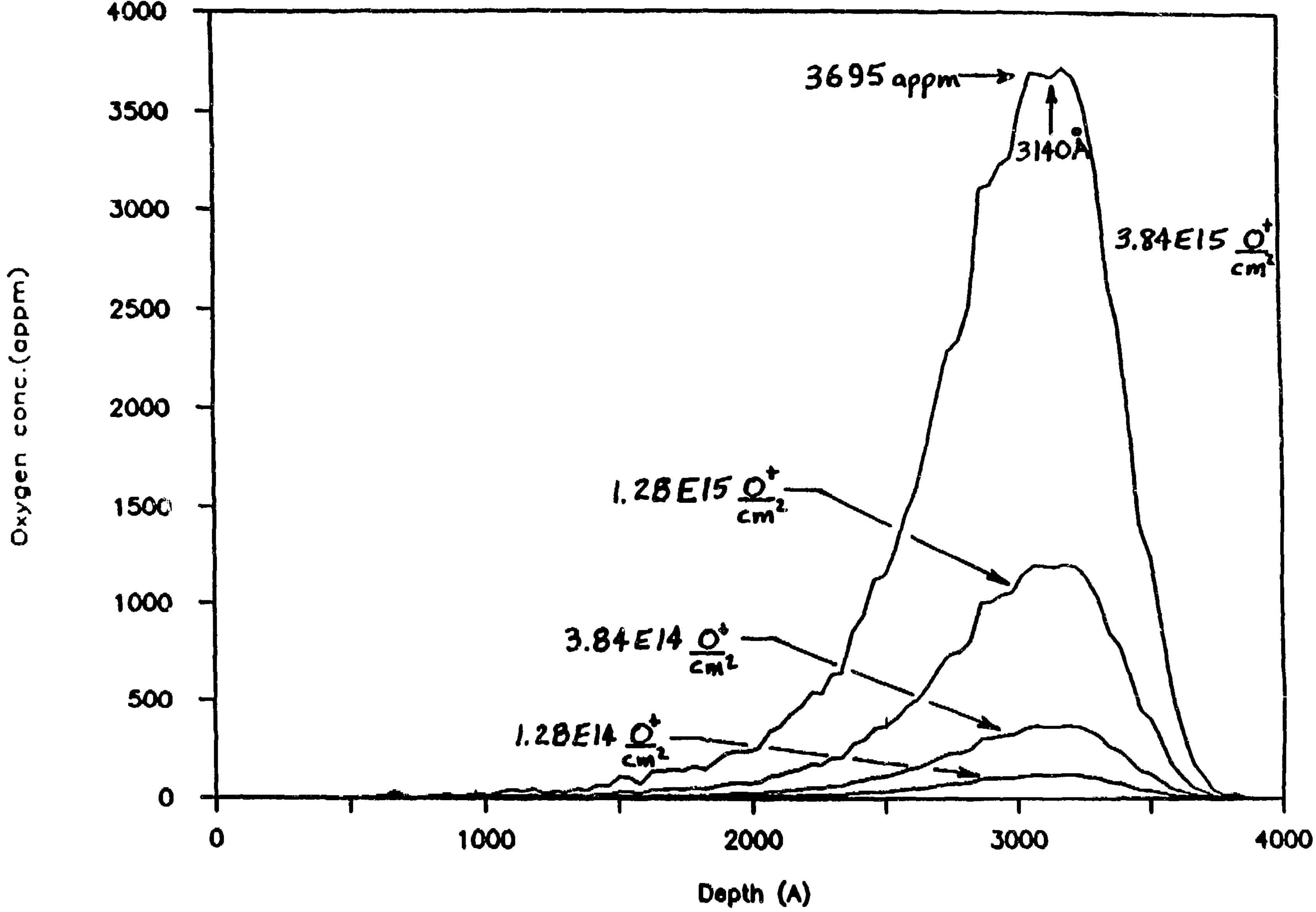


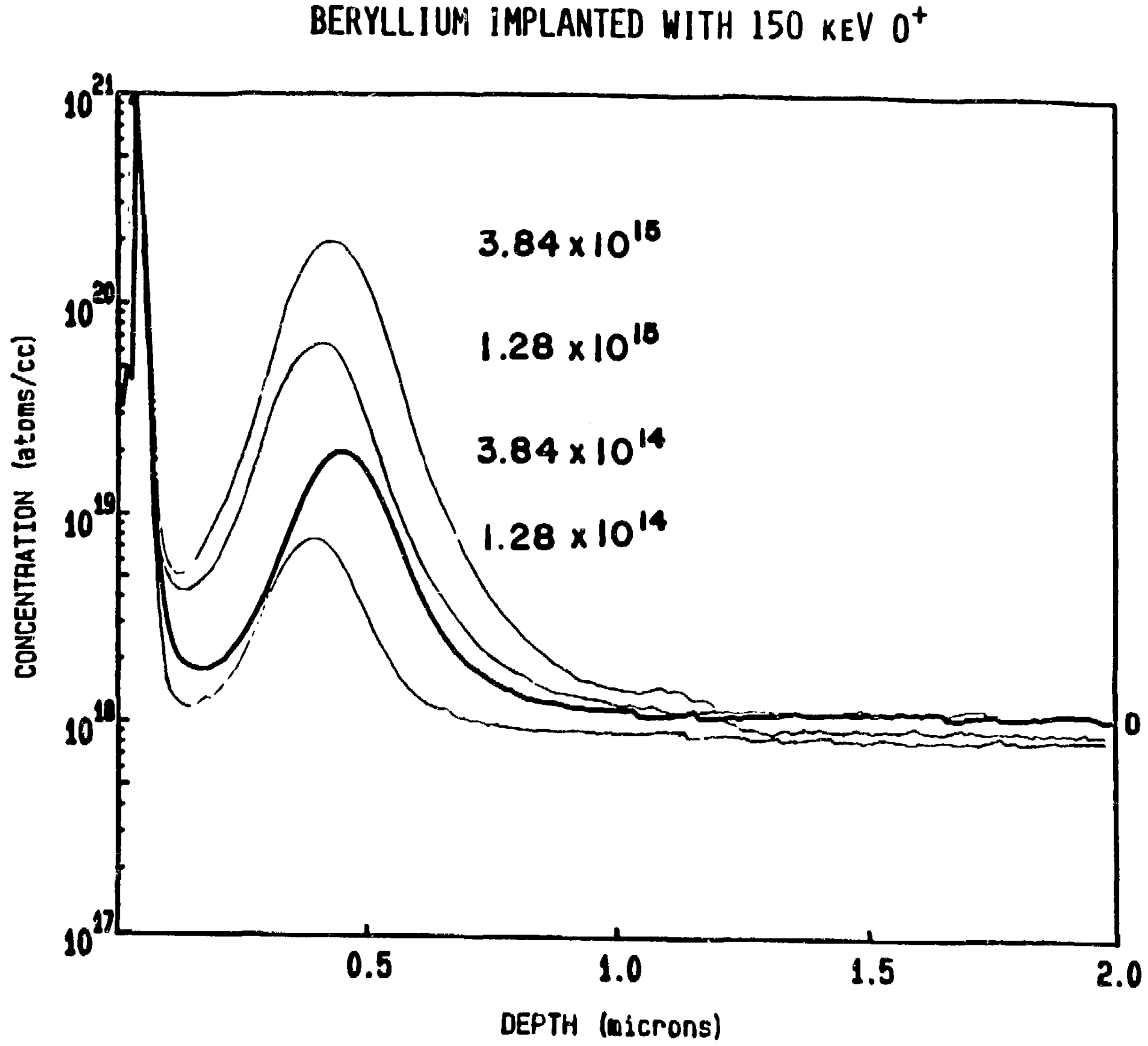
ELECTROFUSION MATERIAL



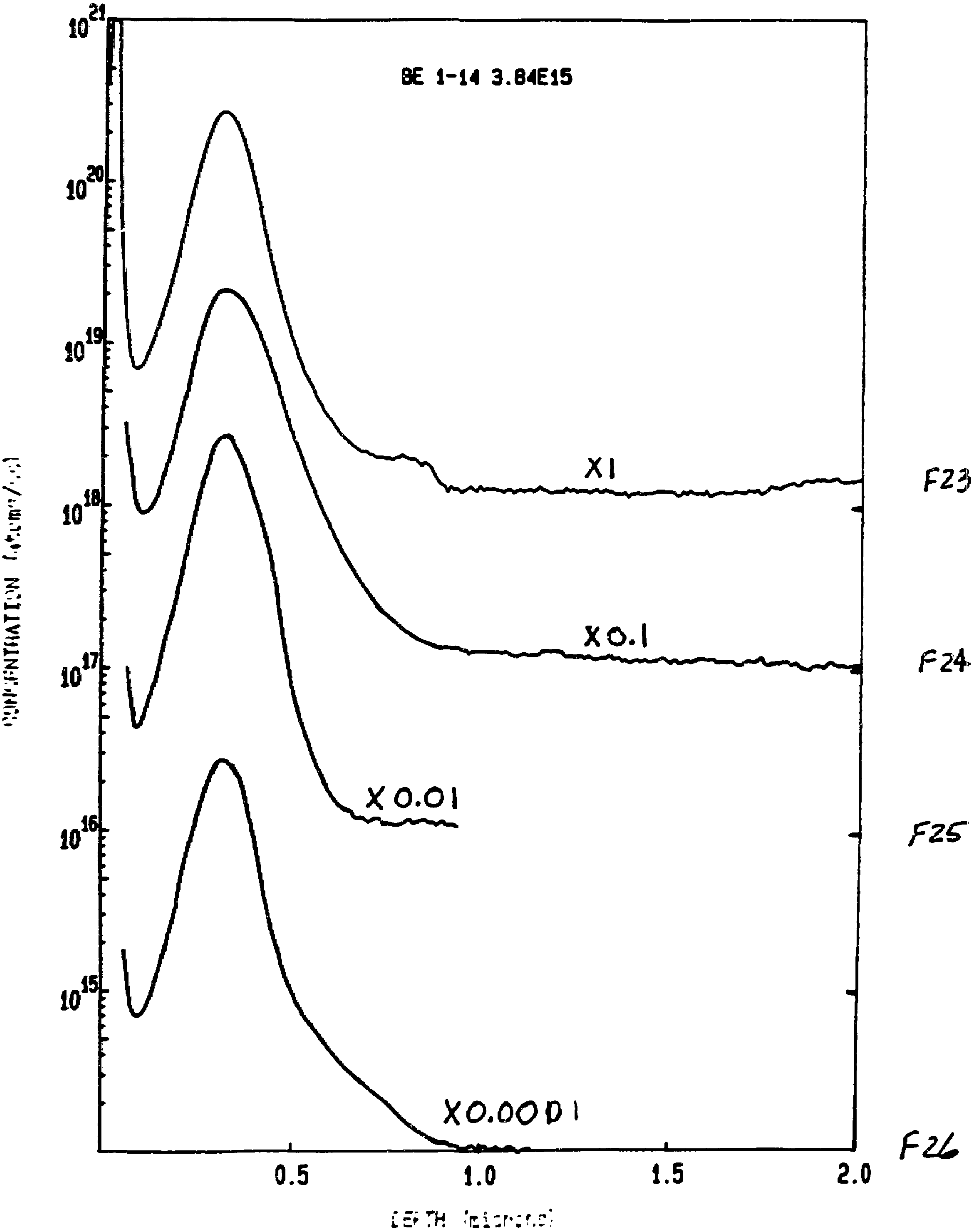
TRIM-86 CALCULATED PROFILE FOR O+ IN BE

(150 keV O+, 20000 h, 40 A intervals)

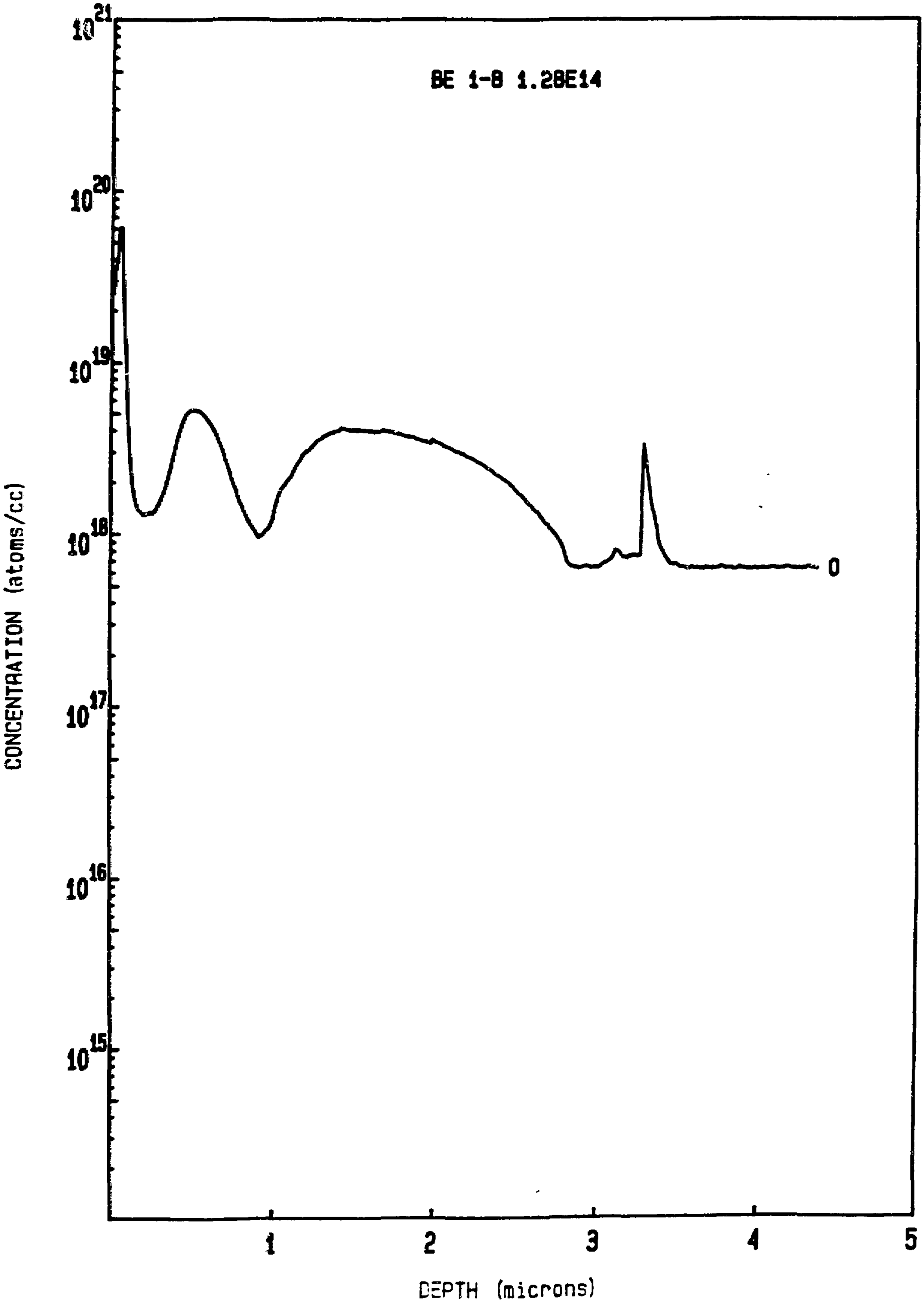


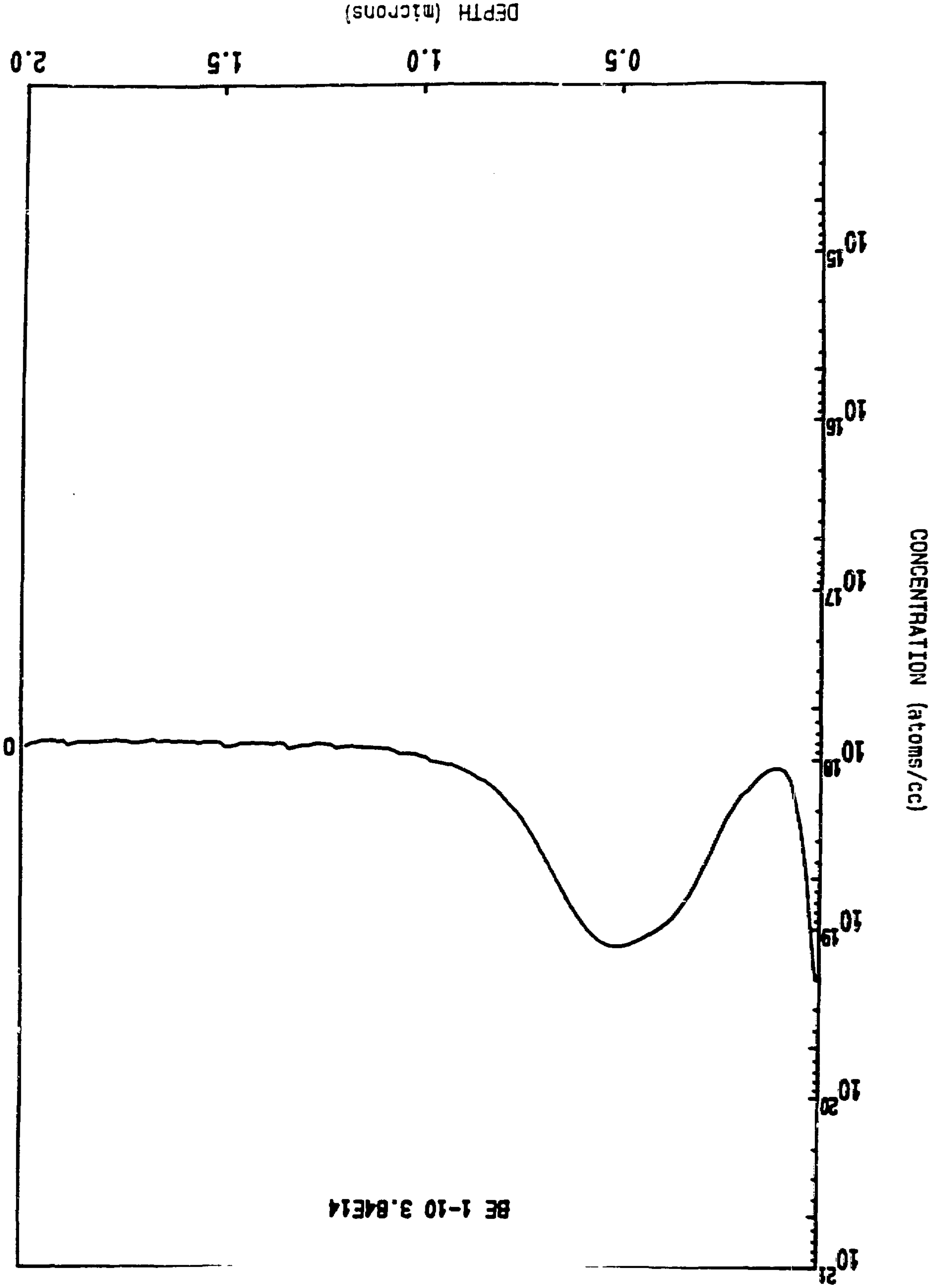

BERYLLIUM IMPLANTED WITH 150 keV O+
10^{21}
10^{20}
10^{19}
10^{18}
10^{17}
CONCENTRATION (atoms/cc)
0.5
1.0
1.5
2.0
DEPTH (microns)
3.84 x 10^15
1.28 x 10^15
3.84 x 10^14
1.28 x 10^14
0

BERYLLIUM IMPLANTED WITH 150 keV O^+



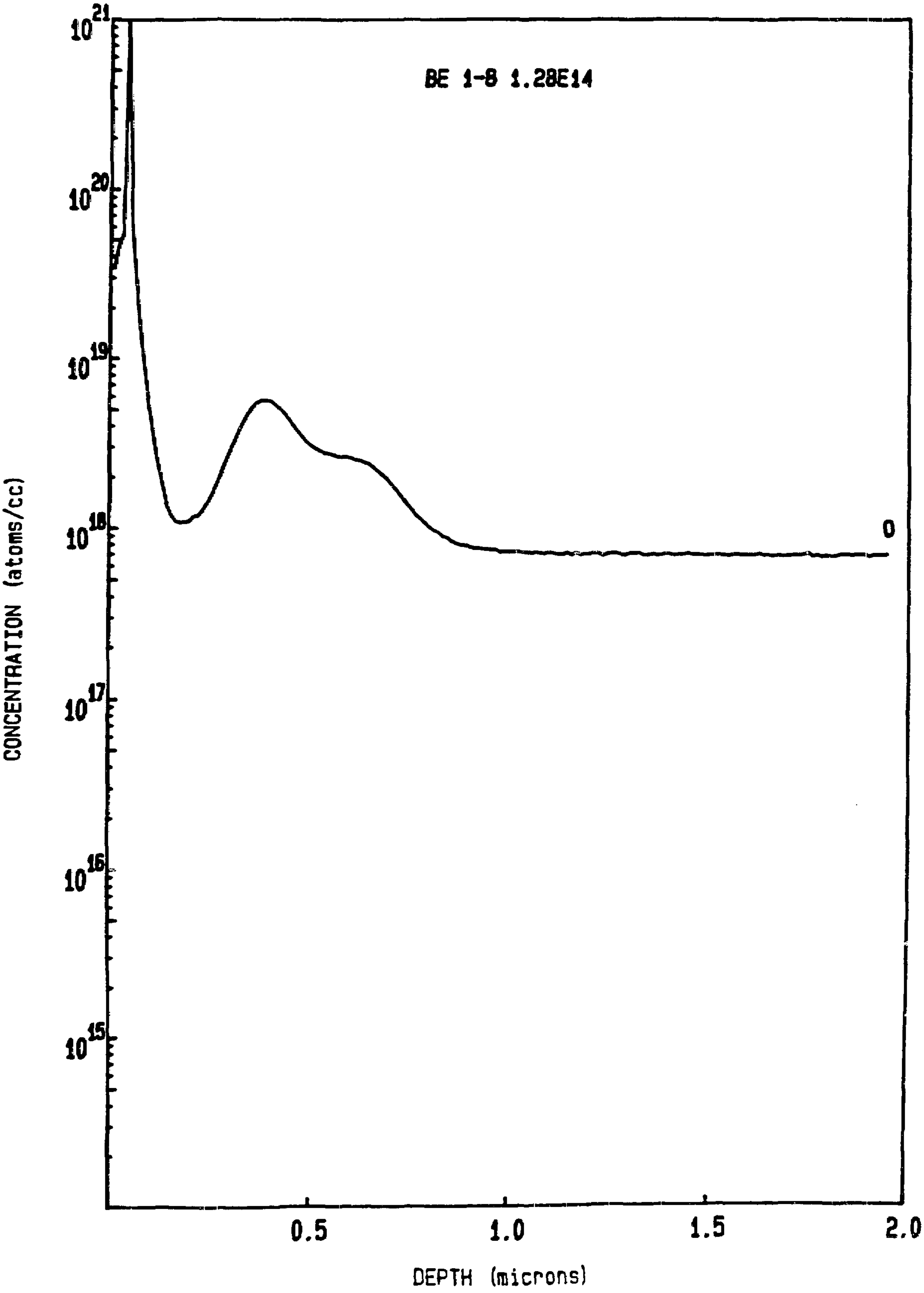
BERYLLIUM IMPLANTED WITH 150 keV O^+



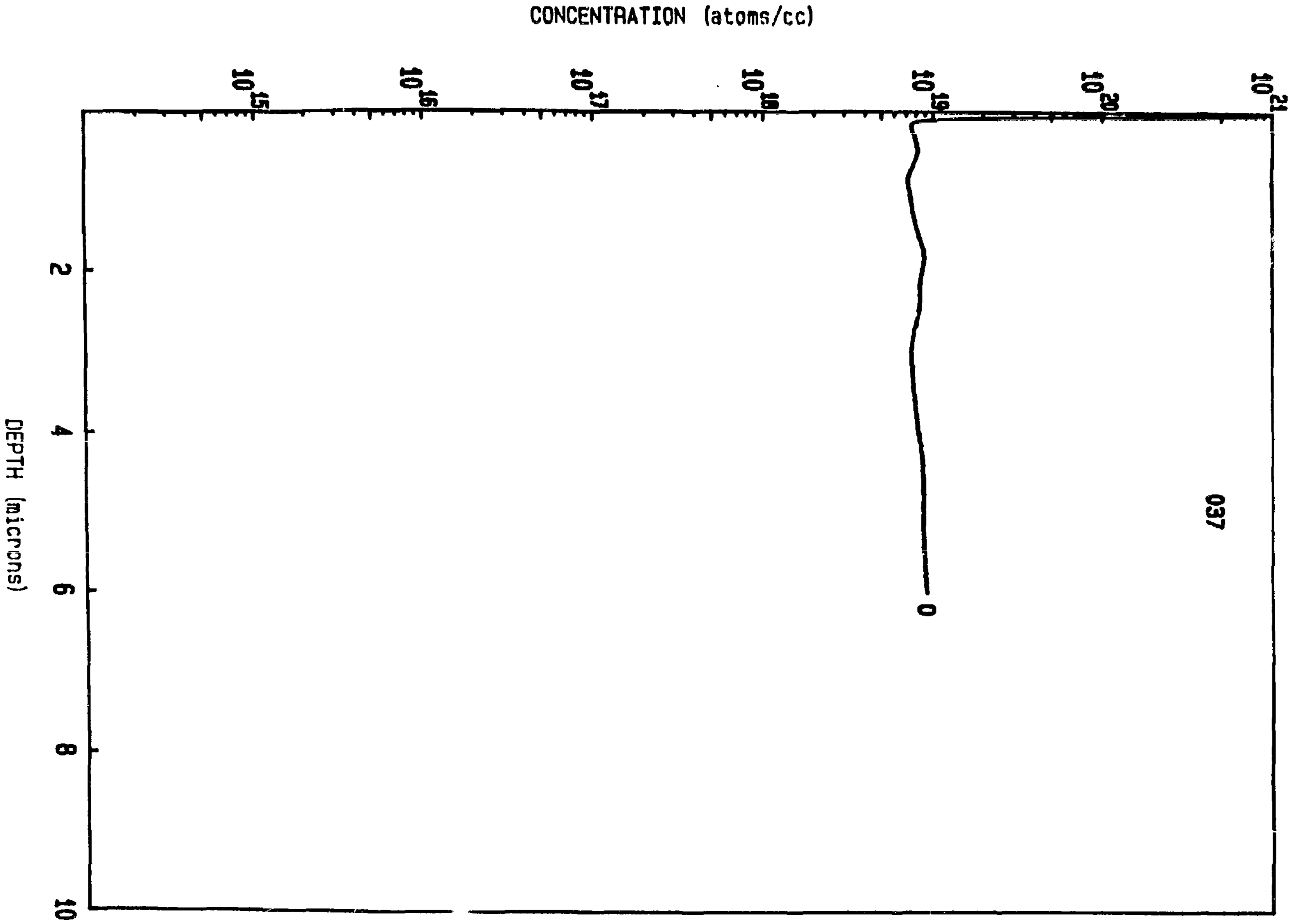


BERYLLIUM IMPLANTED WITH 150 keV O^+

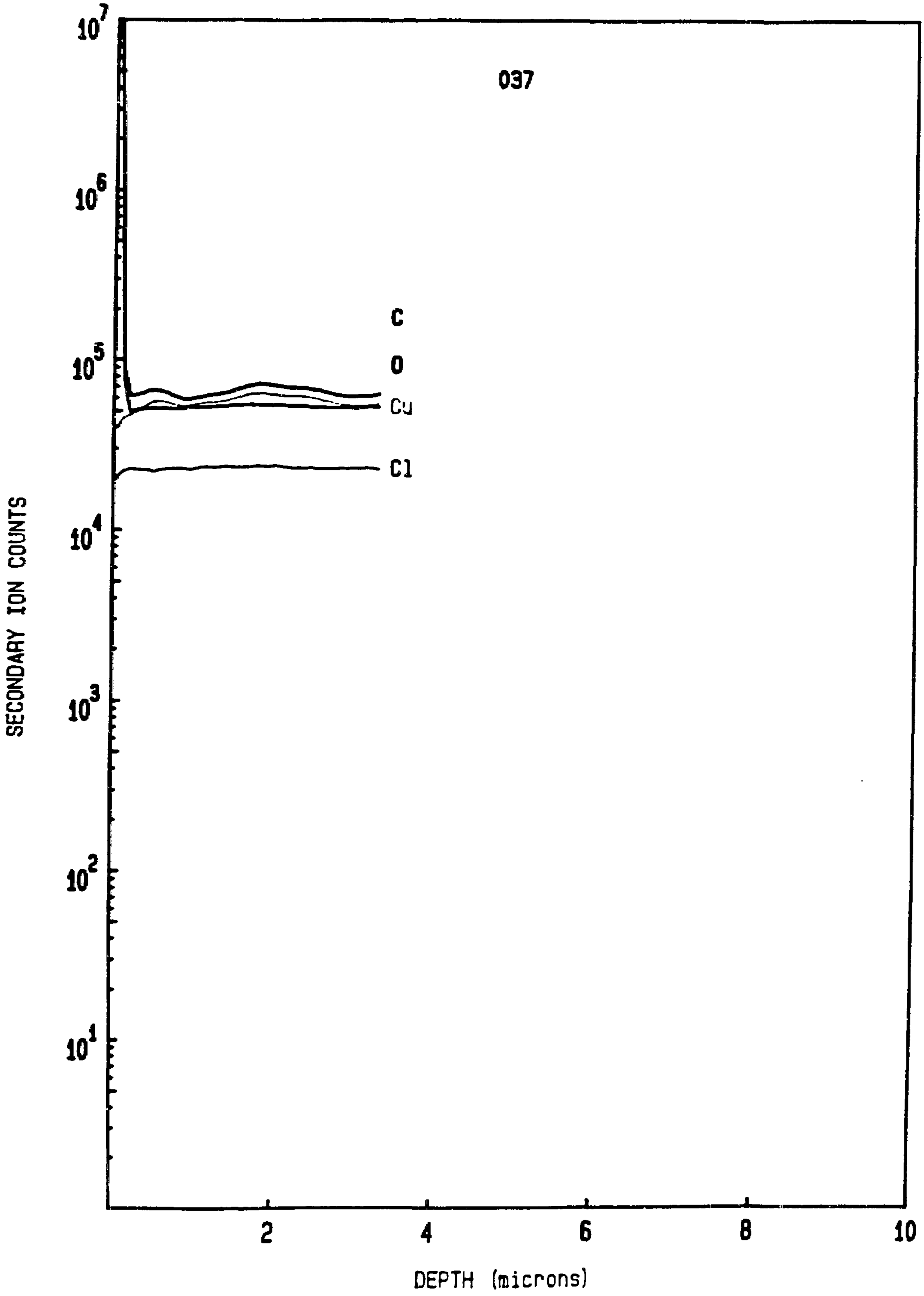
BERYLLIUM IMPLANTED WITH 150 keV O^+

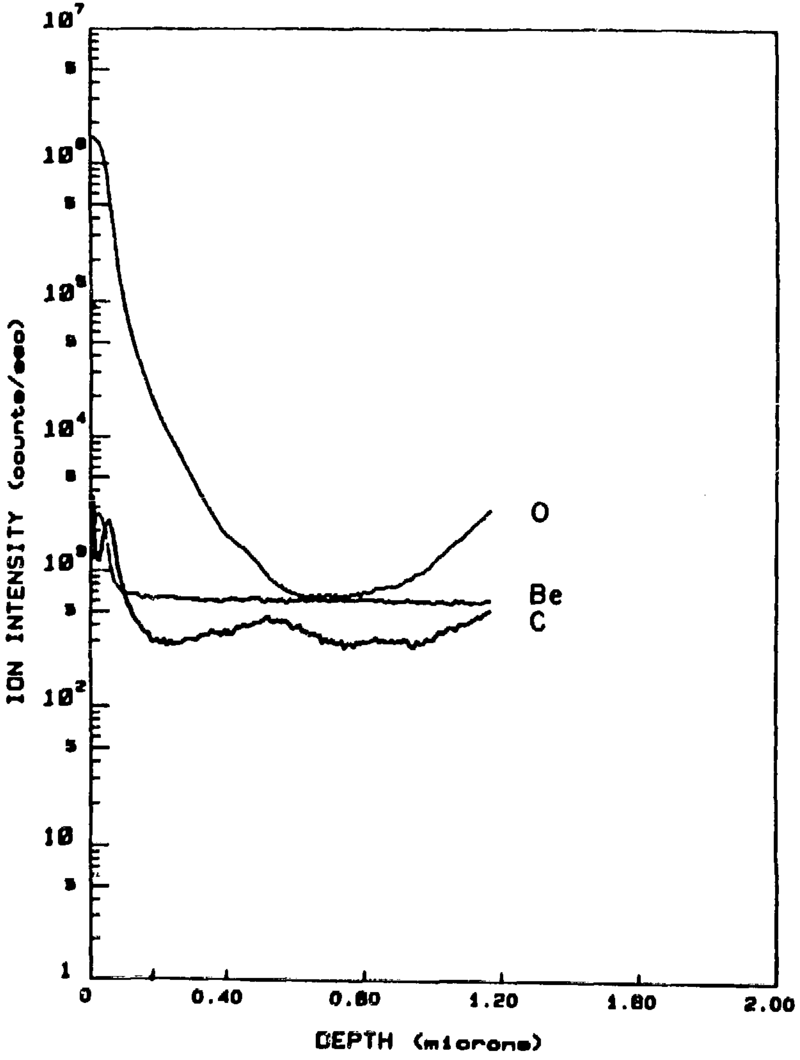


SPUTTERED BERYLLIUM

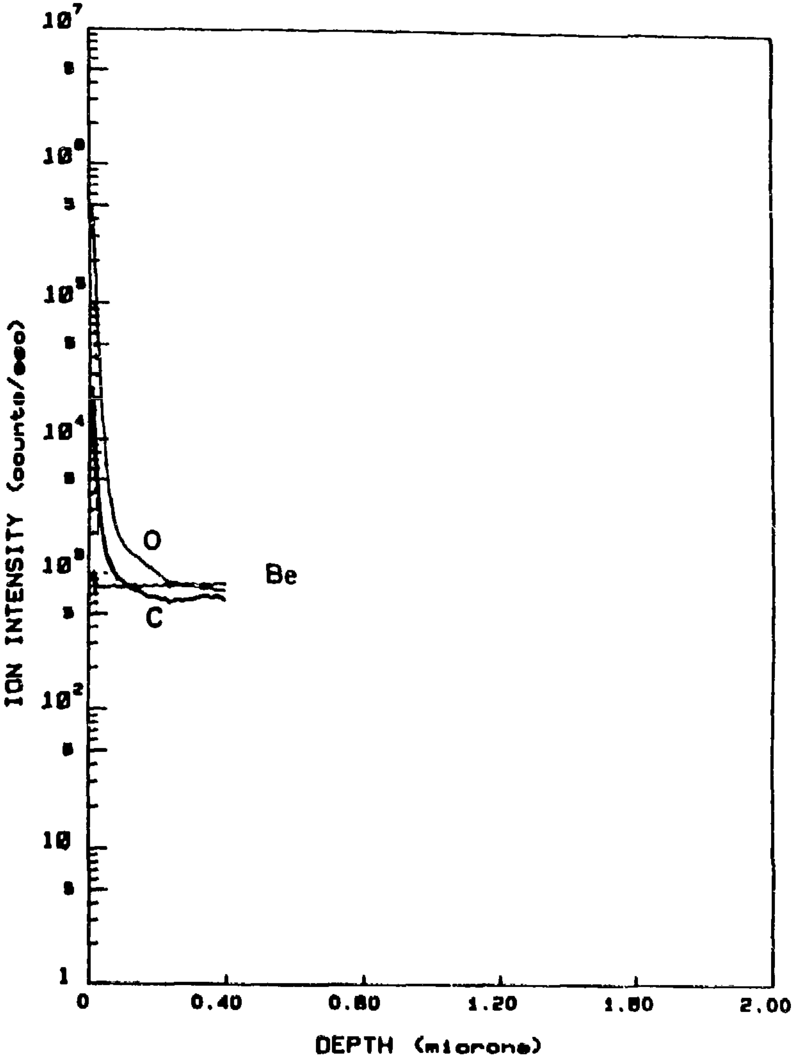


SPUTTERED BERYLLIUM



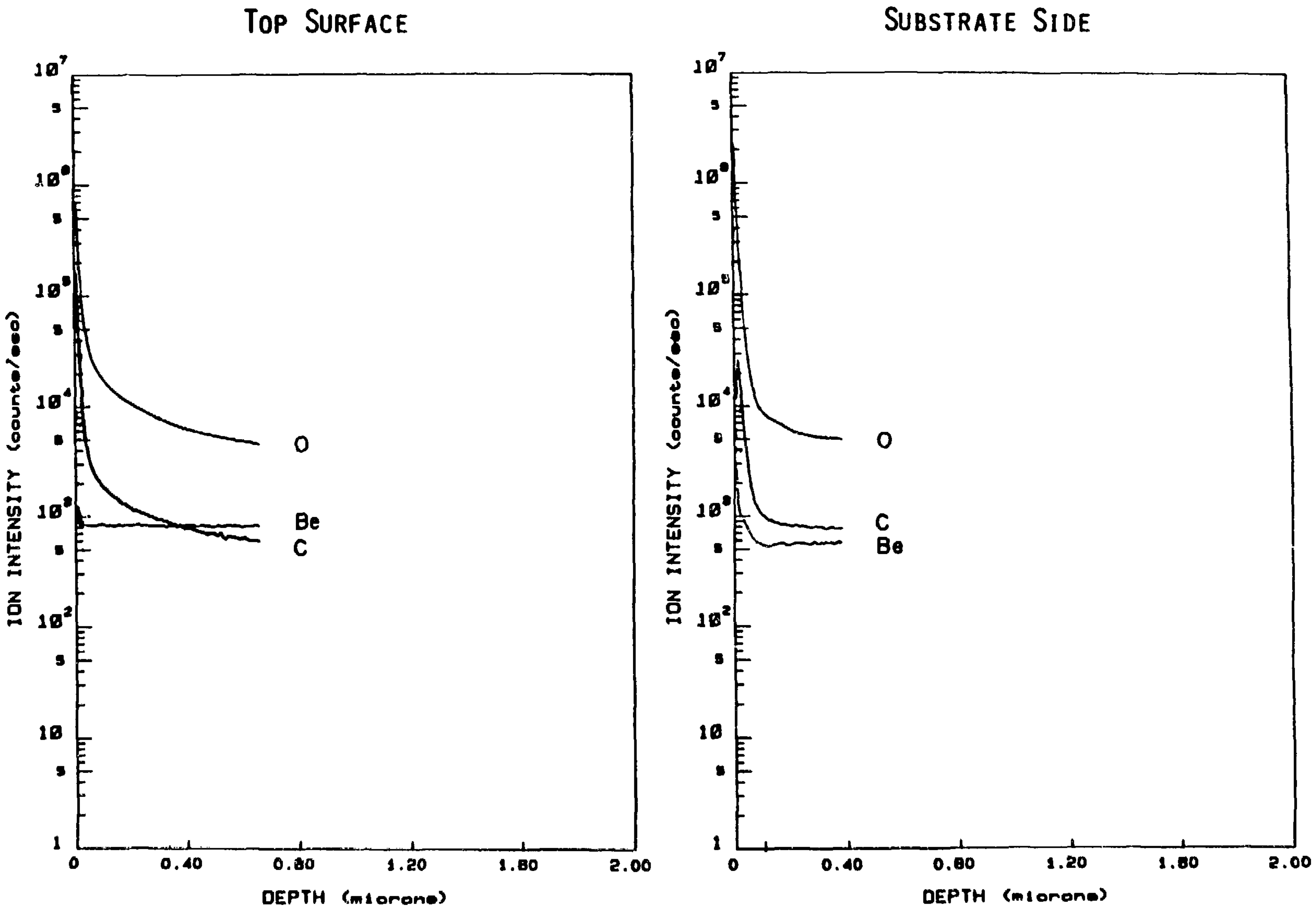


ELECTROFUSION FOIL

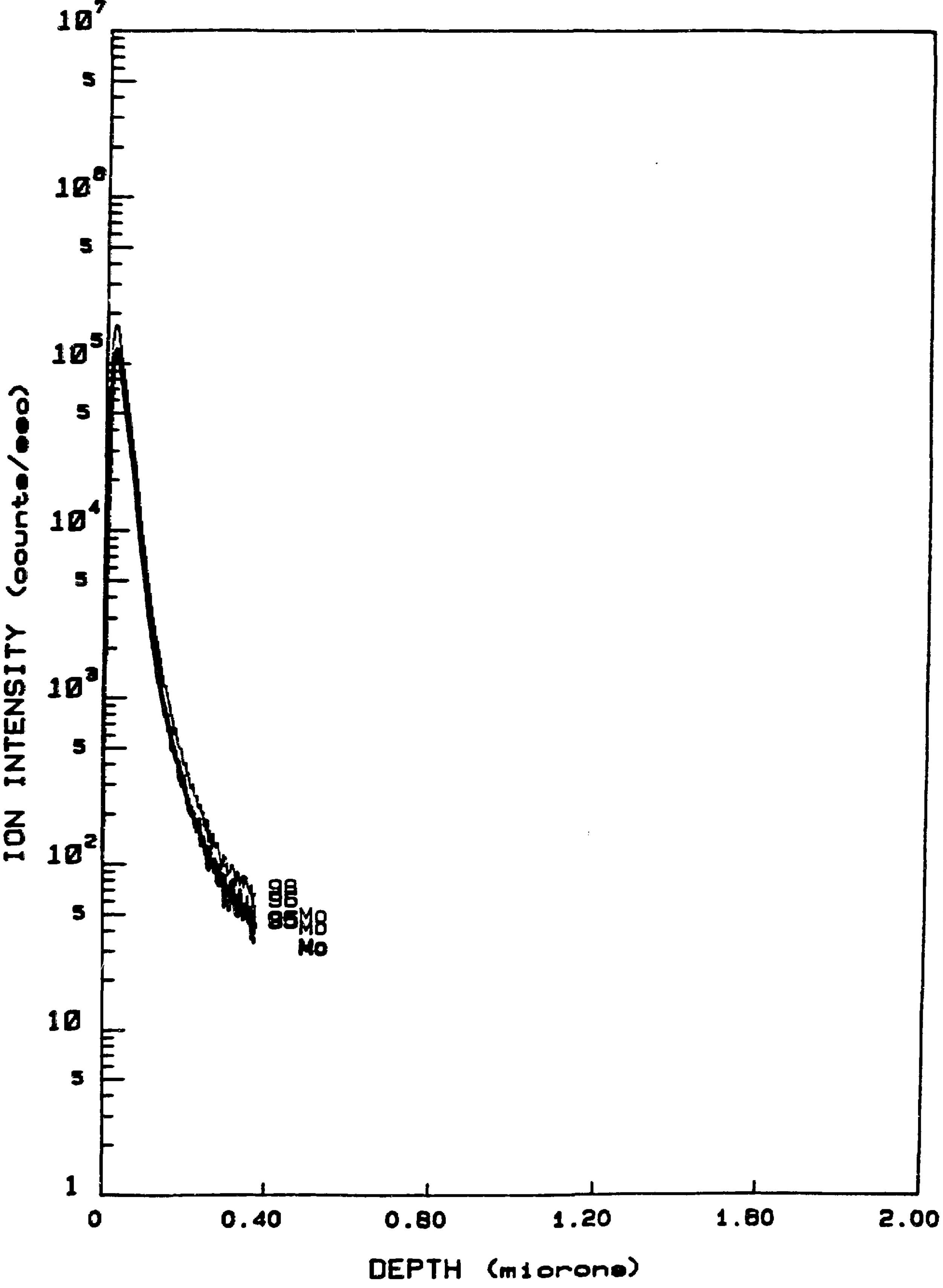


CHEMICALLY ETCHED

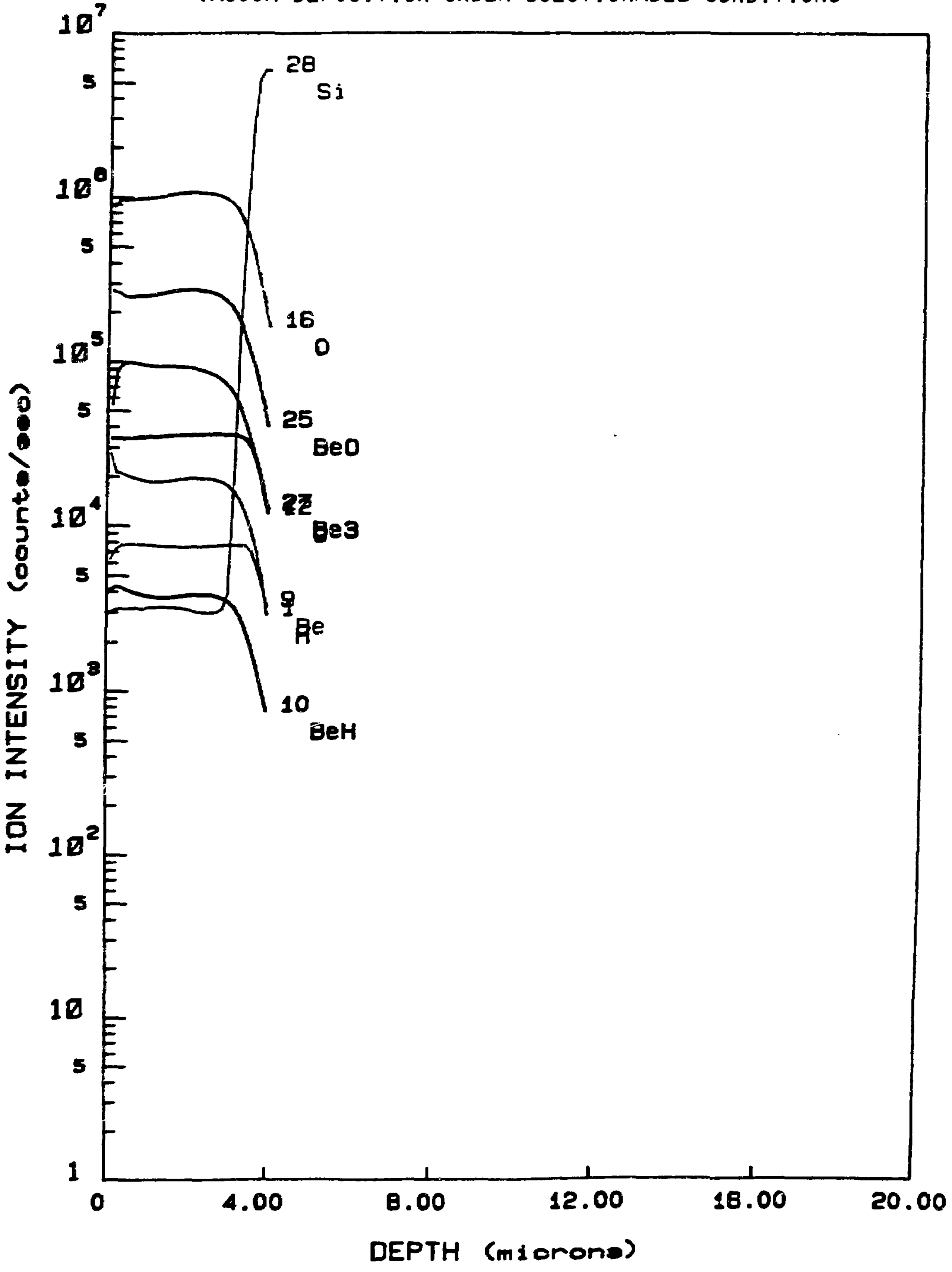
FOIL PREPARED BY PHYSICAL VAPOR DEPOSITION



FOIL PREPARED BY PHYSICAL VAPOR DEPOSITION



VACUUM DEPOSITION UNDER QUESTIONABLE CONDITIONS



PARTICLE DIAMETERS OBSERVED IN A 150-μm SQUARE FIELD AS A FUNCTION OF OXYGEN CONCENTRATION AND THE NUMBER OF OBSERVED PARTICLES, N_{OB}

N_{OB}	Oxygen Atom Fraction			
	10^{-5}	10^{-4}	10^{-3}	10^{-2}
	Particle Diameter (μm)			
1	0.474	1.50	4.74	15.00
2	0.335	1.06	3.35	10.61
3	0.274	0.866	2.74	8.66
5	0.212	0.671	2.12	6.71
10	0.150	0.474	1.50	4.74

SUMMARY

ION MICROANALYSIS IS A POWERFUL AND VERSATILE TECHNIQUE TO ANALYZE MATERIALS FOR LOW LEVELS OF CONSTITUENT AND IMPURITY ELEMENTS AND TO DETERMINE SPATIAL DISTRIBUTIONS OF THE ELEMENTS ON A MICROSOCPIC SCALE.

WITH PROPER SURFACE PREPARATION, ION-IMPLANTED BERYLLIUM SPECIMENS WILL PROVIDE RELIABLE STANDARDS FOR QUANTITATIVE ANALYSIS OF OXYGEN IN BERYLLIUM.

THE OXIDE CONTENT RESIDING IN OXIDE PARTICLES MUST BE ACCURATELY DETERMINED WHEN PERFORMING SIMS QUANTITATIVE ANALYSES FOR OXYGEN IN BERYLLIUM.

SIMS DEPTH PROFILES PROVIDE EXCELLENT CHARATERIZATION OF THE OXYGEN AND CARBON DISTRIBUTIONS IN BERYLLIUM FOILS.

APPLICATION OF MEV ION BEAM TECHNIQUES FOR DETERMINATION OF OXYGEN ON AND IN BERYLLIUM

R. G. Musket
Materials Division
Chemistry & Materials Science Department
Lawrence Livermore National Laboratory
Livermore, California 94550

ABSTRACT

Routine backscattering measurements of MeV helium ions from beryllium can be used to determine quantitatively the amount of oxygen on the surface with a sensitivity of a few times 10^{14} oxygen atoms per cm^2 and the bulk oxygen concentration with a detection limit of about 350 atomic parts per million (appm). We have performed simultaneous backscattering and particle-induced x-ray emission (PIXE) measurements to improve the detection limit for bulk oxygen to about 10 appm. The basis for and the results from these and other ion beam analysis measurements will be described.

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ANALYSIS OF OXYGEN ON AND IN BERYLLIUM USING 2 MeV HELIUM IONS *

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Analysis of oxygen on beryllium can be routinely performed using helium-ion backscattering (RBS). However, determination of the bulk oxygen concentration by this technique is limited to about 350 atomic parts per million (appm). We have performed simultaneous RBS and particle-induced X-ray emission (PIXE) measurements to improve the detection limit for bulk oxygen. The RBS measurements allowed determination of the surface oxygen before and after in situ sputter cleaning by argon ions in an ultra-high-vacuum system. PIXE measurements of specimens with surfaces maintained clean by sputtering permitted assessment of the concentration of oxygen in the bulk. For our geometry and detector sensitivities, 90% of the oxygen X-ray signal originated in the first 2.1 μm of the beryllium and a detection limit of 10 appm was found.

1. Introduction

We have investigated the application of simultaneous Rutherford backscattering (RBS) and particle induced X-ray emission (PIXE) measurements [1] for the analysis of oxygen on and in beryllium. The measurement goals were to determine surface oxygen with a detection limit of a few times 10^{14} oxygen atoms/cm^2 and bulk oxygen with a sensitivity approaching 10 appm in a volume large enough to be representative of the bulk.

The RBS measurements gave the surface oxygen before and after in situ sputter cleaning by argon ions in an ultra-high-vacuum system. PIXE from surfaces being maintained clean by the argon ions permitted assessment of the concentration of oxygen in the bulk. Simultaneous measurements ensured identical surface conditions for the RBS and PIXE spectra. Since the RBS results are quantitative and the PIXE results can be directly related to an analytical calculation without any unknown parameters, this combined approach is quantitative. Another important aspect of this measurement is the relatively large area (~ 5 × 5 mm^2) sampled, which should correctly assess the area-averaged amounts of oxygen in solution and in BeO particulates. Finally, this approach is basically nondestructive because (1) only the surface oxide need be removed for the highest-precision analysis, and (2) lower-precision measurements can be made without removing the surface oxide. To our knowledge there is no other measurement procedure that meets the measurement goals.

2. Calculation of helium-induced O(K) X-rays

The RBS determination of the oxygen surface density on beryllium from the ratio of the net counts under the oxygen surface peak to the height of the beryllium edge, corrected by appropriate scattering and stopping cross-sections, followed standard calculational procedures [2]. Consequently, this determination needs no further discussion. In contrast, a brief description of the calculation of the helium-induced O(K) X-rays based on the measurement geometry shown in fig. 1, will be presented.

For the surface oxide, the relevant equations have been previously published [3]. In particular, for oxides thin enough for the assumption of uniform X-ray production throughout the oxide and negligible X-ray absorption, the relevant equation for the detected O(K) X-ray intensity I_{DS} per 10^{15} oxygen atoms/cm^2 per 100 μC of 2 MeV He$^+$ can be written as

$$I_{DS} = 6.24 \times 10^{29} \eta_0 \omega_k \sigma_i (2\ \text{MeV})\, d\Omega/4\pi, \qquad (1)$$

where η_0 is the overall detection efficiency for O(K) X-rays, ω_k is the fluorescence yield for O(K), σ_i (2 MeV) is the oxygen K-shell ionization cross section (cm^2) for the incident 2 MeV helium ions, and $d\Omega/4\pi$ is the fractional solid angle subtended by the X-ray detector. Estimating the Si(Li) detector constant $C_d = \eta_0\, d\Omega/4\pi$ to be 7×10^{-7}, using $\omega_k = 7.7 \times 10^{-3}$ [4], and using $\sigma_i = 9.127 \times 10^{-19}$ cm^2 [5], we calculated that I_{DS} would be 2.22×10^4 O(K)/100 μC for 10 Å of BeO with a density of 3.01 g/cm^3. Considering the tabulated $\sigma_i(E)$ values given by Cohen and Harrigan [5] and Ziegler's dE/dx values [6], eq. (1) holds (within 2%) for BeO thicknesses below 160 Å.

Consider beryllium, with a clean surface, that is pure enough to permit use of the mass absorption coeffi-

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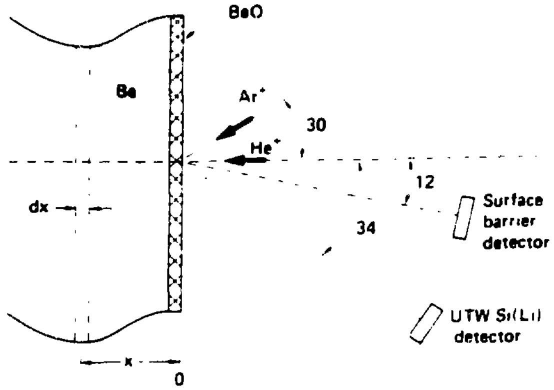

Fig. 1. Schematic of measurement arrangement.

cients and stopping powers for pure beryllium. For this initial calculation, assume that the concentration of oxygen is not a function of depth; this assumption should be very good for beryllium after removal of a considerable amount of surface material. The signal from oxygen in the bulk was calculated using stepwise integration of the contributions of each thickness interval dx of fig. 1. For each dx at a depth x, the contribution to the detected signal per 1 appm per 100 μC of helium ions can be written as

$$\mathrm{d}I_{\mathrm{DB}} = 7.7 \times 10^{27} \eta_0 \omega_k \sigma_i [E(x)] \times \exp[-10^{-4} \mu x / \cos 34°] \frac{\mathrm{d}\Omega}{4\pi} \mathrm{d}x, \qquad (2)$$

where the units of x and dx are μm, $\mu = 3920$ cm^2/g [7] is the mass absorption coefficient of O(K) X-rays in beryllium, and the constant includes the atomic density of 1 appm oxygen in beryllium, the number of ions in 100 μC, and the conversion of μm to cm. With dx = 0.0115 μm, the calculation yielded $I_{\mathrm{DB}} = 37.4$ O(K)/(appm 100 μC). The stepwise integration also showed that 50, 90, and 95% of the oxygen X-ray signal originate, respectively, in the first 0.66, 2.1, and 2.6 μm of the beryllium.

Comparing this result for bulk oxygen to that found above for a surface oxide, leads to the conclusion that the O(K) X-ray signal from 10 Å of surface BeO would be equivalent to that from about 600 appm of oxygen in the bulk. Therefore, high precision determination of the bulk oxygen level requires that the surface oxide be reduced to a negligible average thickness. The signal from 0.33 Å of surface BeO (i.e., 2.4×10^{14} O/cm^2) would equal that from 20 appm of oxygen in the bulk. These considerations were the basis for the requirement of in situ sputter cleaning of the beryllium surface during simultaneous RBS and PIXE measurements.

3. Experimental considerations

The measurements were made in an ultra-high-vacuum (UHV) chamber to minimize reoxidation of the beryllium surfaces during sputtering. Ultrahigh purity beryllium specimens were obtained from a commercial supplier [8]. They were chemically etched to remove any thick oxides, oxygen concentration profiles, and other surface contaminates. After etching, specimen ≠29A was exposed to laboratory air for more than six months before the as-received measurements were made.

The helium ions were generated by a Penning ion source and accelerated by a 3 MV Van de Graaff accelerator. Visual observation of the helium beam (typical current of 50 nA) on a phosphor screen at the target position showed that the beam was slightly less than 5 × 5 mm^2. A differentially pumped 5 kV ion gun provided the argon ions for sputtering. We used 3.25 keV Ar$^+$ ions (typical current of 5 μA) because this was the maximum energy, consistent with the available electronic raster, to achieve a clean spot of approximately 6 mm^2 at the target position.

Backscattered helium particles were analyzed using a UHV-compatible, ion-implanted silicon detector at room temperature [9]. Detection of the PIXE spectrum was accomplished using an ultra-thin-windowed Si(Li) detector having a compact magnetic deflection unit for removal of the backscattered ions from the X-ray axis [10].

The high-precision measurement procedure consisted of simultaneous RBS and PIXE measurements for three different specimen conditions: (1) as-received condition using accurate current integration of the helium beam, (2) after removal of the surface oxygen, but during continuous argon-ion sputtering to maintain the cleanliness, and (3) after sputter removal of various amounts of beryllium, but during argon-ion sputtering.

4. Results and discussion

Fig. 2 shows the relevant part of the RBS spectra of beryllium ≠29A for both the as-received and sputter-cleaned conditions. The initial surface oxygen level was determined to be 1.4×10^{16} O/cm^2 (i.e., about 19 Å of BeO), and the spectrum for the cleaned surface displayed no signs of surface oxygen. However, the 3-sigma value for the background counts at the position of the oxygen peak sets the maximum amount of surface oxygen on the cleaned surface to less than 4×10^{14} O/cm^2.

The PIXE spectra displayed in fig. 3 correspond to the as-received and sputter-cleaned RBS spectra of fig. 2. Determination of the net O(K) counts required the subtraction of the bremsstrahlung background from each spectrum. The shape of this background was obtained

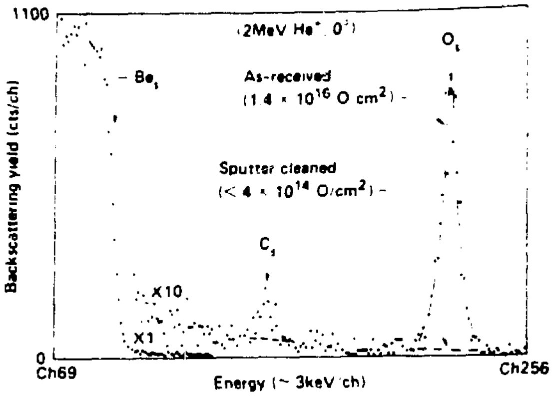

Fig. 2. Helium-ion backscattering spectra for Be≡29A in the as-received and sputter-cleaned conditions.

by a spectrum stripping procedure. There was a large dip in the bremsstrahlung spectrum corresponding to absorption by the oxygen (K edge) in the ice layer on the entrance surface of the Si(Li) detector. This effect has been described previously [11].

We determined the Si(Li) detector constant C_d by comparing the measured change in the net O(K) signal for the measured change in the surface oxygen level (as determined by RBS) with the values calculated by eq. (1). By this procedure C_d was found to equal 1.24×10^{-6} instead of the estimated 7×10^{-7} value used above in the calculation section. Thus, the calculated, detected O(K) yield from the bulk, I_{DB}, becomes 68.1 O(K)/(appm 100 μC). This value was used to determine the oxygen concentration in the bulk from the measured, net O(K) yield. For the sputter-cleaned spectrum of fig. 3, the bulk oxygen concentration was found to be 220 appm. The minimum detectable level was calculated from the 3-sigma value of the background counts under the O(K) peak to be 10 appm.

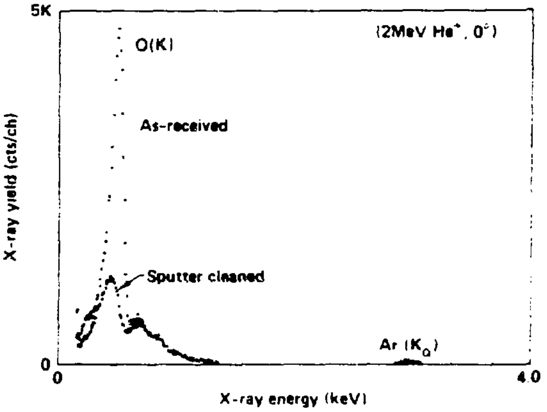

Fig. 3. Helium-ion induced X-ray spectra for Be≡29A obtained simultaneously with the backscattering spectra of fig. 2.

The sputtering yield for 3.25 keV argon ions incident at 30° from the normal can be estimated to be 1.5 Be/Ar [12]. Once the surface was clean, the O(K) yield from the bulk was constant within the statistics of the measurement (i.e., within ~ 10%) for removal of up to 0.18 μm of beryllium. This supports the assumption that the bulk oxygen concentration was independent of depth because we are most sensitive to oxygen near the surface.

5. Concluding remarks

We have demonstrated high-sensitivity analysis of oxygen on and in beryllium using simultaneous RBS and PIXE measurements. The novel aspect was the detection and use of the helium-induced O(K) X-rays for determination of the bulk oxygen concentrations in a representative volume of beryllium with a detection limit of about 10 appm. We have used this quantitative approach to measure oxygen in beryllium at concentrations between 170 and 3600 appm.

It is pleasure to acknowledge S.J. Holmes and J.R. Thournir for their able technical assistance in the experimental aspects of this work, F. Fulton for supplying the etched beryllium, and A. Toor for his initial and continuing support of this analysis method.

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SELECTION OF AN INSTRUMENTAL TECHNIQUE FOR THE TRACE ELEMENT ANALYSIS OF BERYLLIUM

by

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Summary

The trace element analysis of beryllium is discussed with particular reference to those techniques available at the Royal Ordnance Factory. These are:-

1. Gas fusion techniques.
2. Ultra-violet visible spectrometry.
3. Atomic absorption spectrometry.
4. X-ray fluorescence spectrometry.
5. Energy dispensive X-ray analysis.
6. Inductively coupled plasma atomic emission spectrometry.

Each method is discussed separately beginning with an introduction to it's principles and then assessing it's advantages and disadvantages.

SELECTION OF AN INSTRUMENTAL TECHNIQUE FOR THE TRACE ELEMENT ANALYSIS OF BERYLLIUM.

INTRODUCTION

Many elements, found in trace quantities, can significantly influence the mechanical and nuclear properties of beryllium. It is therefore necessary to select a method of analysis that provides the required sensitivity, accuracy and precision at the concentrations found in commercial grades.

This paper is not meant to provide an exhaustive assessment of all possible methods available to the analyst but to discuss only those used at the Royal Ordnance Factory - Cardiff.

These are :-

(1) Gas fusion techniques.

(2) Ultra-violet / visible spectrometry.

(3) Atomic absorption spectrometry.

(4) X-ray fluorescence spectrometry.

(5) Energy dispersive X-ray analysis.

(6) Inductively coupled plasma optical emission spectrometry.

Each of the above methods will be discussed seperately, begining with a brief introduction to each of the technique's principle. The advantages and disadvantages will then be discussed with particular reference to the analysis of beryllium.

Gas fusion techniques.

The instruments used at ROF(C) are :-

(a) Leco CS 46 for the determination of carbon & sulphur.

(b) Leco RO 17 for the determination of oxygen.

Principle.

(a) For carbon and sulphur.

The sample is combusted with oxygen in an induction furnace. Approximately 97% of the carbon is oxidised to CO2 and about 3% combusts to CO, the sulphur oxidises to SO2. All these gases are detected seperately by passing them to an infra-red detector and measuring the absorption of radiation at the three gases characteristic wavelengths. The degree of absorption is proportional to the concentration of carbon and sulphur present in the sample.

(b) For oxygen.

The sample is fused in a graphite crucible in an electrode furnace. Argon carrier gas transports the released oxygen. as carbon monoxide, to an infra-red detection cell. and is measured as per (a) above.

Advantages.

1. The method is simple and automated, consisting essentially of weighing the sample, pressing the analyse switch and reading the results from a digital display or printer.

2. Calibration can be from easily available standards, or in the case of the oxygen analyser, carbon monoxide gas.

3. Good dynamic range. The oxygen analyser, for example, can determine levels between 0.01 & 50% oxygen.

Disadvantages.

1. These instruments can only be used for a limited number of elements ie. carbon, sulphur and oxygen.

2, Each machine costs in the region of £30,000 ($50,000)

Ultra-violet / visible spectrometry. (UV/Vis)

Principle.

Ultra-violet or visible radiation, characteristic of the species being analysed, is passed through a cell containing the sample in liquid form. A detctor measures the intensity of radiation transmitted through the cell so that, in accordance with Beer's law, the amount of radiation absorbed is proportional to the quantity of analyte present.

Advantages.

1. Methods are available for measuring most of the important trace elements in beryllium, including some that are difficult to determine by other techniques, eg silicon and boron.

2. Good precision can be obtained with typical RSDs - 0.5 to 5.0%

3. Low cost instrumentation; typically £3000 - £7000 ($5000 - $12000)

4. Good sensitivity - LOD of silicon in beryllium metal - 10ppm
LOD of nickel in beryllium metal - 0.5ppm

Disadvantages

1. Each individual element to be analysed, will need a seperate preparation, each of which can be time consuming and may require a high level of scientific expertise.

Atomic absorption spectrometry. (AAS)

Principle.

A solution of the sample is aspirated into an acetylene/air or acetylene/nitrous oxide flame, producing an atomic vapour of the analyte. Characteristic radiation (of the element to be determined) is produced from a hollow cathode lamp and directed through the atomic vapour onto a photomultiplier. Electrons from atoms of the species to be determined absorb some of this radiation, causing them to be promoted to the next higher energy level. This absorption is detected by the photomultiplier and via the electronics is presented as a concentration.

Advantages

1. This is truely a trace element technique, with many elements having practical detection limits of less than 0.05ppm in solution.

2. Because the radiation source is specific for each element, very little spectral interference is observed.

3. Good precision - typically 0.5 - 2.0 RSD.

4. Instruments are available from approx £10000 ($16000)

Disadvantages

1. As the sample is required in solution a significant amount of preparation is needed. Each element must then be determined sequentially - although this is automated at ROF(C).

2. Individual source lamps are needed for each element.

3. As beryllium is a light metal; compared to other materials; it produces a high dissolved solids solution for a given dilution. The dilution used at ROF(C) is 0.5g / 100mls which gives a dilution factor of 200. Consequently, although AAS can theoretically be used to determine 70+ elements, this high dilution factor causes some of the elements, present in trace

concentrations (eg Bi & Mo), to be below the detection limit of the method.

4. Interferences due to stable compound formation, ionisation or matrix effects can be problematic using AAS. Most of these difficulties, however, can be overcome by selecting the most suitable atomising conditions for each particular element and by matching the standards to the samples.

X-Ray Fluorescence Spectrometry. (XRF)

Principle

A primary beam of X-rays directed onto a sample upsets the atomic equilibrium by removing inner orbital electrons, and a return to equilibrium, by transfer of electrons from one shell to another, resulting in the emission of secondary (fluorescent) X-radiation. This radiation contains wavelengths which are characteristic, qualitatively and quantitatively, of each element present. By means of a collimator, the parallel beam of secondary radiation is directed onto an analysing crystal which seperates it according to wavelengths.These in turn are reflected into a detector mounted on a high precision goniometer.Since the angular position of the crystal and goniometer is a function of wavelength, the elements of the specimen can be identified. And, since the intensity of the radiation is proportional to the concentration of the element, the amount of each element present can be determined.

Advantages.

1. XRF may theoretically be used for the determination of all elements in the periodic table from fluorine to uranium.

2. This is a non-destructive technique that may be used for solid or liquid samples.

3. Depending on sample composition, concentrations of a single element may be measured from ppm to 100%. Relative standard deviations of 0.5 - 5.0% are typical.

4. Sequential (for the highest precision and lowest detection limits) or simultaneous (for rapid analysis) instruments are available.

5. The technique is highly suitable for the trace element analysis of beryllium; providing solid samples can be easily prepared. Beryllium is almost transparent to X-rays so that good sensitivity can be obtained for many elements, giving LODs of between 5 & 50 ppm.

Disadvantages.

1. There are many factors that can influence the intensity of the characteristic fluorescent radiation, such as spectral interference, matrix absorption or surface finish. However, with the introduction of faster, more powerful computers, modern machines can deal with many of these interferences quickly and automatically.

2. High capital cost ranging from £50000 - £150000 ($80000 - $250000)

3. The sensitivity of the technique decreases with decreasing atomic number; elements with Z Nos less than 12 (magnesium) are particularly difficuult to determine, producing poor limits of detection.

4. Standards for use with the analysis of beryllium are not available.

Energy dispersive X-ray analysis. (EDX)

Principle

The principle is similar to that described above for XRF. With the EDX system used at ROF(C), however, the sample is exited, not from a beam of X-rays, but from a primary beam of electrons produced from a scanning electron microscope (SEM). These electrons cause ionisation of the analyte elements and, as per the XRF method, characteristic X-radiation is produced. Analysis is accomplished by energy dispersion, which is the segregation and measurement of X-rays according to their energy.

Advantages.

1. The unique combination of a SEM with an energy dispersive analyser can permit macroscopic and microscopic samples to be analysed for elemental composition in the region traversed by the electron beam. This focused electron beam allows a "spot analysis" of a $1\mu m^2$ section of specimen.

2. Display of the entire X-ray spectrum in digital format giving simultaneous multi element analysis and rapid qualitative analysis of non-standard samples.

Disadvantages.

1. Spectra obtained show poorer resolution than for fluorescent wavelength dispersion. Consequently precision is relatively poor.

2. Limits of detection, for all elements, are poor when compared with XRF, particularly those with low Z Nos.

Inductively coupled plasma optical emission spectrometry (ICP)

Principle.

The sample, in liquid form, is asperated into a high temperature (6000-10000 deg K) plasma causing electrons of the elemental atoms or ions to be promoted to higher energy levels. When these electrons drop back to their ground state, characteristic radiation is emitted; the intensity of which is proportional to the quantity of analyte present. This emitted light from the tail flame of the plasma is focused onto the entrance slit of a spectrometer which can either be of the simultaneous type or of the scanning, sequential type, used at ROF(C).

The instrument operated at ROF(C) is the Perkin Elmer Plasma-2. It is not sited in the Beryllium laboratory and so is not used routinely for the analysis of beryllium. However, experience of using the machine, with other materials, allows the following assessment to be made.

Advantages.

1. In principle all elements, with the exeption of argon, can be determined. In practice several elements (F, Cl, Br) require special optics for the transmission of the very short wavelengths.

2. Calibration curves are often linier over 5 orders of magnitude.

3. Good detection limits are obtainable for many elements, many below 0.01ppm.

4. Good accuracy and precision; RSDs of better than 1% are possible.

5. Although spectral and matrix interferences do occur, they are usually easily resolved with the aid of modern computer software.

Disadvantages.

1. ICP is a solution technique which therefore may require a significant amount of sample preparation.

2. Although generaly a sensitive technique, several elements (eg Cr, Ni, Zn) have poorer detection limits when compared to other methods such as AAS.

3. Instruments providing the best limits of detection and precision can be expensive - up to £140000 ($225000), although more modest machines are available for less than £50000 ($80000)

DISCUSSION

The choice of method of analysis depends, like anything else, on what is required. If only a small number of elements need be analysed on a small number of samples and capital cost is paramount, then UV Vis would be difficult to better. This technique is considered too time consuming, for routine analysis at ROF(C), however, although it is still used for the validation of other methods.

When accurate results for the determination of carbon and oxygen are required (as they usually are) the fusion instruments are almost essential. Although these elements can theoreticaly be determined by XRF or ICP, their sensitivity, accuracy and precision make these methods a poor second choice.

AAS has many advantages (as mentioned earlier), so that if only the common, metalic contaminents of beryllium (ie Fe, Al, Mg, Cr, Cu & Ni) need be determined, then the low instrument cost, relatively rapid analysis, good precision and detection limits make this technique the favoured choice. AAS has been used extensively at ROF(C) for many years, not only for the determination of trace metals in beryllium but also for the determination of beryllium in Health Physics samples. The technique has proved to be highly suitable in both areas of operation.

When large numbers of elements need to be determined then high capital cost can be justified. XRF and ICP are both suitable being highly automated and each being capable of analysing almost the whole of the periodic table of the elements. If solid samples need be tested, then XRF is the only choice.

The use of SEM/EDX cannot be considered a viable method of routine quantitative analysis because of its high capital cost, combined with its relatively poor resolution, precision and detection limits. However, no other technique combines the ability to observe a sample and at the same time produce a rapid qualitative, or even quantitative, analysis of the same area. Therefore this technique must be considered more as a tool for investigation. It can, for example, be used to observe a small inclusion and then chemically identify it: in this respect it is unique.

SUMMARY AND CONCLUSIONS

1. For the analysis of a limited number of elements on a small number of samples with a low capital invetement:-	UV/Vis
2. For the analysis of carbon & oxygen:-	Fusion techniques
3. To determine all the common contaminents of beryllium with good detection limits and reasonable capital cost:-	AAS
4. For the analysis of large numbers of elements on many samples:-	XRF or ICP
5. Metallographic reserch or the investigation of faults:-	SEM/EDX

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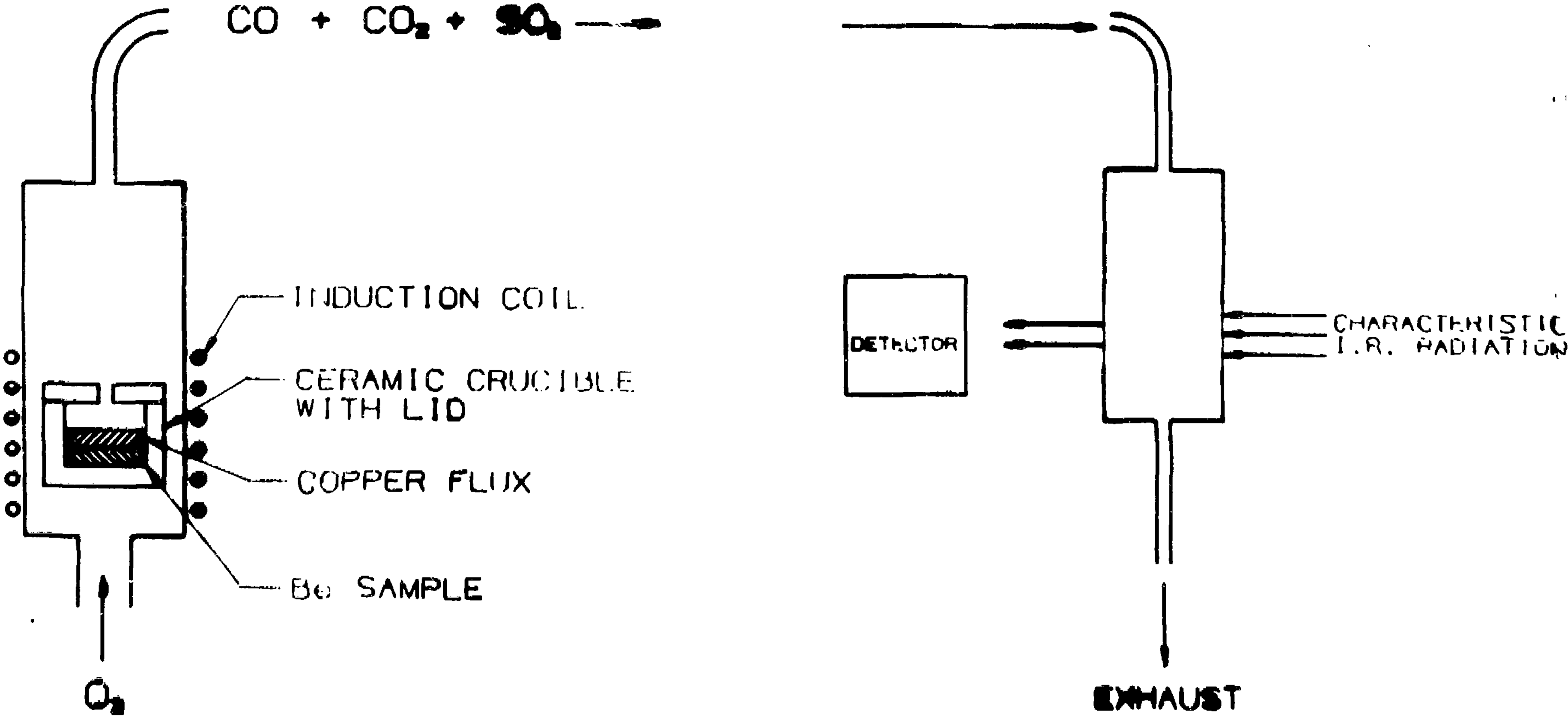
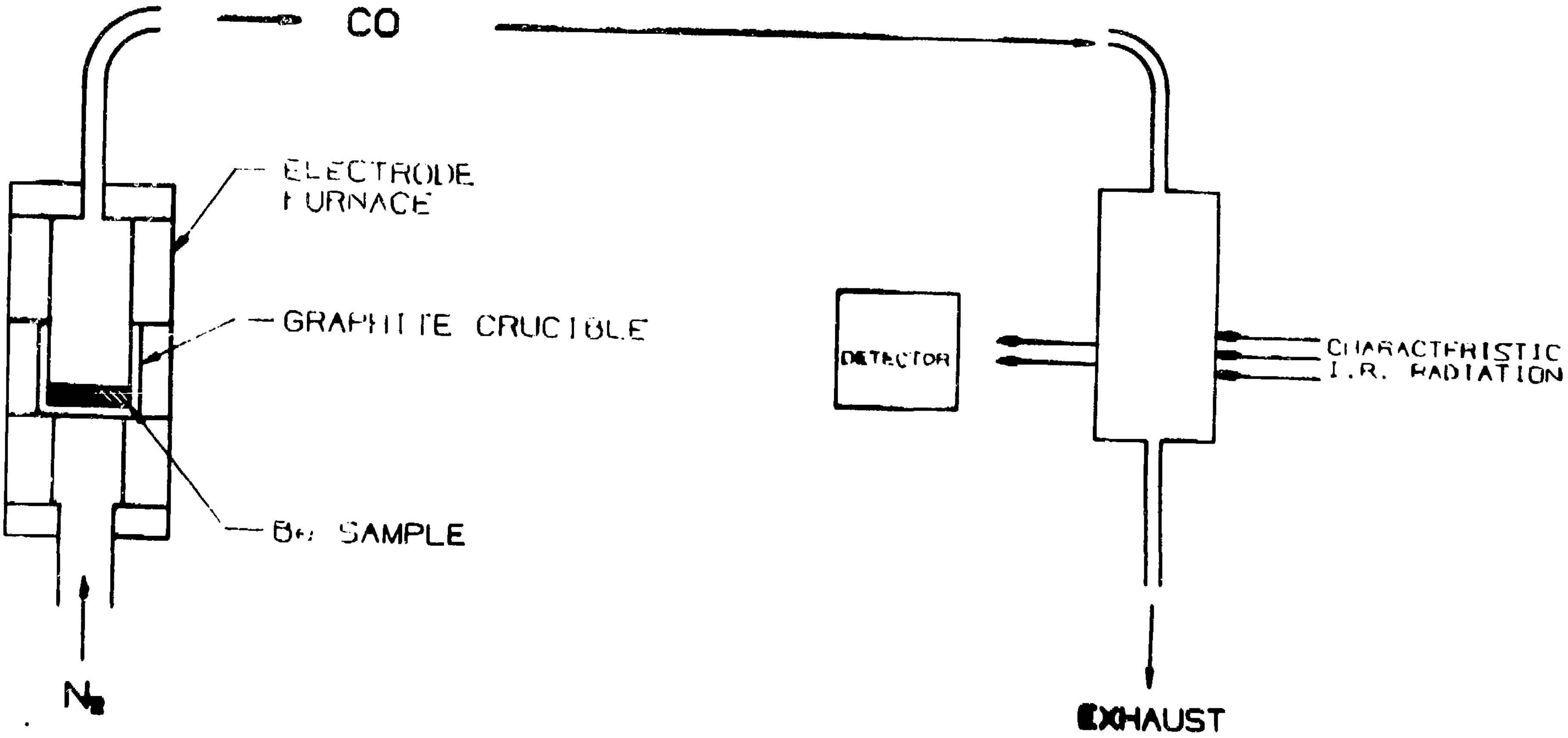
June 1987

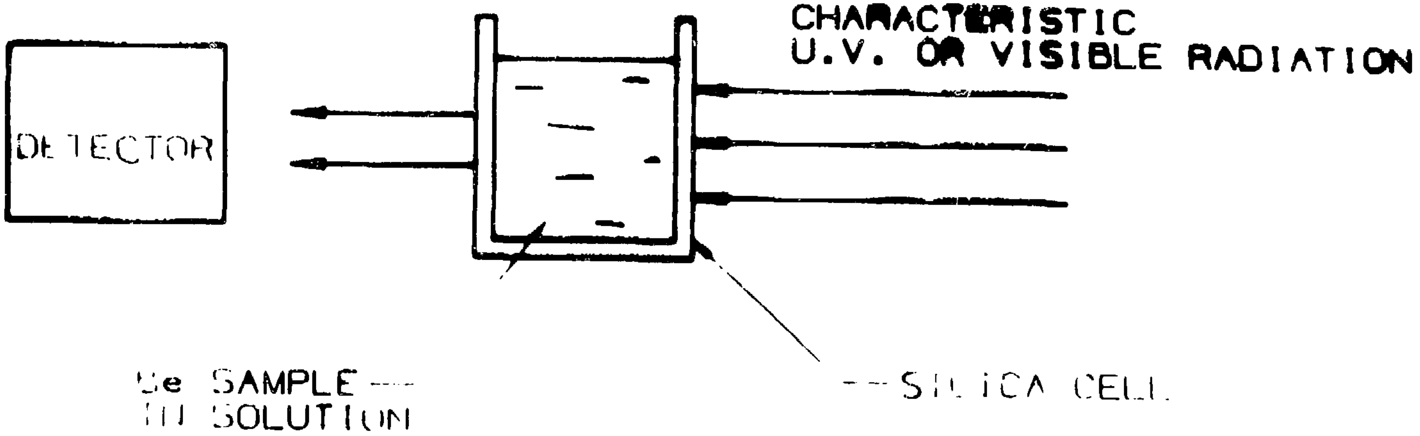
DETERMINATION OF CARBON AND SULPHUR USING THE FUSION TECHNIQUE



DETERMINATION OF OXYGEN USING THE FUSION TECHNIQUE



ULTRA-VIOLET/VISIBLE SPECTROMETRY



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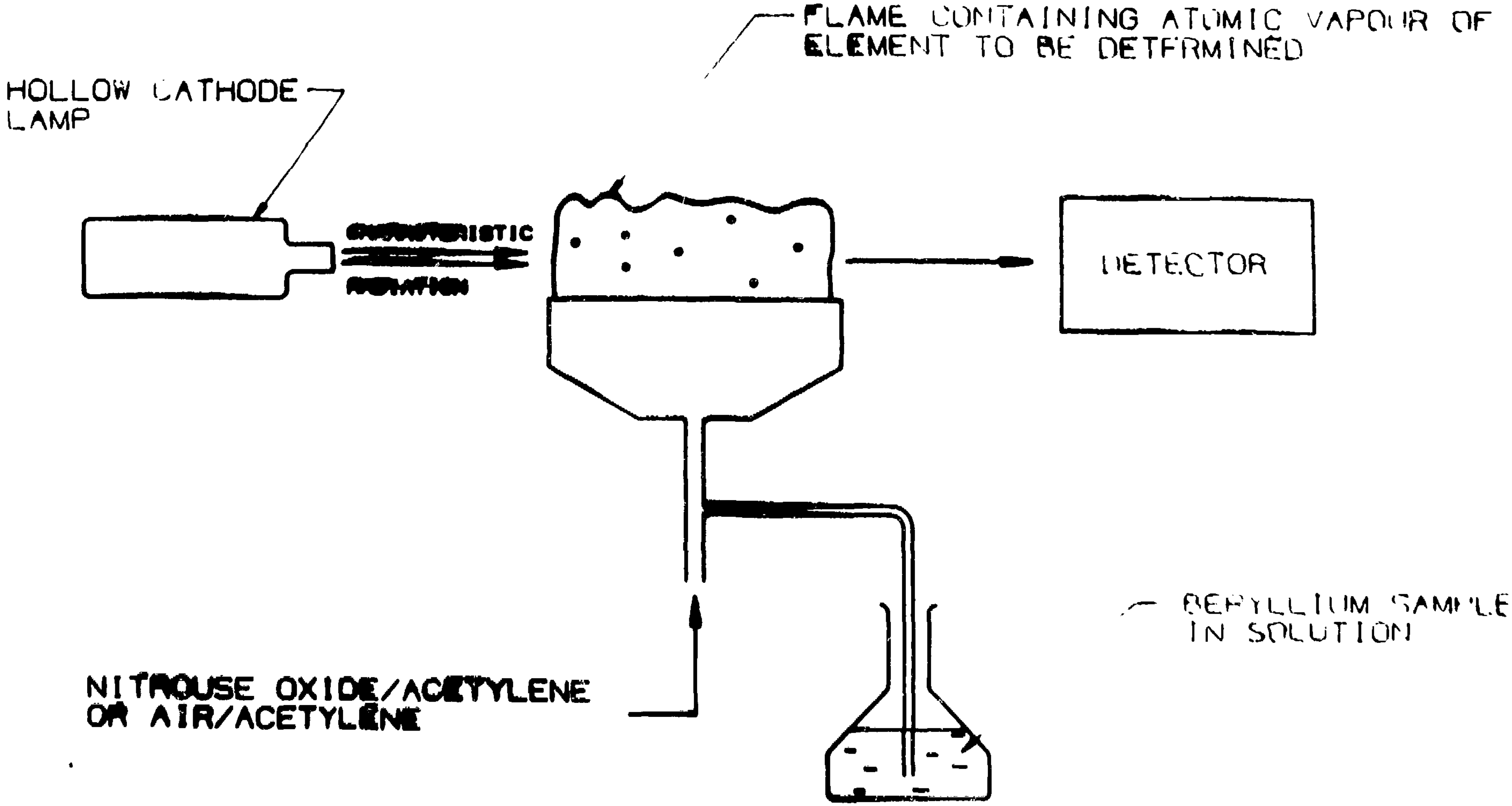

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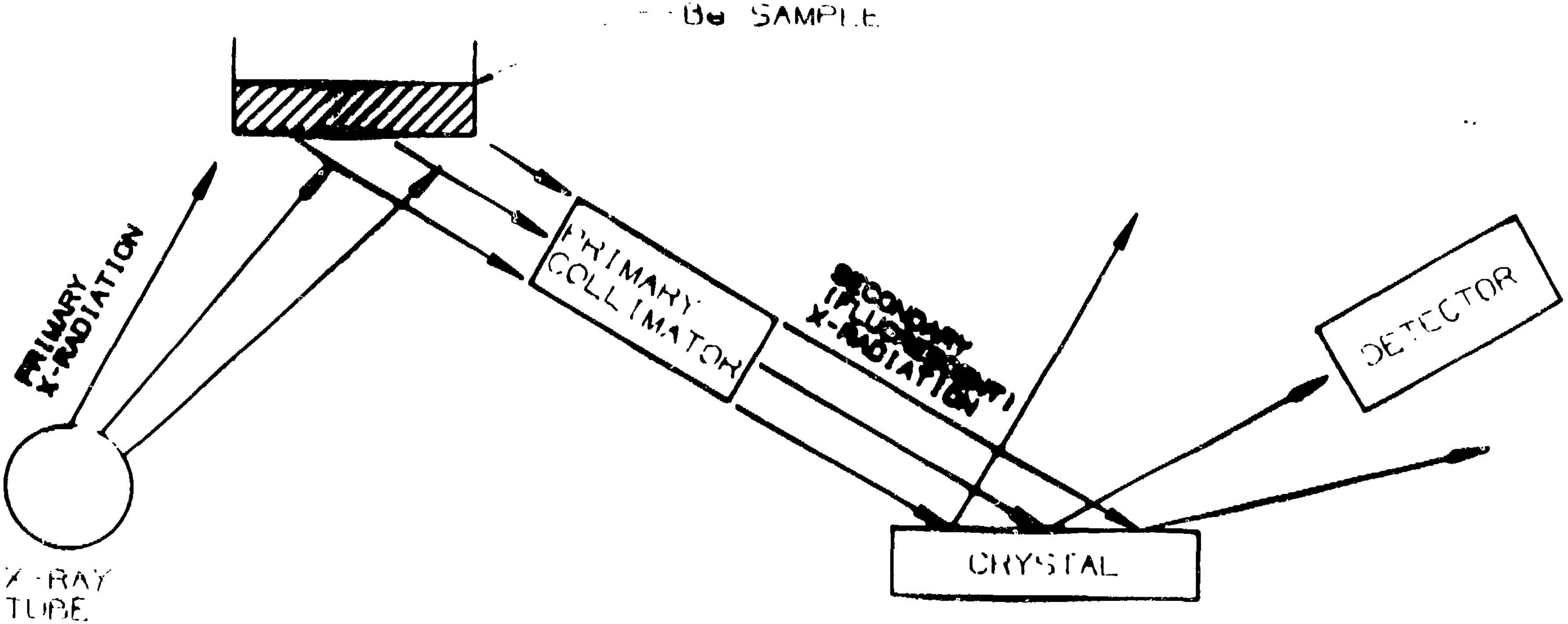

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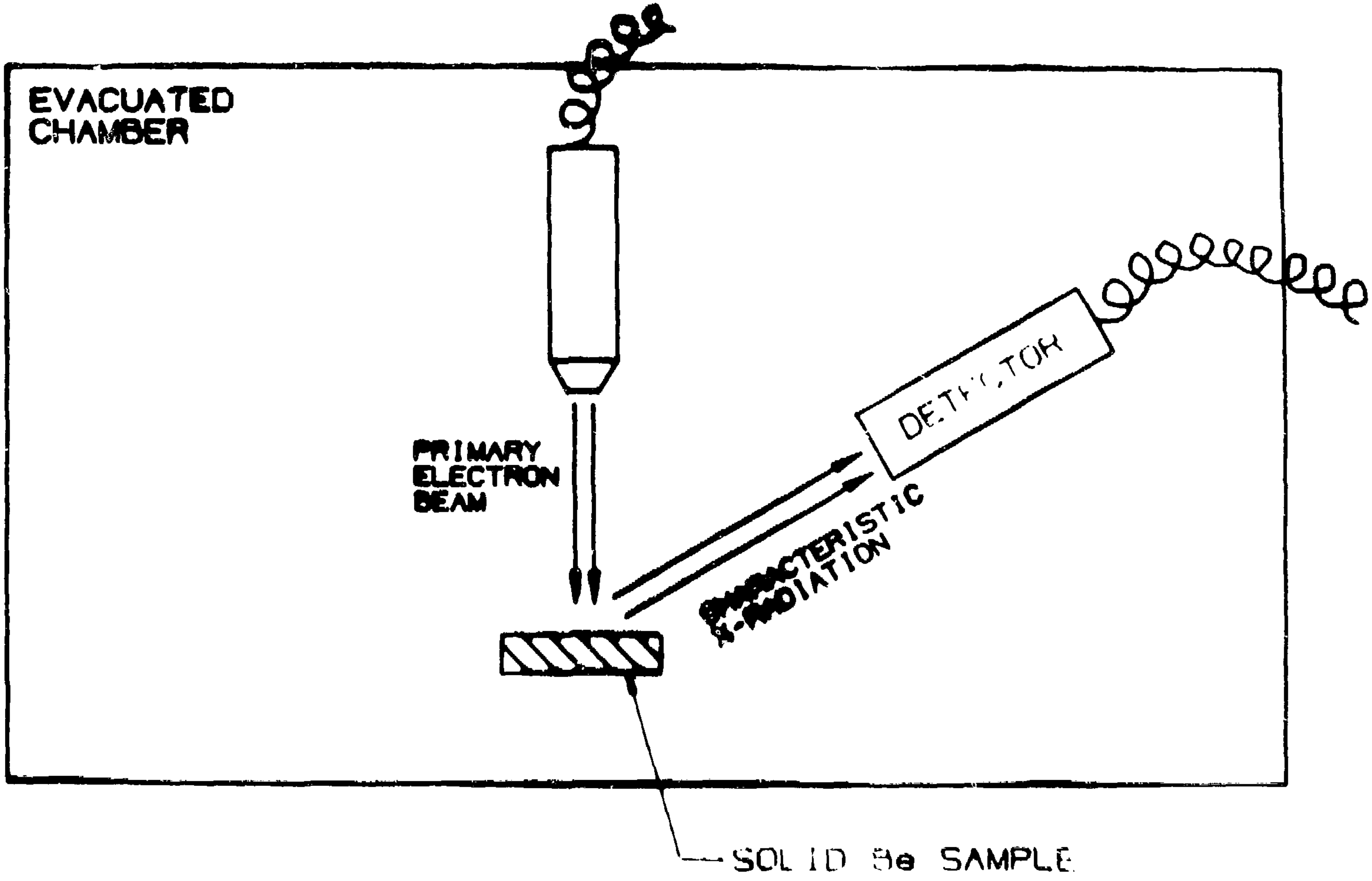
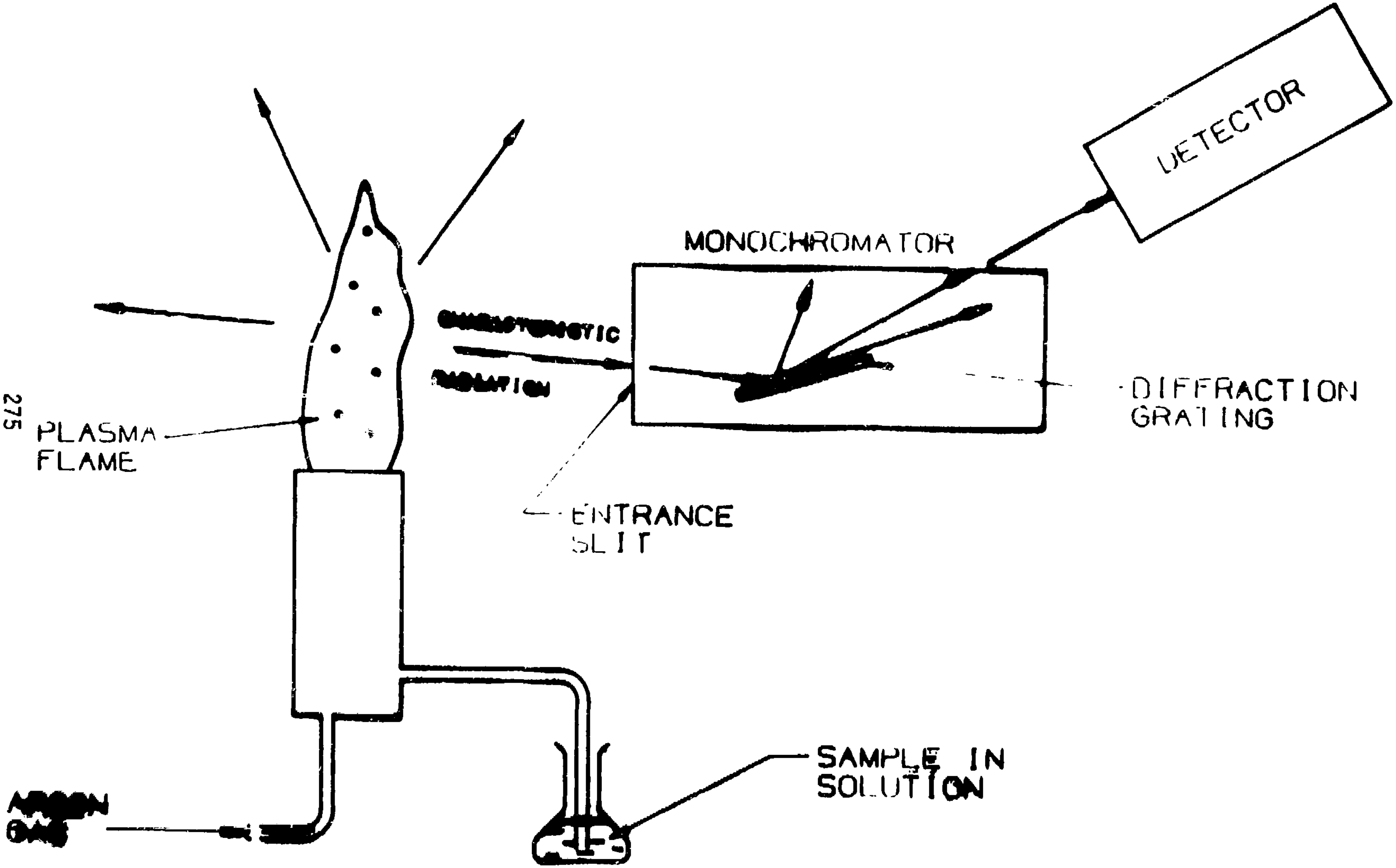


INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETRY



BERYLLIUM INERT GAS ATOMIZATION

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An atomization unit was constructed and operated to determine the feasibility of inert gas atomization as a method of producing spherical beryllium powder.

Twenty-nine atomization runs were performed to characterize the effects of process variables upon the product powder. The variables investigated were melt superheat, nozzle diameter, atomization gas pressure, and atomization gas oxygen content. Under the range of process variables employed, the -200 mesh powder yield varied from 3.9 to 26%. The powder was characterized by several methods, including chemical analysis, flow rate and packing density.

Billets consolidated from spherical atomized powder were fabricated by hot isostatic pressing and vacuum hot pressing. Evaluation of these billets revealed that tensile properties were similar to those made from other types of input powder with similar grain size. X-ray pole figure analysis revealed the absence of preferred crystallographic orientation.

This program successfully demonstrated the feasibility of atomization as a powder-making process for beryllium. It also gave considerable insight into the engineering considerations which should be addressed in the next atomizer design iterations.

ATOMIZATION OF BERYLLIUM

1. Review of Previous Work

2. Atomizer Upgrade.

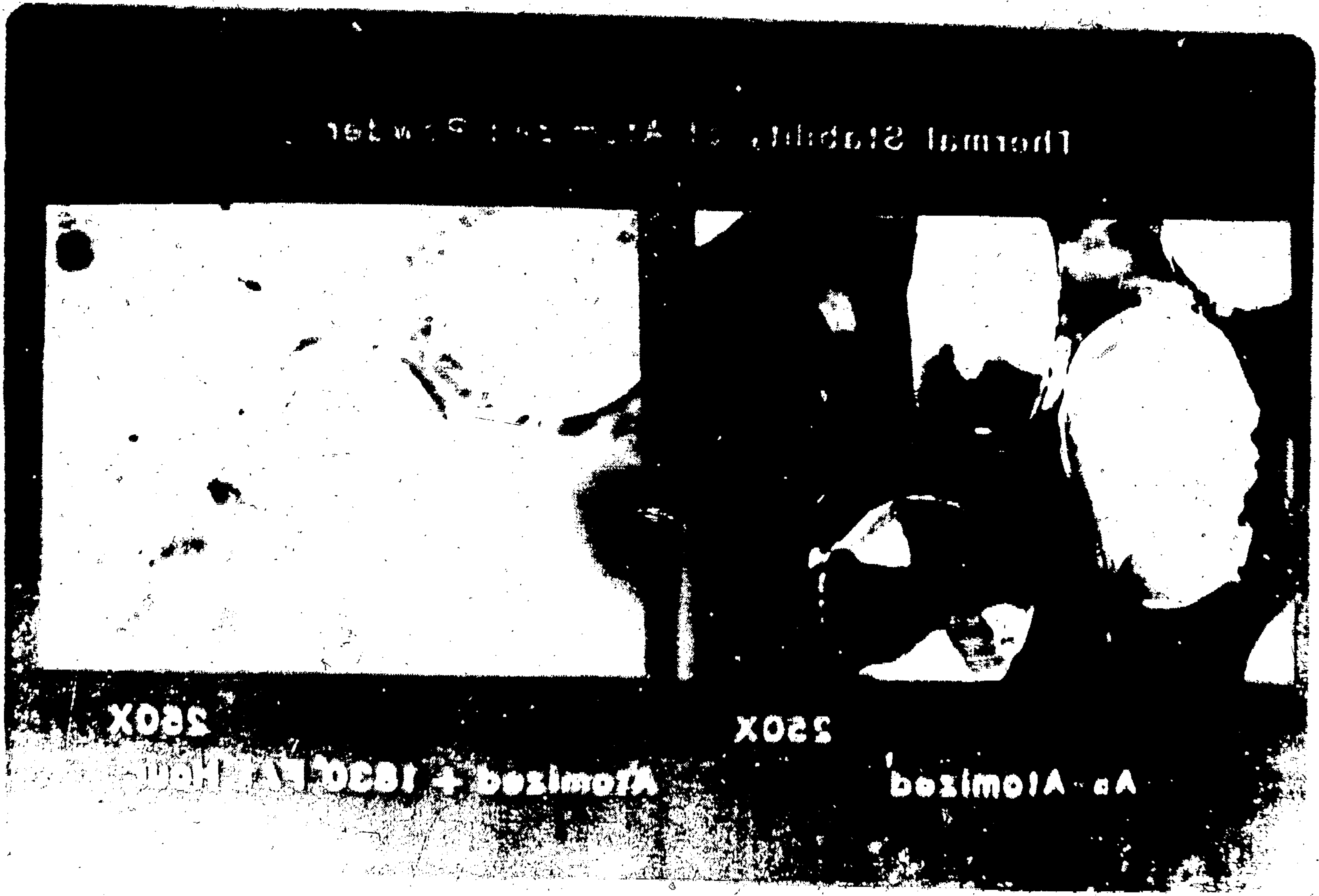
BACKGROUND

- DOE – Sponsored Atomizer Showed Laboratory Feasibility
  - 5lb. Unit
  - Low Fine Powder Yields Due to Small Size

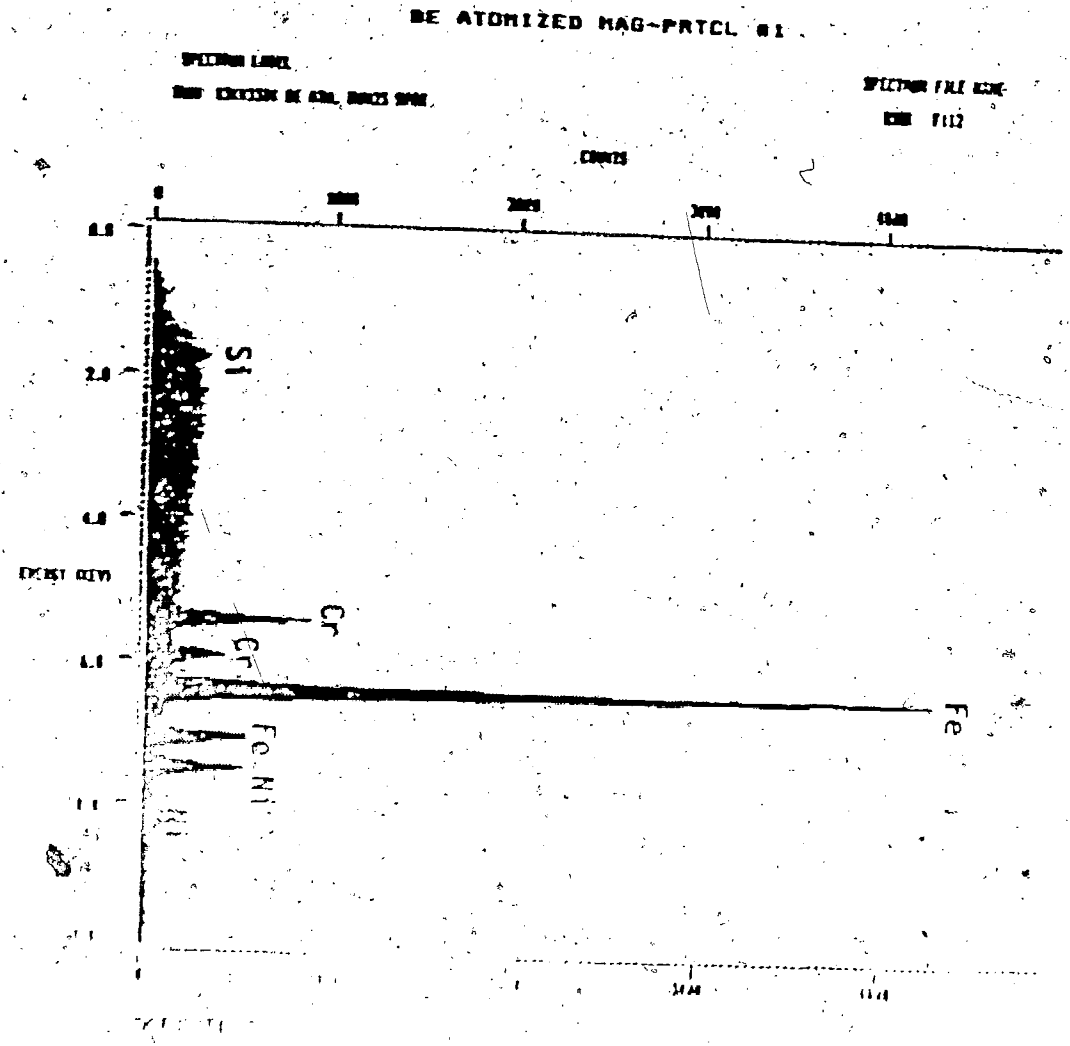
    Conservative Equipment Design

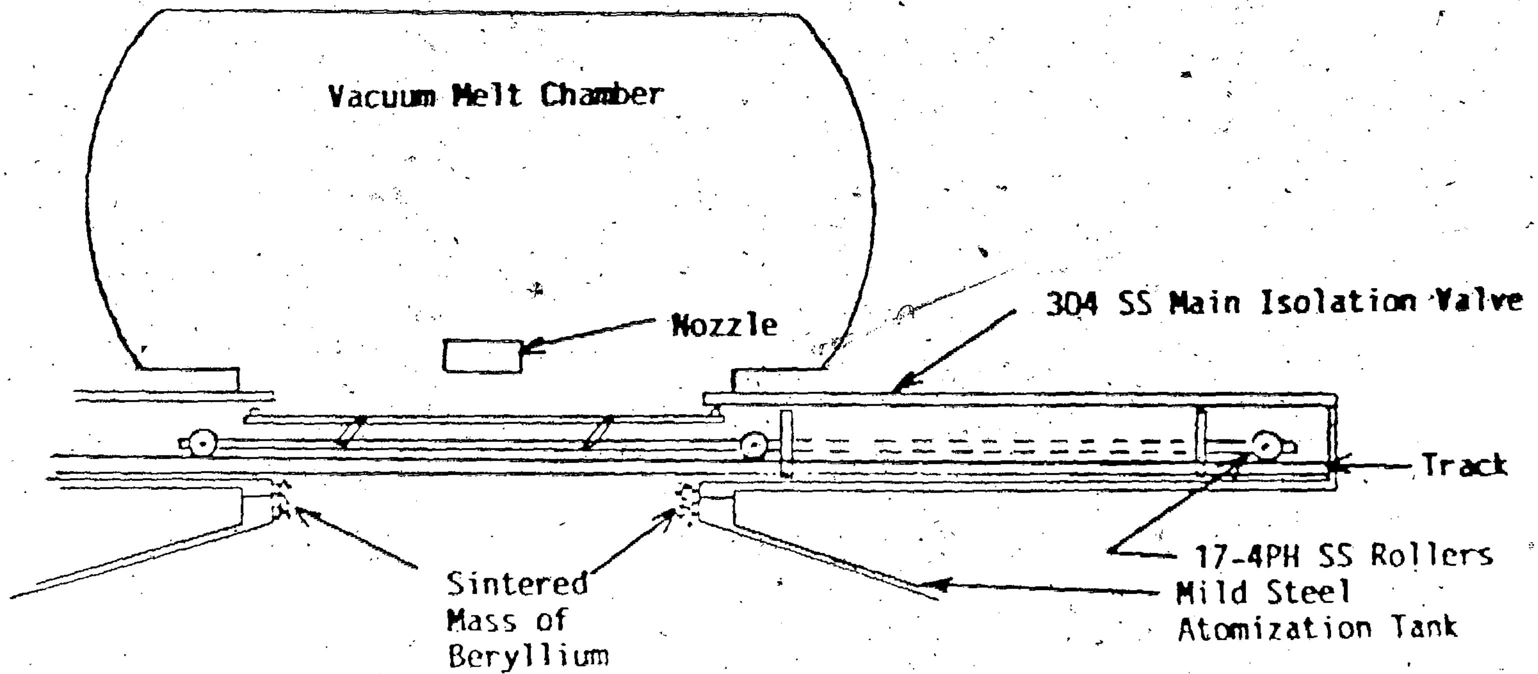
ATOMIZED POWDER CHARACTERISTICS

- Spherical Particles
- Generally Polycrystalline
- Grain Size Stable After 1000 C Heat Treatment
- Approximately 15-25% -200 mesh
- 64% Packing Density
- Fe Contamination Due to Position of Nozzle



Thermal Stability of Atomized Powder

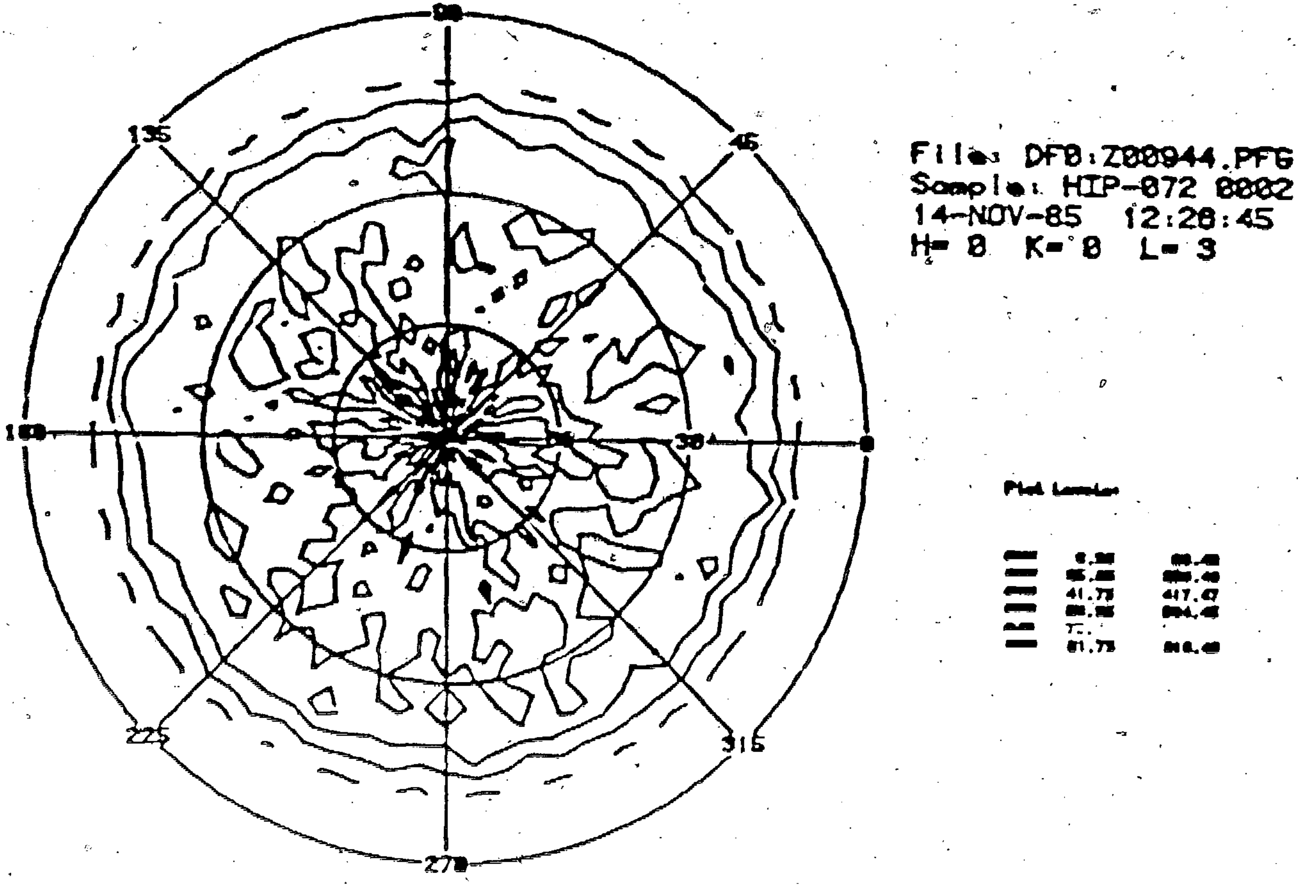

BE ATOMIZED MAG-PRTCL #1
SPECTRUM LABEL
COUNTS
0
1000
2000
3000
4000
0.0
2.0
4.0
6.0
ENERGY (KEV)
Si
Cr
Cr
Fe
Fe
Ni
Ni



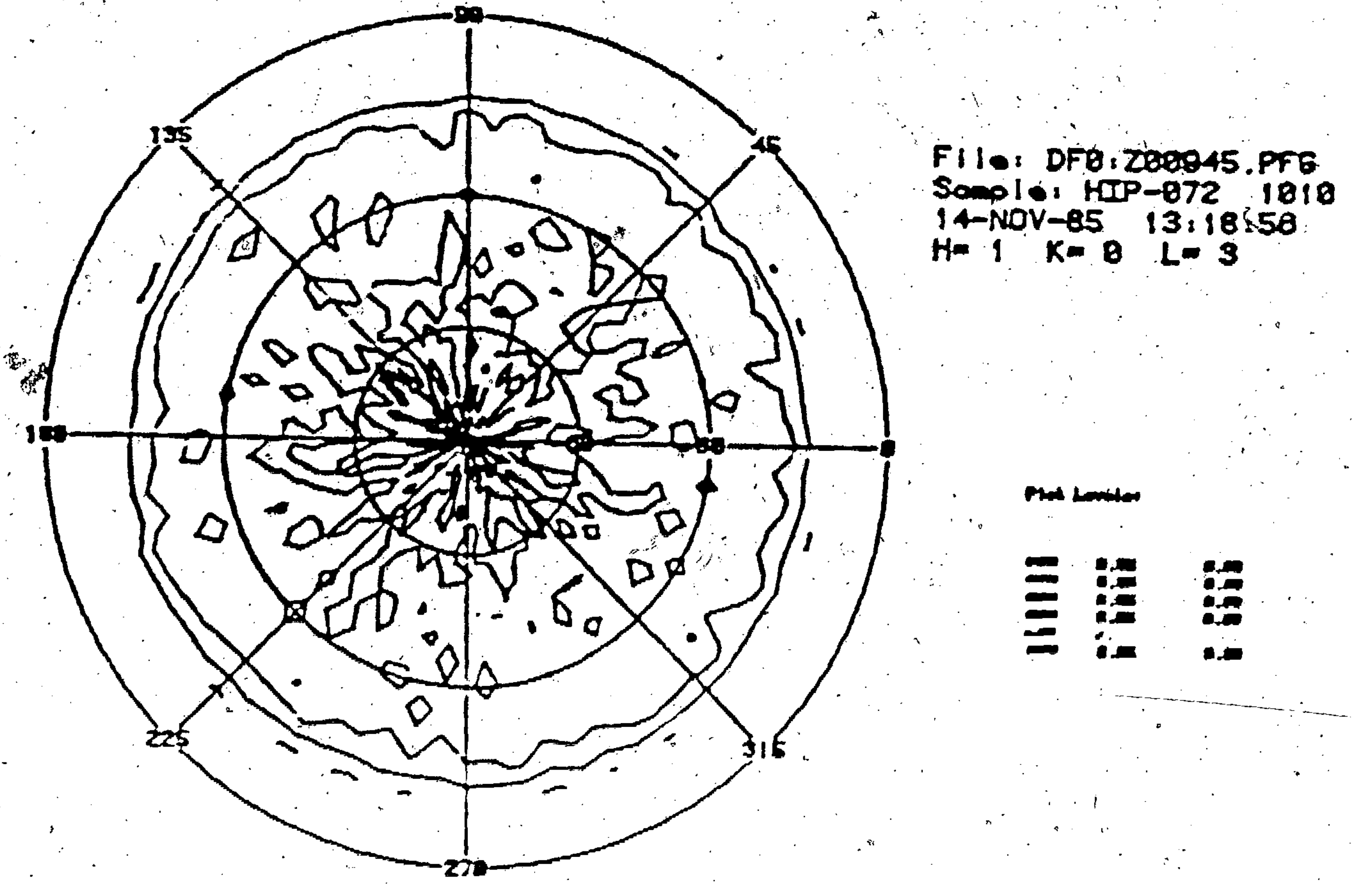
Location of Sintered Be Mass and Schematic of the Main Isolation Valve

SPHERICAL POWDER BILLET CHARACTERISTICS

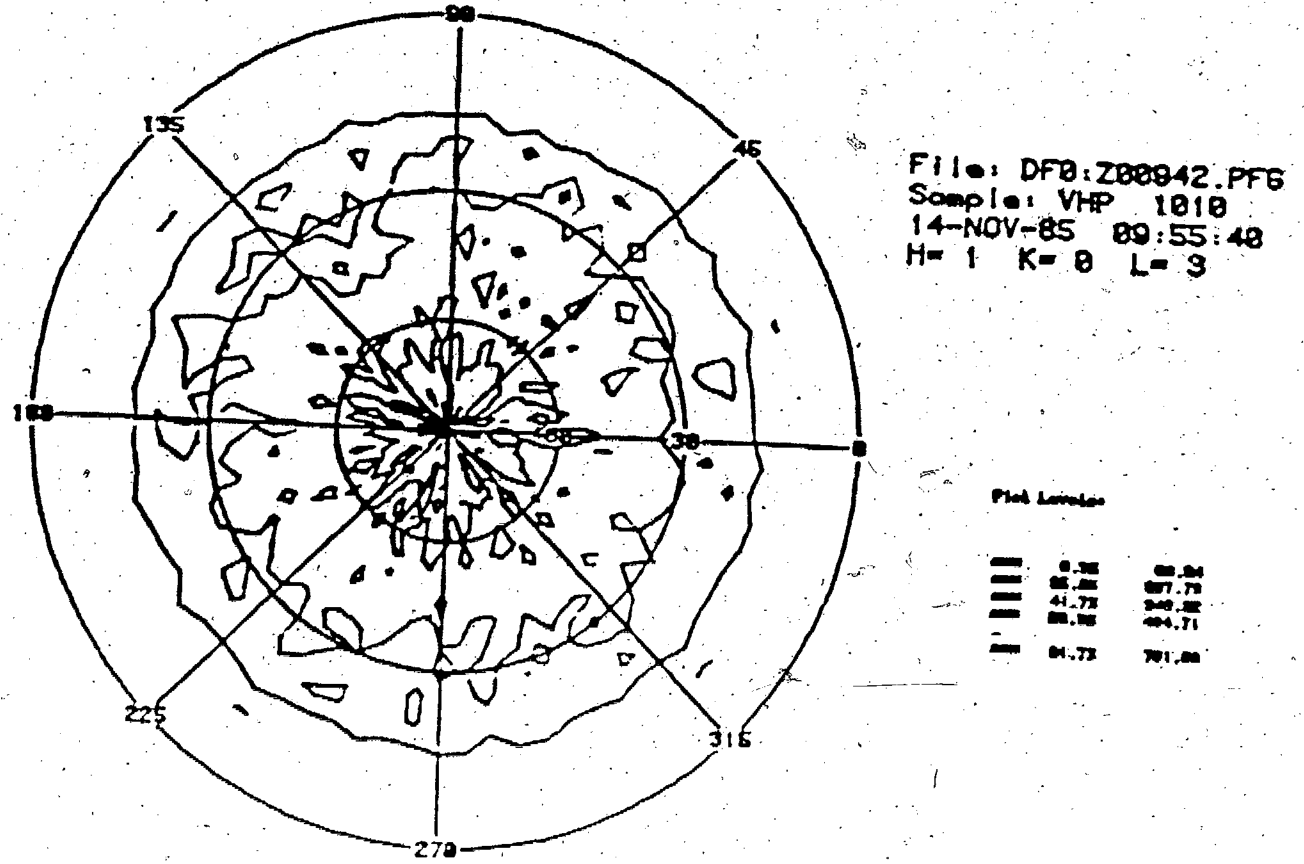
- Hot Isostatic Pressing Showed Isotropic Contraction
- X-Ray Pole Figures Demonstrated a Random Crystallographic Structure
- Mechanical Properties Consistent with Grain Size and Iron Content



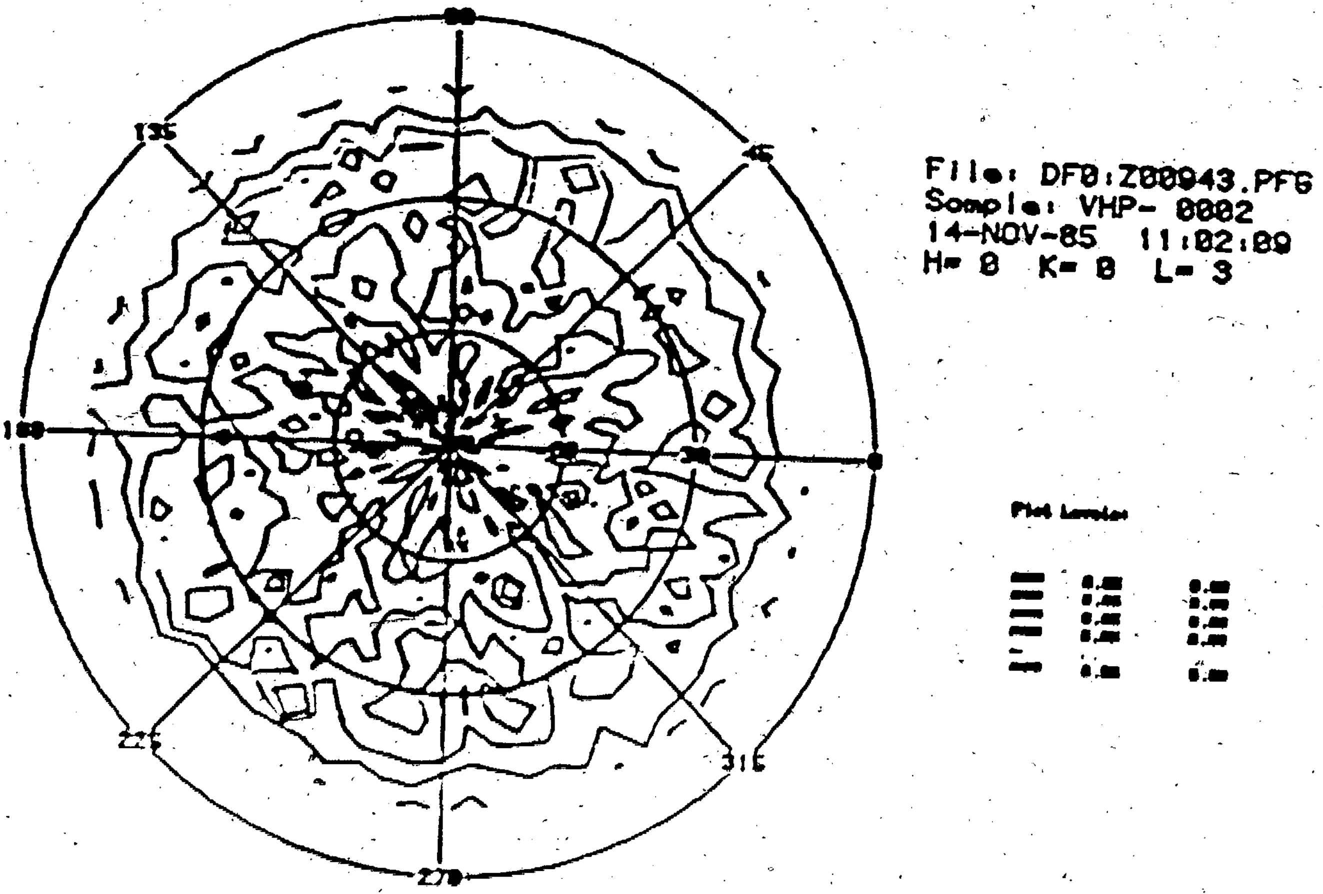
HIP #072 X-ray Pole Figure (0002)



HIP #072 X-ray Pole Figure (10$\bar{1}$0)



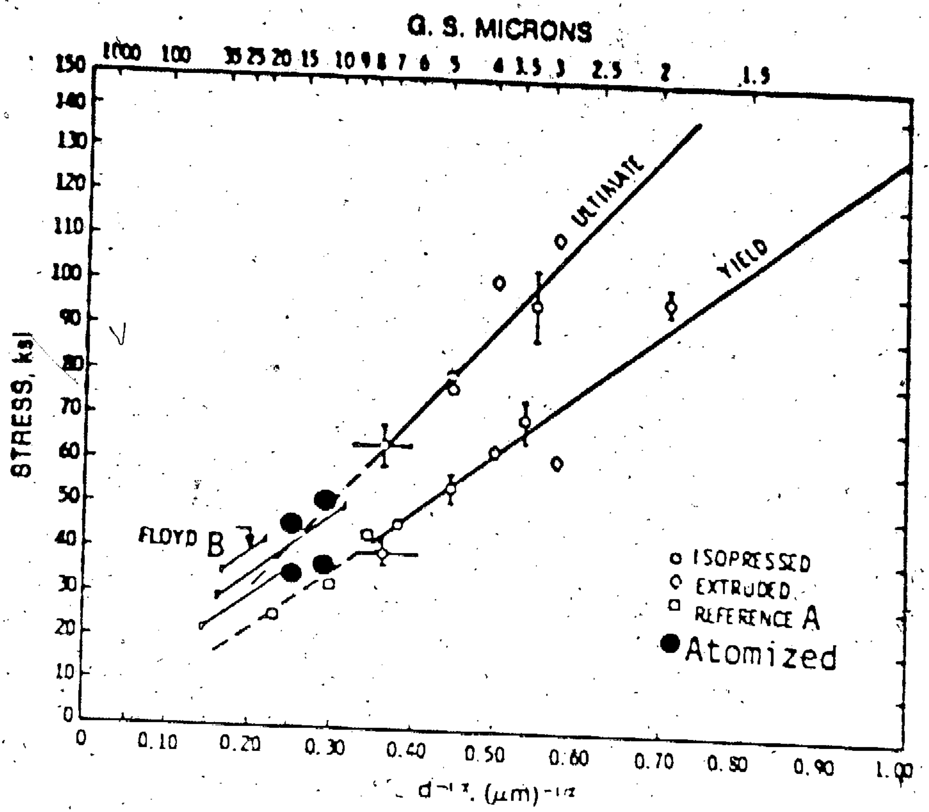
VHP 2172 X-ray Pole Figure - (10$\bar{1}$0)



V P 2172 X-ray Pole Figure - (0002)

HIP BILLET =067 TENSILE PROPERTIES

Orientation	UTS (ksi)	0.2% YS (ksi)	Elongation (%)
Longitudinal	56.9	49.9	2.3
	58.7	48.1	2.0
	58.8	49.4	2.2
Transverse	57.2	46.3	2.6
	58.9	48.4	2.6
	59.1	48.6	2.2



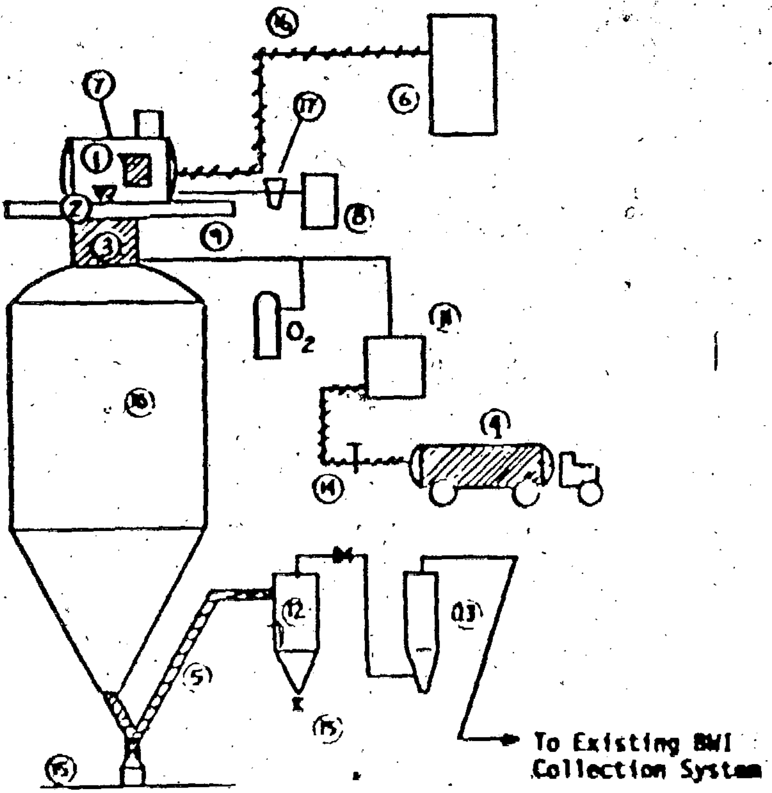
Strength vs Grain Size of HIP Consolidated Atomized Powder. The plot shows data from HIP billets #072 and #076

ATOMIZER UPGRADE

- 12lb. Capacity
- Vacuum OR Inert Gas Melting
- Atomize into 0.5% Oxygen Atomosphere
- Relocate Main Isolation Valve to Prevent Particle Impingement (Fe Contamination)
- Electrically Preheat Nozzle to 1400 C
- 400 psi Atomization Pressure
- 800 C Gas Preheat Temperature

New Components

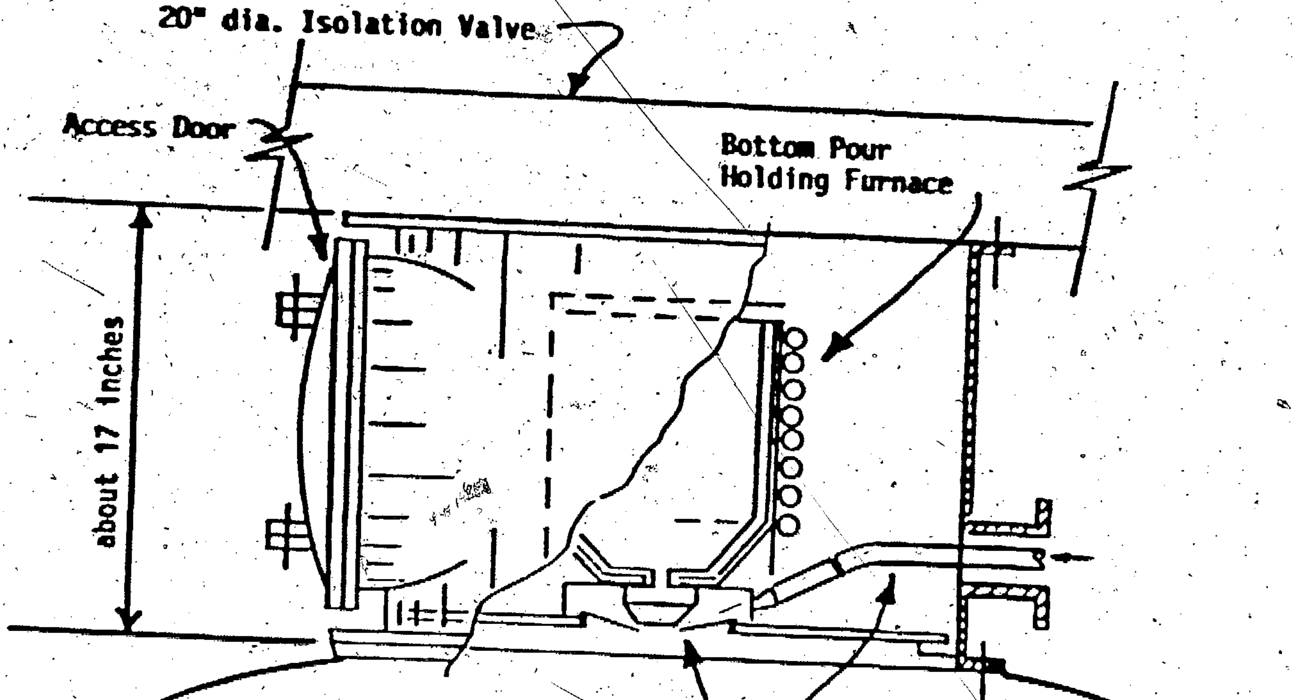
1. Furnace and Coil
   12 lb. Be Capacity
2. Pouring Funnel
   Magnesia Lined
3. Spacer Section with
   Holding Fce., Graphite
   Susceptor, MgO Crucible
   and Atomizing Nozzle
4. Mobile High Pressure
   Gas Bulk Supply
5. Powder Transfer
   Pipe
16. Elect. Bus from
    Exist. 150 KW Supply
    to Fce.
17. Vacuum System
    Cold Trap



Existing Components

6. 150 KW 1 KC Power
   Supply
7. Vacuum Melt Chamber
8. Vacuum Pump
9. 20" Isolation Valve
   (Relocated)
10. Atomization Tank
11. Gas Preheater
    (Modified)
12. Cyclone Separator
13. Primary Filter
14. Grove Pressure
    Regulator
15. Powder Collection Can

GAS ATOMIZER SCHEMATIC



ATOMIZING SPACER
CONCEPTUAL DESIGN

BERYLLIUM ELECTROREFINING PROJECT

D. L. Mitchell, RFP

Abstract

The beryllium scrap recycle project was established to provide technical and economic information to the Beryllium Supply Program. Beryllium Chloride Synthesis on a manufacturing scale has produced the salt required for electrorefining cell operation. The report describes the equipment and operating experience for the $BeCl_2$ production. In addition, design changes are reported that have recently been incorporated into the Beryllium Electrofining Cell. These changes were necessary to enhance safety and to assure smooth operation of all supporting systems.

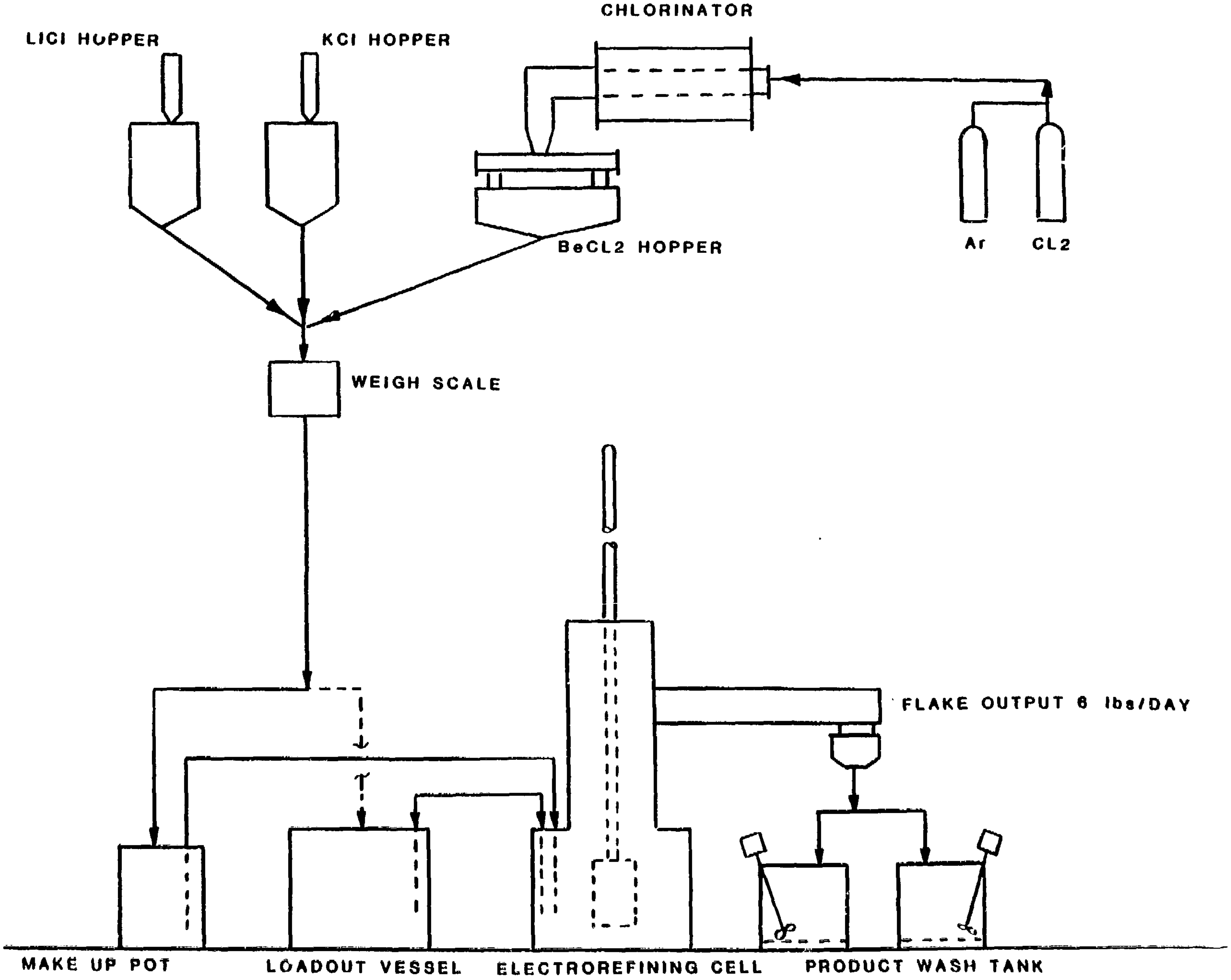
OBJECTIVE

- ESTABLISH TECHNOLOGY
- SUPPORT DOE STUDY
  - ECONOMIC DATA
  - TECHNICAL DATA

APPROACH

- LABORATORY CELL
- TECHNOLOGY PURCHASE
- DEMONSTRATION PROCESS

BERYLLIUM ELECTROREFINING PROCESS



DEMONSTRATION ACTIVITIES TO OCT. 1987

- INSTALLATION
- SUB-SYSTEM OPERATIONAL TESTING
- REPAIRS AND MODIFICATIONS TO SEVERAL MAJOR SYSTEMS
- THREE DIFFERENT CONDENSER DESIGNS
- PRODUCTION OF 350 POUNDS OF $BeCl_2$
- SUCCESSFUL SALT FUSION AND TRANSFER

PRODUCTION OF $BeCl_2$

$2Be\ (metal) + 2Cl_2 = Be_2\ Cl_4\ (g)$ 650°C

$1/2\ Be_2Cl_4\ (g) = BeCl_2\ (s)$

$BeCl_2$ IS EXTREMELY DIFFICULT TO PREPARE AND HANDLE

- Reaction Occurs at 650°C
- Condensation Begins at 415°C
- Four different crystalline forms can be produced between 250°C and 415°C
- It is toxic
- It is hydroscopic and deliquescent

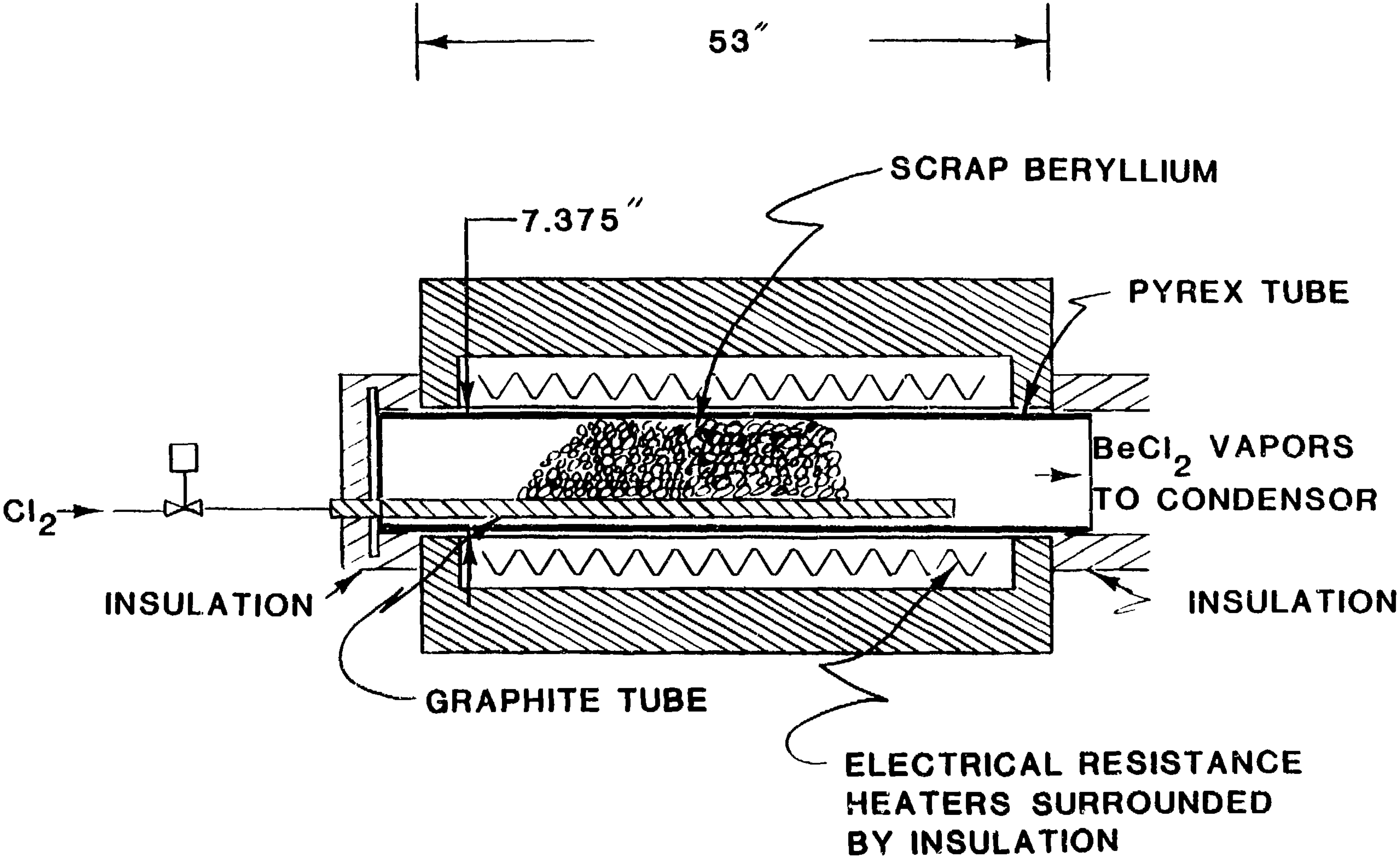
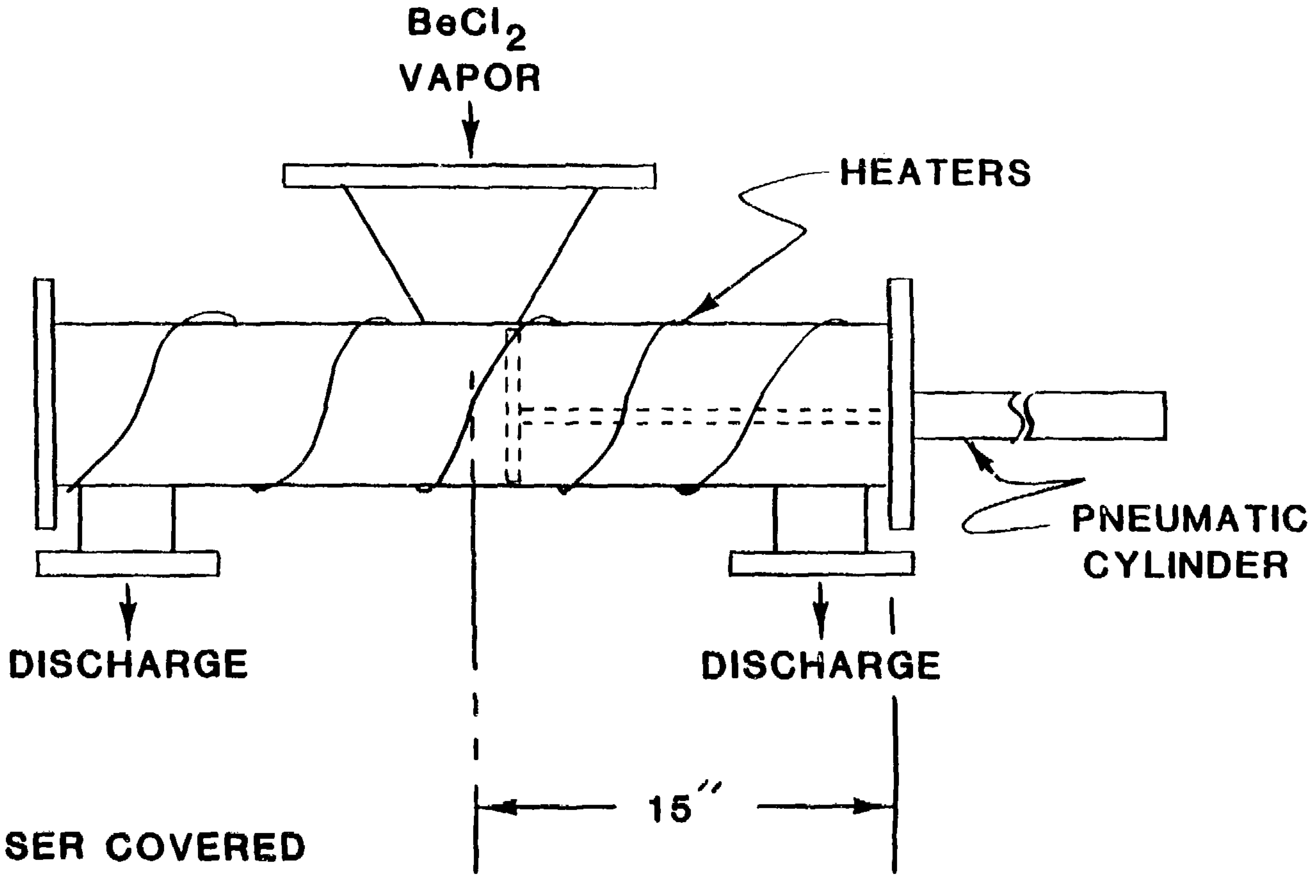

53"
SCRAP BERYLLIUM
7.375"
PYREX TUBE
$BeCl_2$ VAPORS TO CONDENSOR
Cl_2
INSULATION
INSULATION
GRAPHITE TUBE
ELECTRICAL RESISTANCE HEATERS SURROUNDED BY INSULATION

Fig 1

CONDENSER
MODEL 1



CONDENSER COVERED
WITH INSULATION

FIG 2

MODEL #1

PROBLEMS

- PRODUCT WAS FINE POWDER
- PRODUCTION RATE LESS THAN .1 LB/HR
- CONDENSATION OCCURRED IN TRANSITION PIECE

CONDENSER
MODEL 2

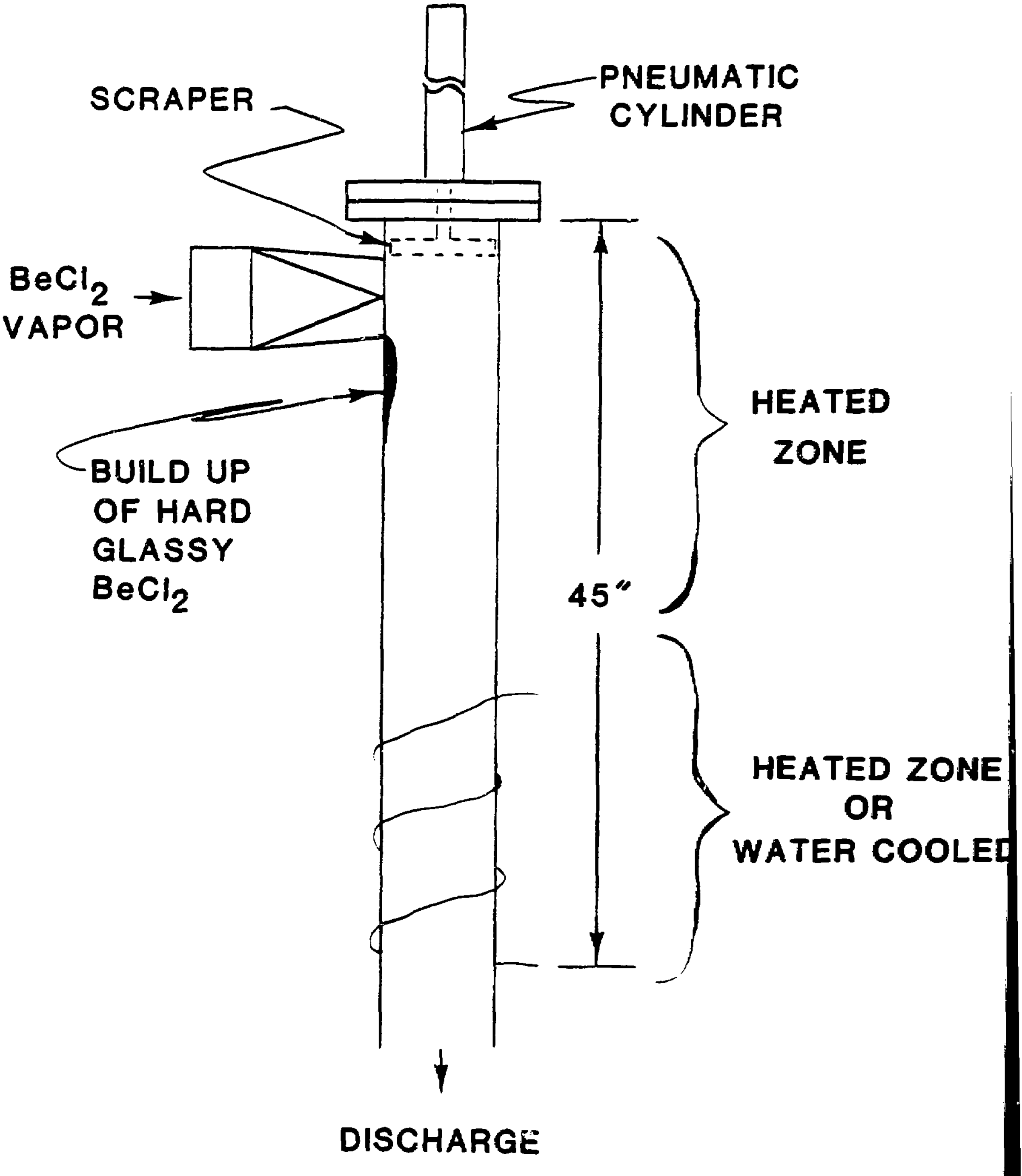


FIG 3

MODEL #2

PROBLEMS

- PRODUCT WAS FINE POWDER
- PRODUCTION RATE LESS THAN .3 LB/HR
- STALLED PNEUMATIC SCRAPER

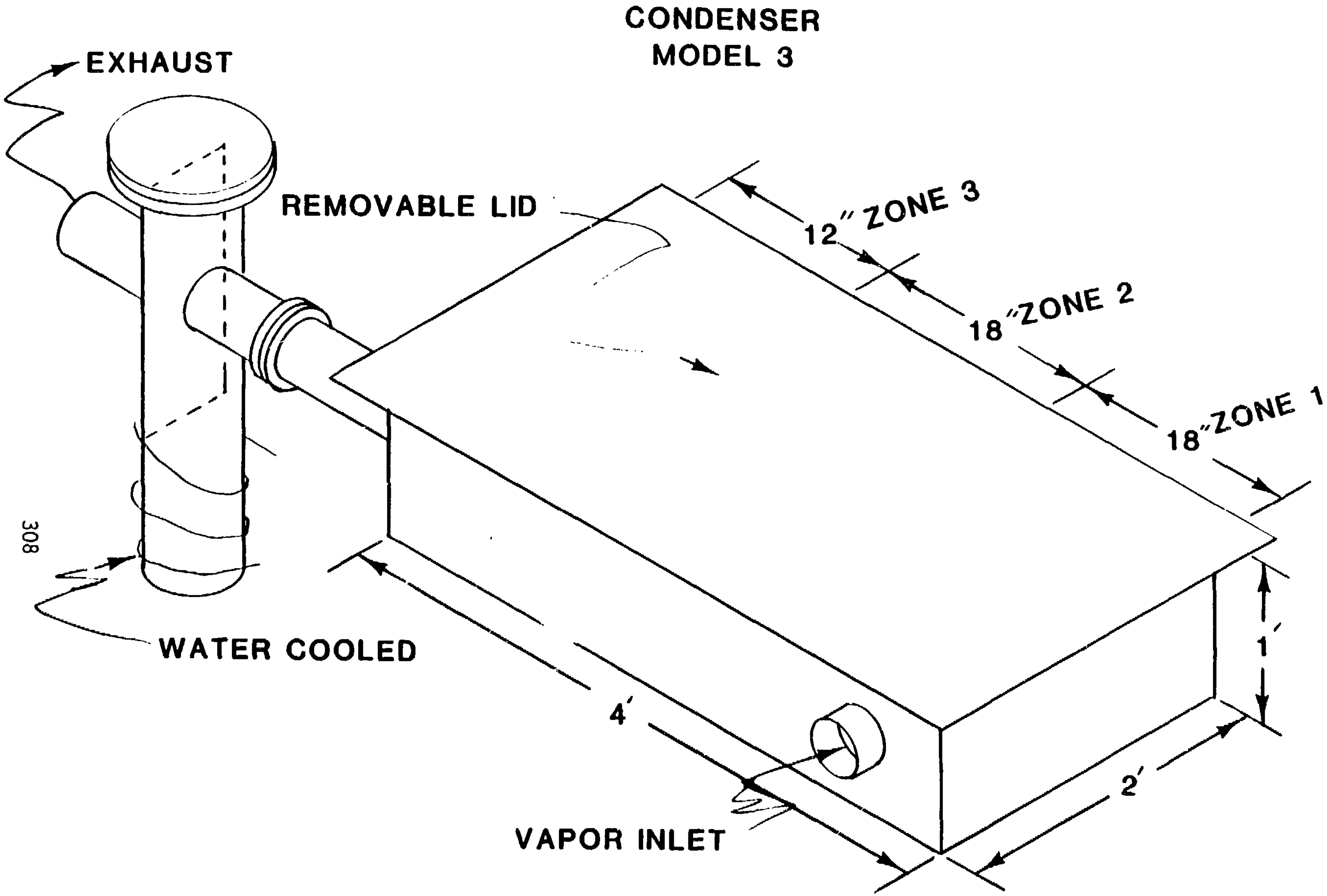

CONDENSER
MODEL 3
EXHAUST
REMOVABLE LID
12″ ZONE 3
18″ZONE 2
18″ZONE 1
WATER COOLED
1′
4′
2′
VAPOR INLET

FIG 4

MODEL #3

- PRODUCED 2 LB/HR
- DESIRED CRYSTALLINE FORM

PROBLEMS

- MELTED CRYSTALS FORMED HARD, FIBROUS MATERIAL THAT ADHERED TO BOTTOM AND SIDE
- EXTREME DIFFICULTY IN REMOVING

RECOMMENDATIONS FOR FUTURE OPERATIONS

- Condensor must:
    - Provide adequate residence time
    - Have good temperature control
    - Be contained in glovebox
- Batch Process Preferable
    - Smaller Dimensions Would Simplify - Trade off is Length of Operation
    - Examine Materials of Construction

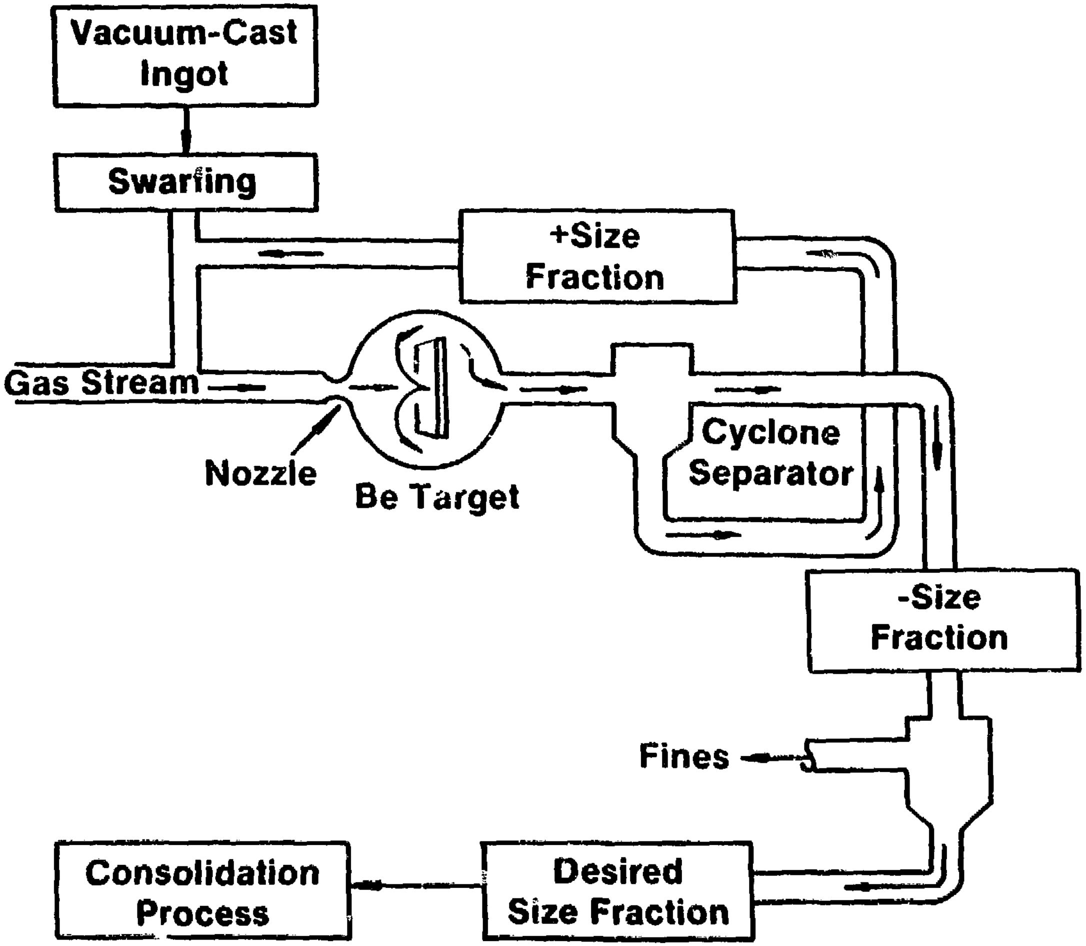
ADVANCES IN BERYLLIUM POWDER TECHNOLOGY

S. P. Abeln, RFP

Abstract

Four powder production technologies are compared and contrasted. Comparisons include the production sequence and the final powder characteristics. The technologies reviewed are: 1) impact grinding, 2) laboratory scale fluid energy milling (Micron Master), 3) gas atomization, and 4) atomization by the rotating electrode process. Powder characteristics evaluated for each process include, morphology, size distribution, chemistry, and outgassing characteristics. It is shown that new atomization processes are more efficient and possess certain powder characteristics that are advantageous to Near Net Shape technologies.

- Powder types
  - Impact ground
  - Fluid energy milled
  - Gas atomized
  - REP atomized
- Characterization
  - Morphology
  - Microstructure
  - Outgas
- Summary



Vacuum-Cast Ingot

Swarfing

+Size Fraction

Gas Stream

Nozzle

Be Target

Cyclone Separator

-Size Fraction

Fines

Desired Size Fraction

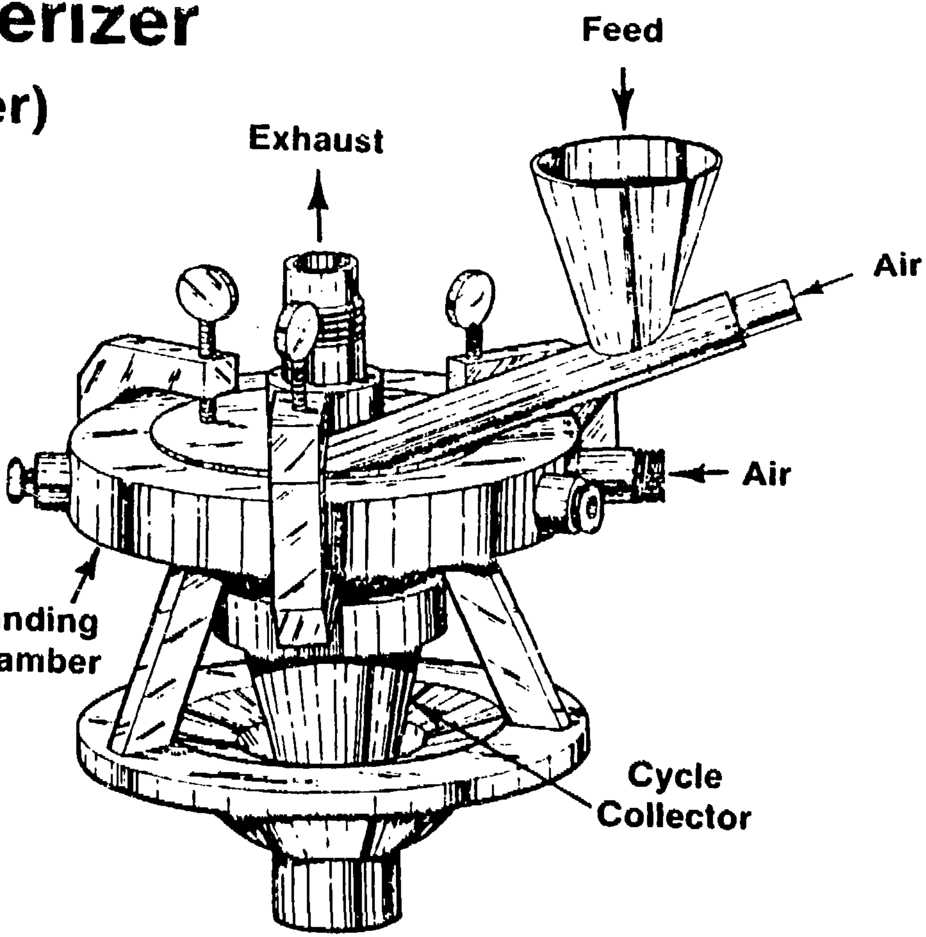
Consolidation Process

Impact Attritioning

Rockwell International

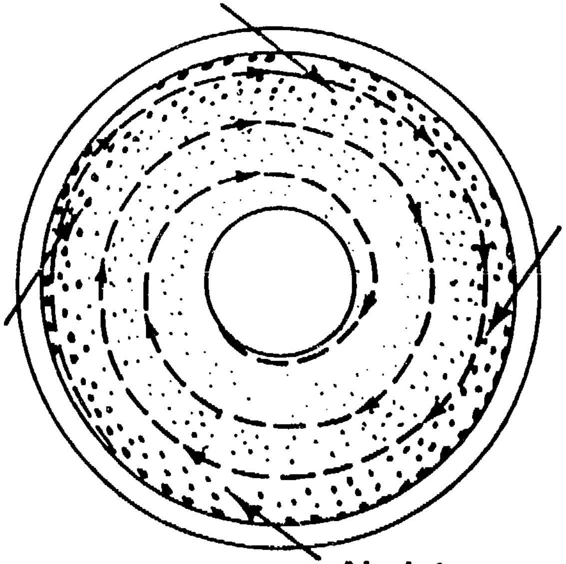
Angle Jet Pulverizer

(Micron-Master)

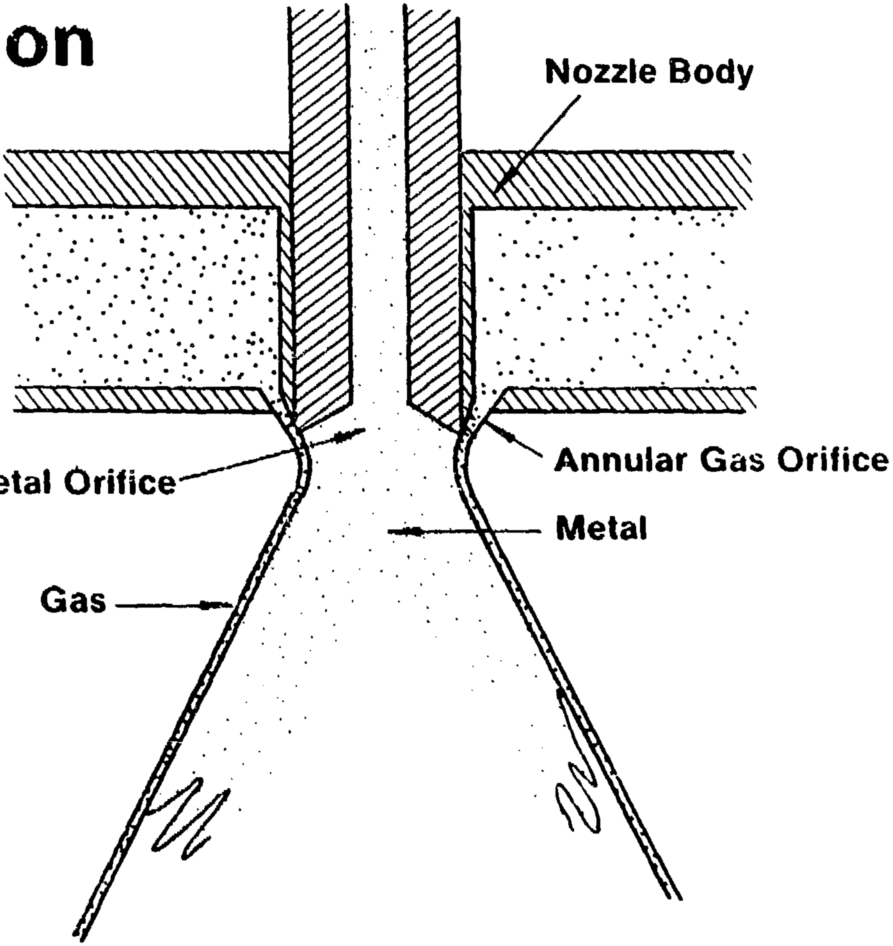


Mill Size: 2" Diameter Grinding Chamber

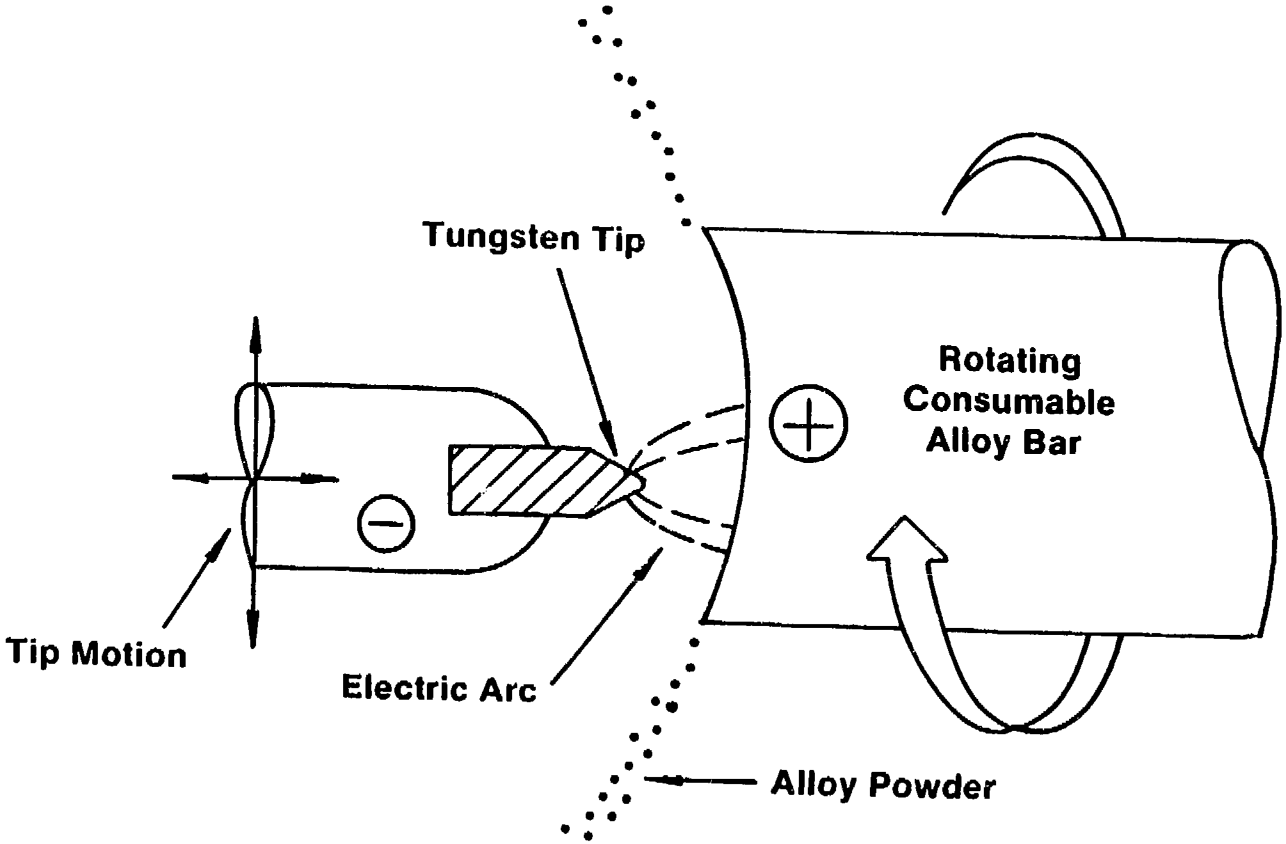
Grinding Chamber Action in Angle Jet Pulverizer



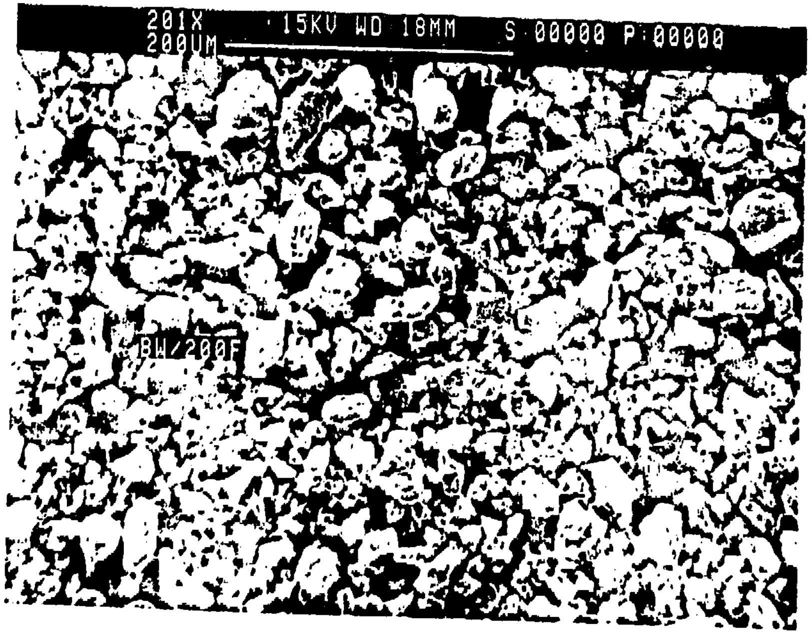
Gas Atomization



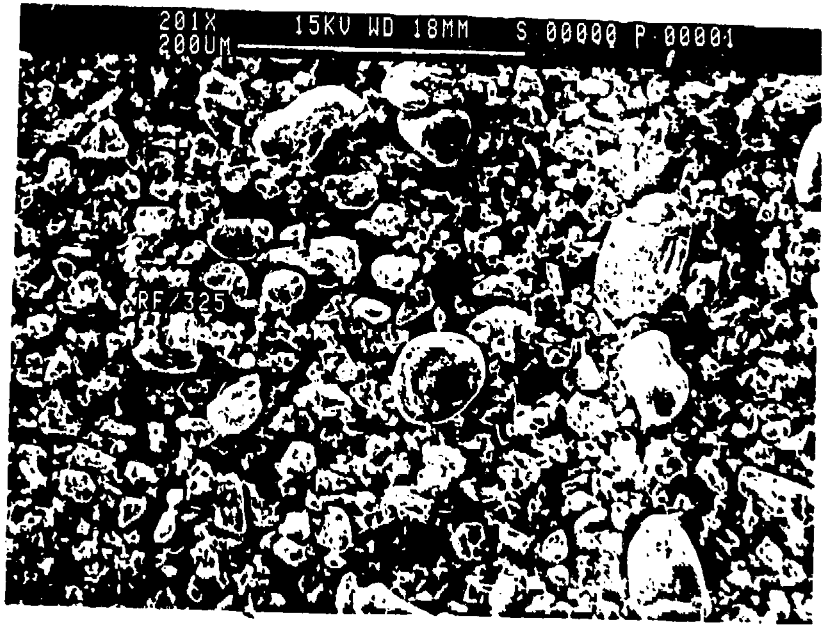
Rotating Electrode Process



Attritioned Powder

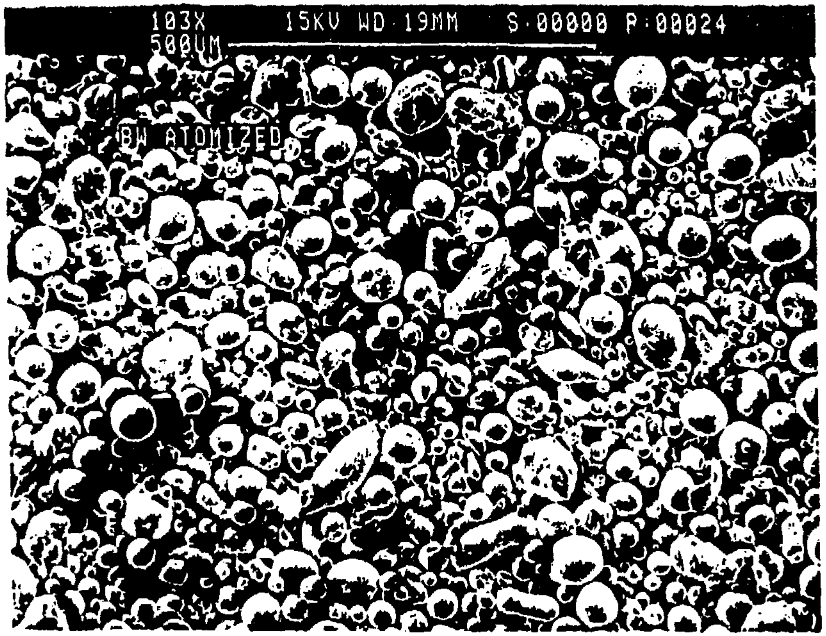


Impact Ground

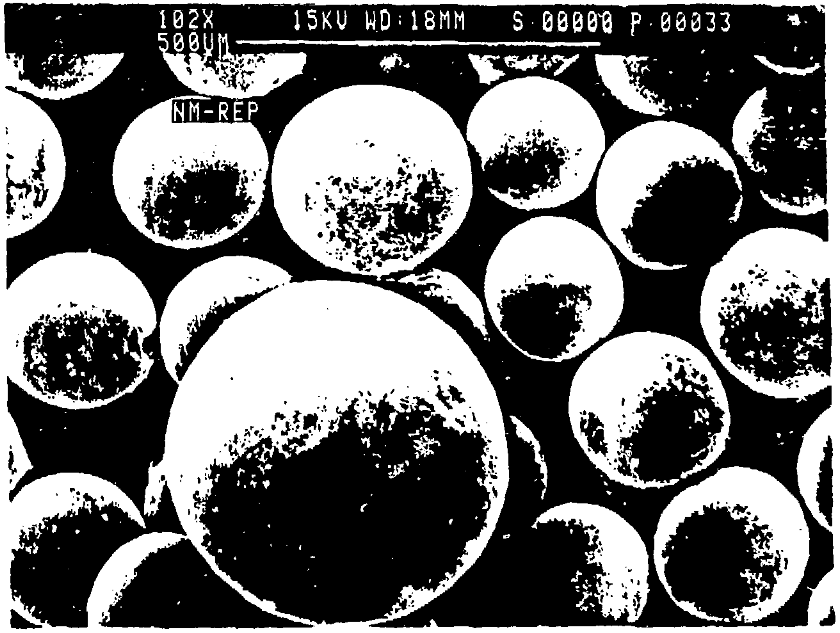


Fluid Energy Milled

Atomized Powder

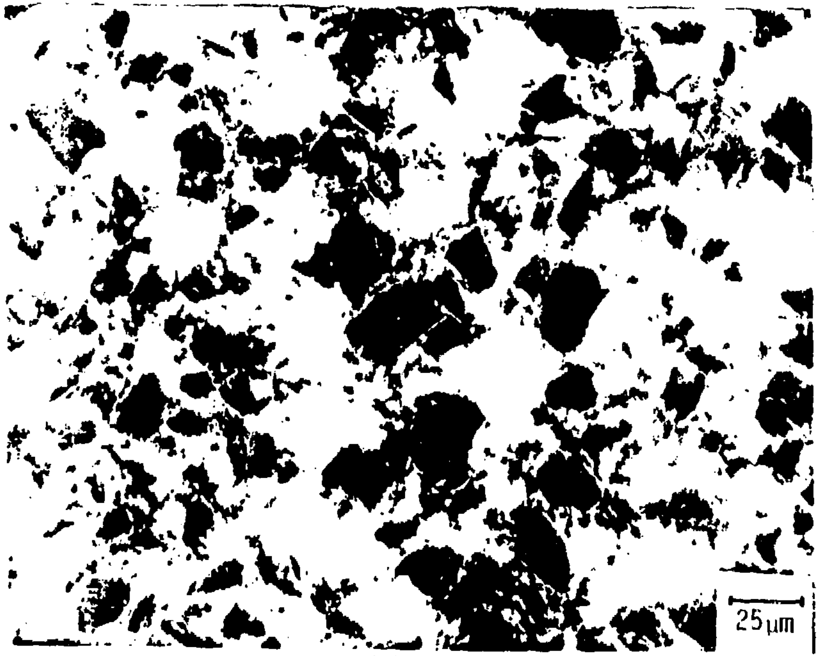


Gas Atomized



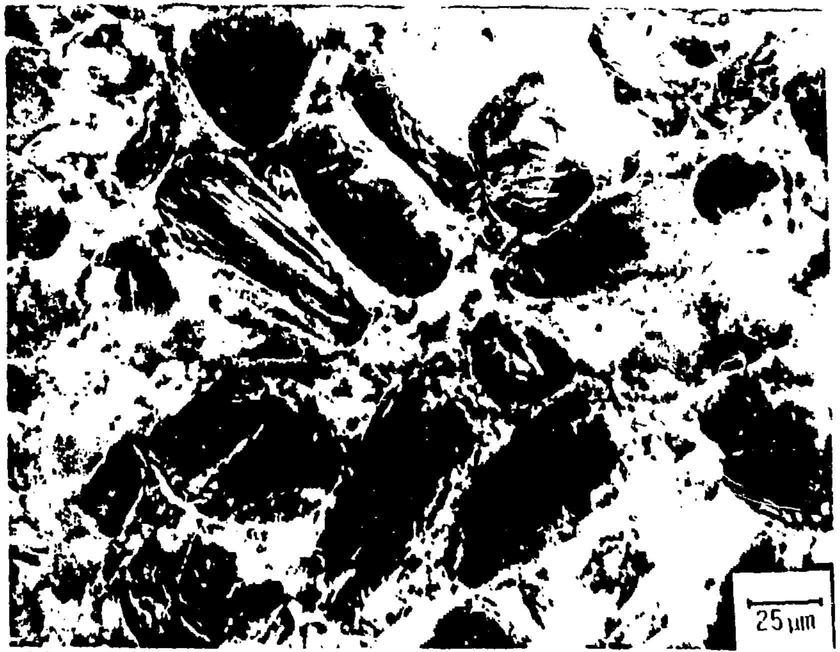
Rotating Electrode Process

Attritioned Powder Microstructure



400x 200F WO-0514 P.L.

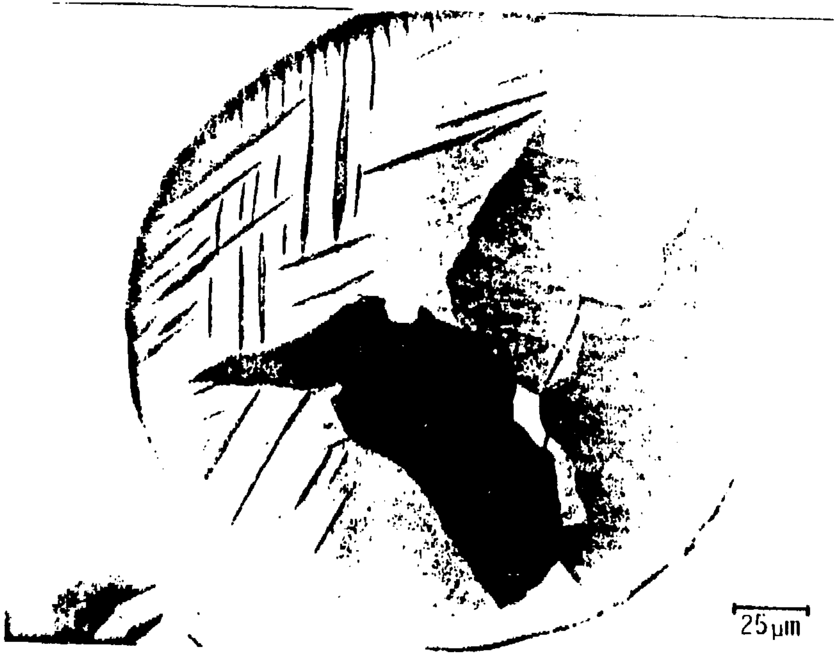
Fluid Energy Milled



400x RF 325 WO-0514 P.L.

Impact Ground

Atomized Powder Structures



400x

NM REP

WO-0514
P.L.

Rotating Electron Process

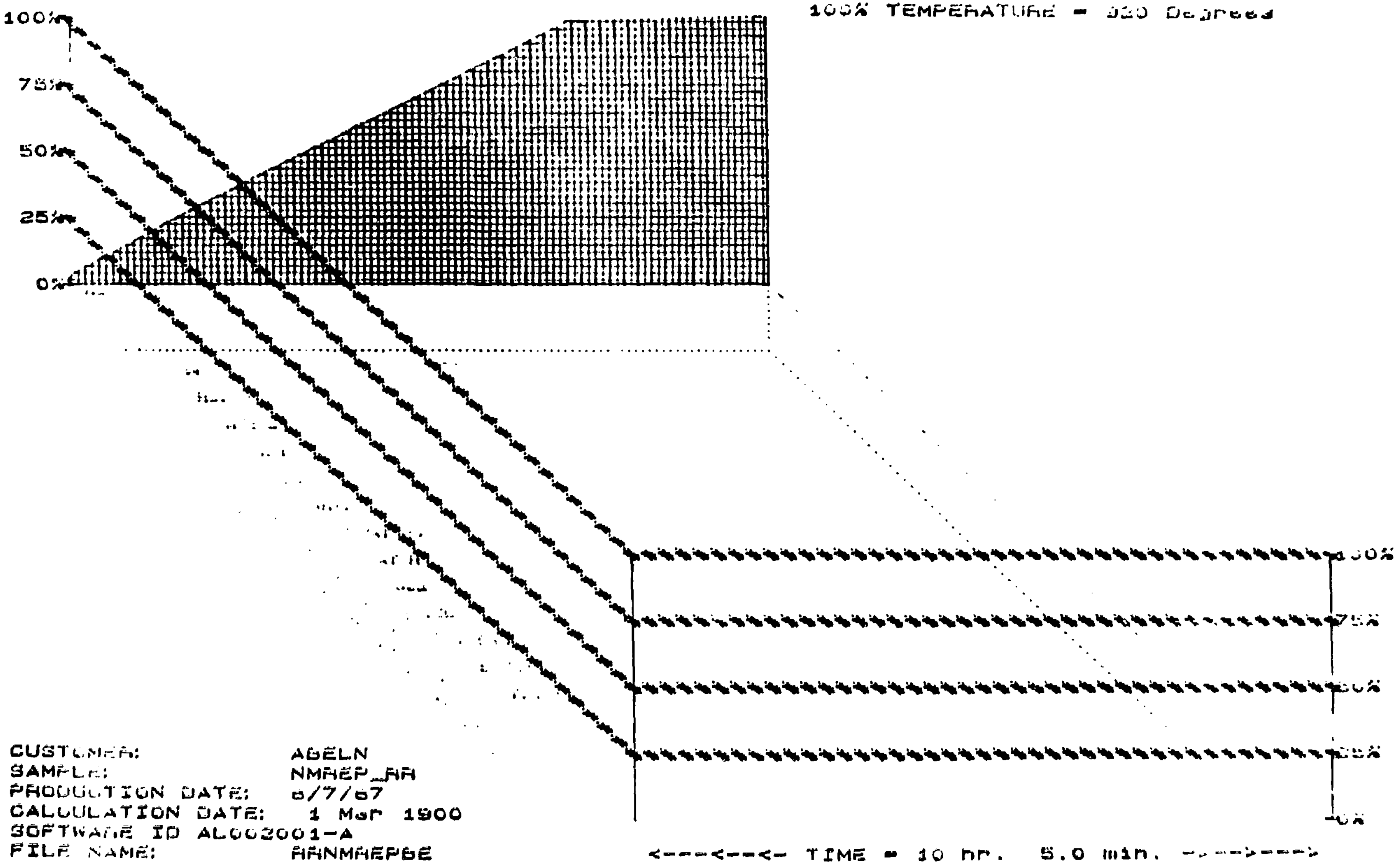
400x

BW
Atomized

WO-0514
P.L.

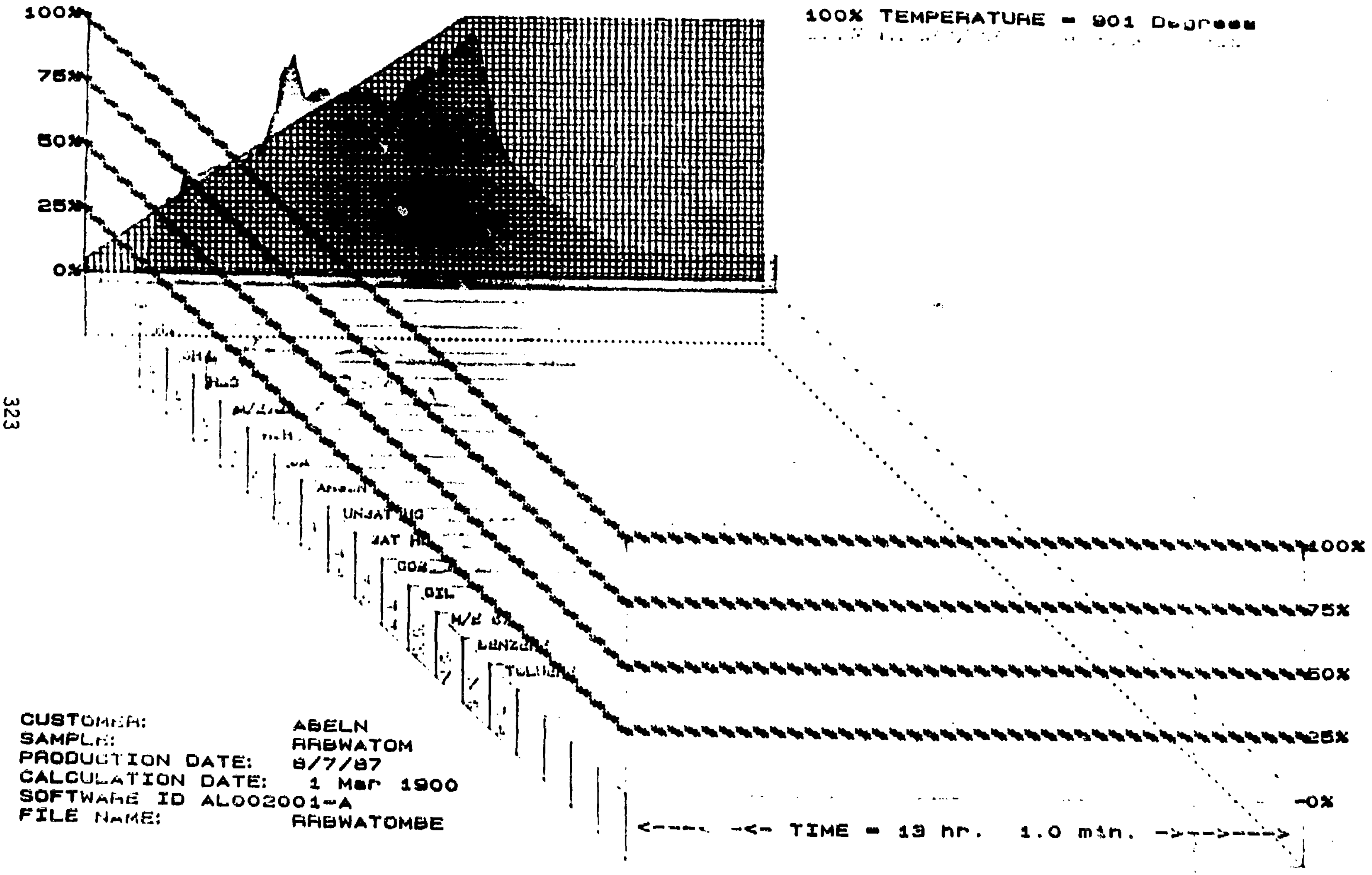
Gas Atomized

Time vs Moles,Sec for all Cases



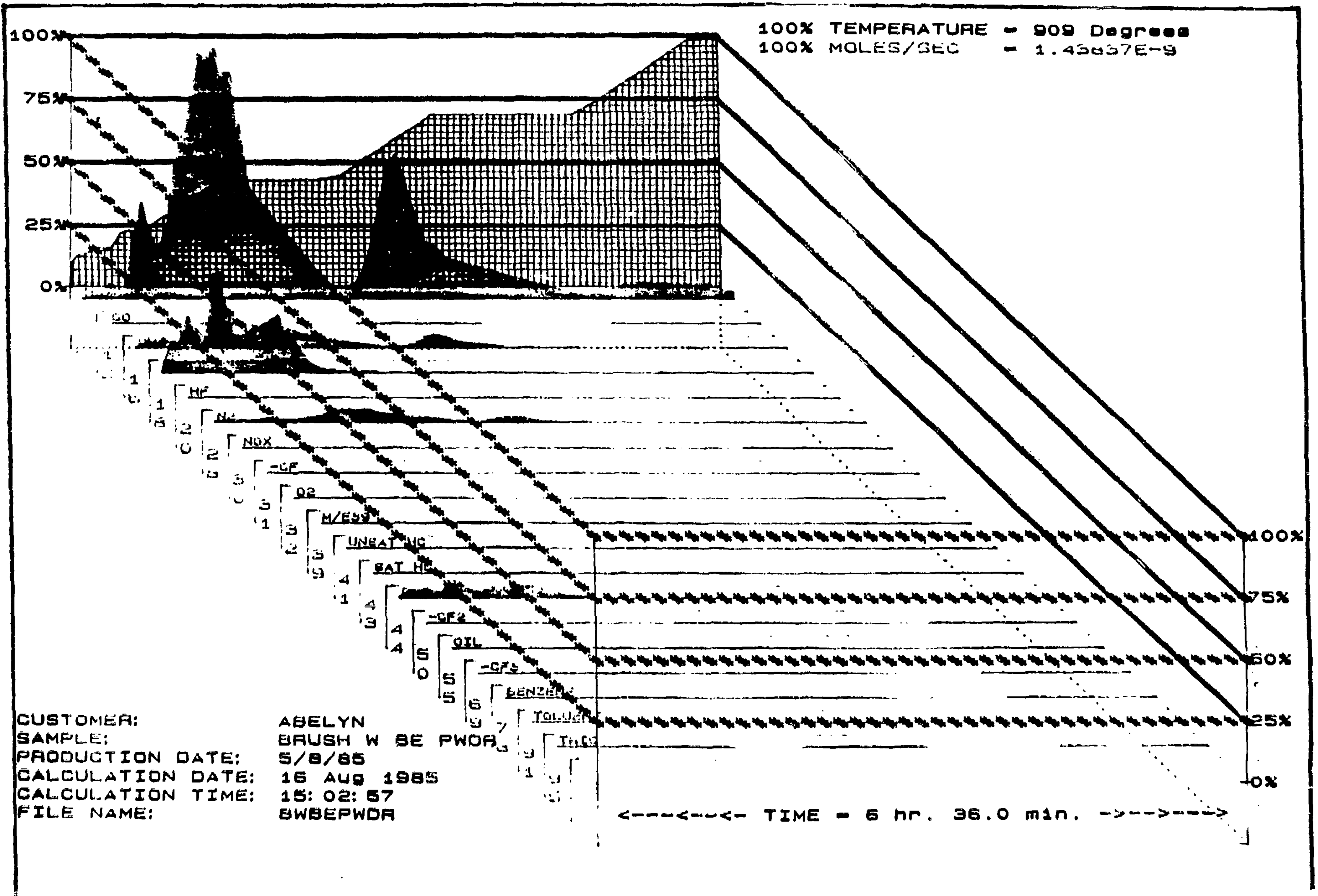
CUSTOMER: ABELN
SAMPLE: NMREP_RR
PRODUCTION DATE: 6/7/67
CALCULATION DATE: 1 Mar 1900
SOFTWARE ID AL002001-A
FILE NAME: RRNMREPBE

Time vs Moles/Sec for all Gases



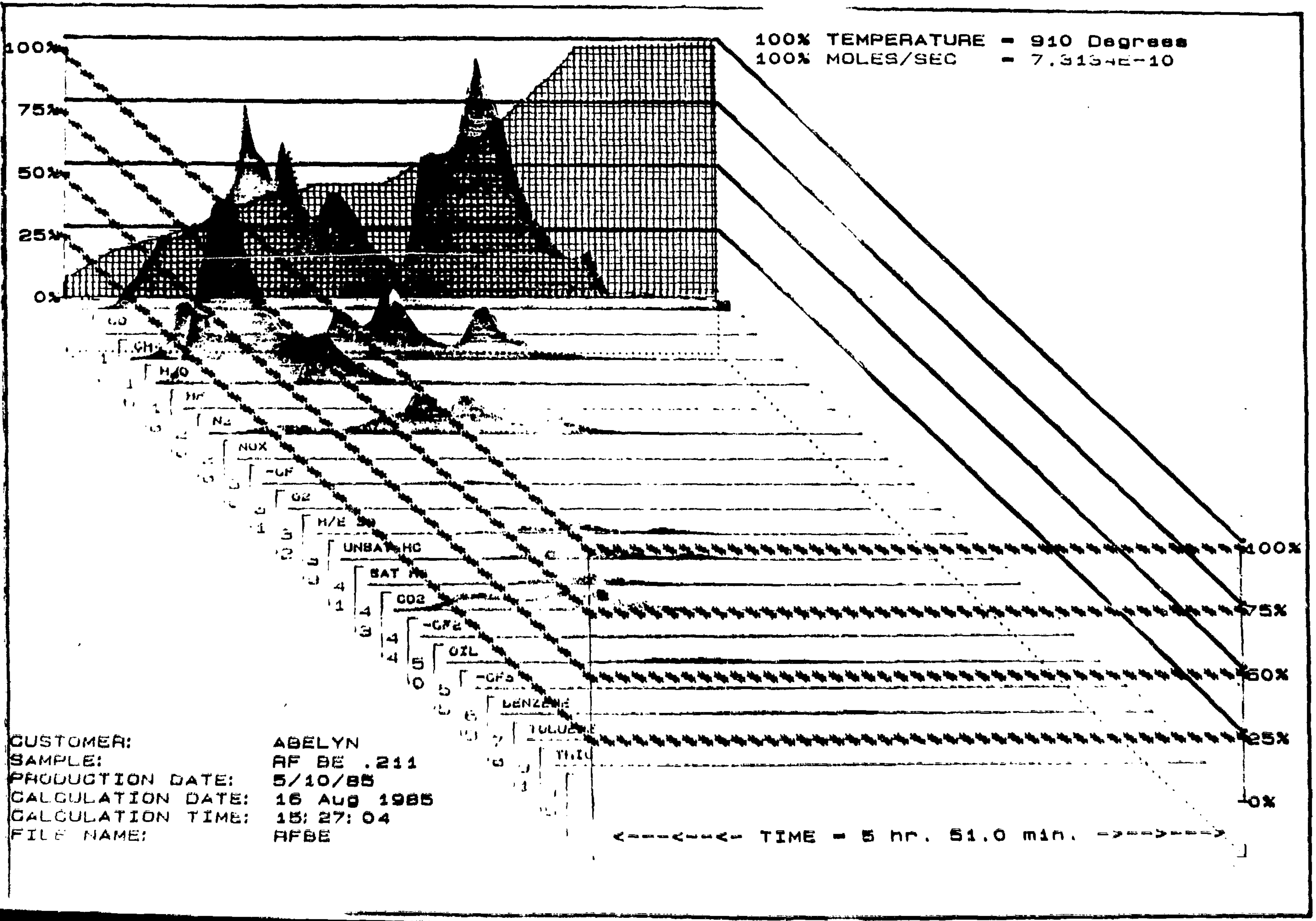
CUSTOMER: ABELN
SAMPLE: RRBWATOM
PRODUCTION DATE: 8/7/87
CALCULATION DATE: 1 Mar 1900
SOFTWARE ID AL002001-A
FILE NAME: RRBWATOMBE

Time vs Moles/Sec for all Gases



CUSTOMER: ABELYN
SAMPLE: BRUSH W BE PWDR
PRODUCTION DATE: 5/8/85
CALCULATION DATE: 16 Aug 1985
CALCULATION TIME: 15: 02: 57
FILE NAME: BWBEPWDR

Time vs Moles/Sec for all Gases



CUSTOMER: ABELYN
SAMPLE: AF BE .211
PRODUCTION DATE: 5/10/85
CALCULATION DATE: 16 Aug 1985
CALCULATION TIME: 15: 27: 04
FILE NAME: AFBE

Summary

- Attrition processes established and show no avenues for further improvement
- Atomization processes more efficient
- Atomized powder characteristics infer manufacturing advantages
  - Lower surface area; lower BeO
    - Reduced tool wear in machining
    - Improved properties
    - Possible SPF
  - Spherical particles
    - Higher packing densities
    - Automation and easy flow
  - Rapid solidification processing
    - Extended solid solubility (alloying)
- No apparent outgassing differences as shown by dynamic mass spectroscopy

Phase Morphology vs. Cracking Potential in Electron Beam Welded Be-Al Alloys*

K. W. Mahin, Sandia National Laboratories, Livermore, Ca
L. Tanner, Lawrence Livermore National Laboratory, Livermore, Ca

Over the years there has been an ongoing interest in eliminating cracking in electron beam welds of beryllium by the addition of aluminum. The typical rule of thumb is that at least 30 wt% Al (~10 at%) must be added to the weld to suppress cracking at room temperature. However, there has been only limited work on determining the relationship between the phase morphology of the aluminum-modified beryllium weld and its crack susceptibility. The existing phase characterization work on welds in this material is based on optical metallography and suggests that cracking is suppressed by the formation of an eutectic structure in the weld.

The purpose of this study was to more fully characterize the phase transformations which occur in Be-Al electron beam welds over a range of aluminum compositions and to try and relate morphology to crack susceptibility. Electron beam welds were made on chill cast button ingots of high purity beryllium modified with controlled amounts of aluminum, in order to control the effects of impurities on microstructure and cracking susceptibility,. The castings ranged in composition from 50 at%Be-50 at% Al to 95 at%Be-5 at%Al. For comparison, an electron beam weld of commercial HIP'ed beryllium (S65 grade), onto which a controlled amount of aluminum (~10 - 15 at% Al) had been physically vapor deposited (PVD'd), was also included in the test matrix. The welds were analyzed using optical, scanning electron microscopy and transmission electron microscopy.

Figure 1 [2] shows the typical Be-Al binary eutectic phase diagram modified to show the metastable miscibility gap centered around the 70 at%Be - 30 at% Al composition. Under equilibrium cooling conditions, the structure in the as-solidified material at compositions above 50 at% Al should consist of primary beryllium dendrites with eutectic in the interdendritic regions. With sufficient undercooling, however, we would expect this structure to be replaced by a liquid phase separated structure over a fairly wide range of aluminum additions, depending on the degree of undercooling in the melt.

---

*This work supported by the U. S. Department of Energy, DOE, under contract #DE-AC04-76DP00789.

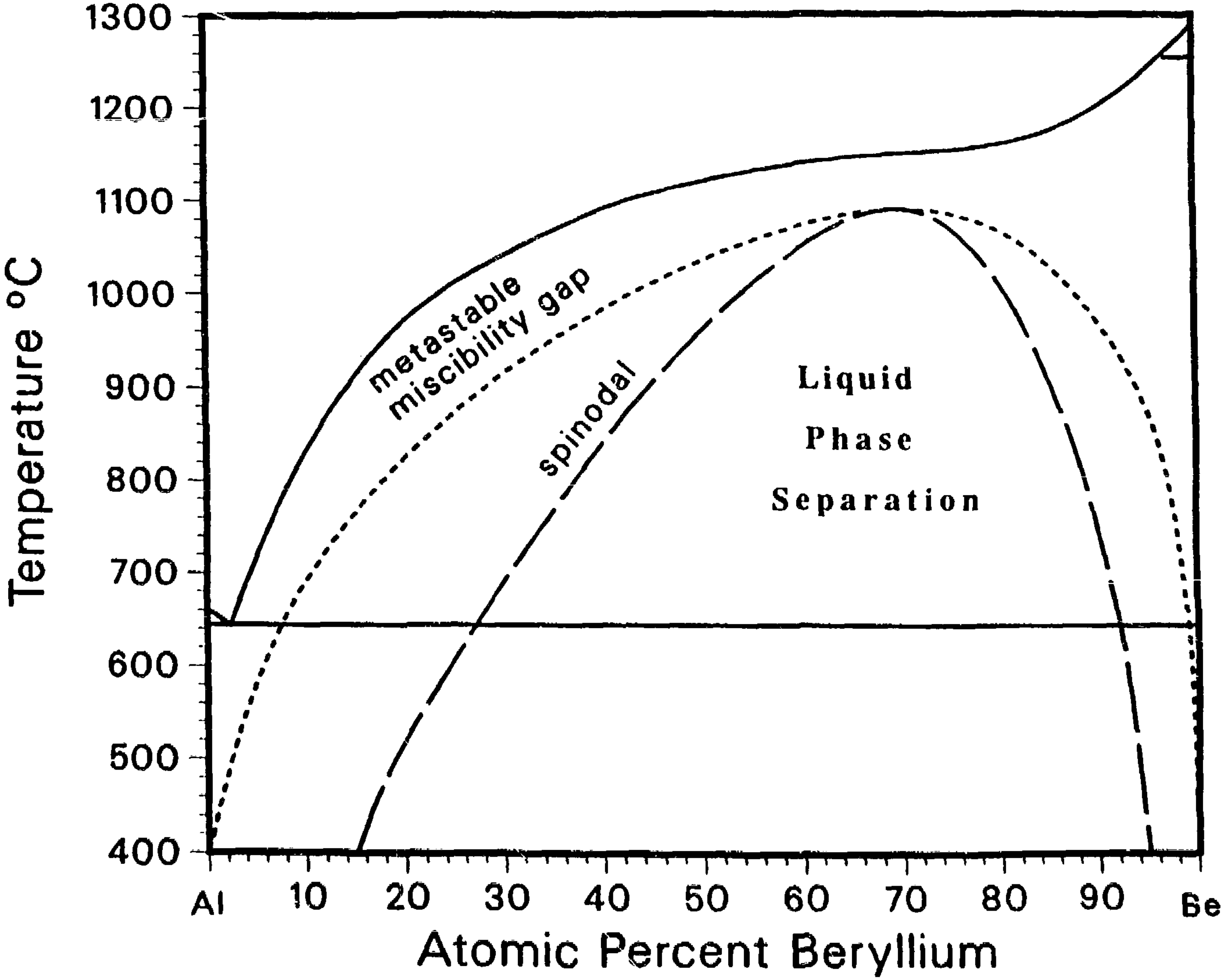
Transmission electron microscopy analysis of the Be-Al welds revealed that a metastable phase starts to appear in alloys with 60 at% Be - 40 at% Al. However, at this composition, the metastable phase forms only in the root of the electron beam weld. A minimum undercooling of 100 °C is required to reach into the spinodal region of the miscibility gap at this composition. At 70 at%Be - 30 at%Al, a fully phase separated structure formed throughout the weld. The structure appears to have an interconnected "swiss-cheese" type of morphology with aluminum grains solidifying around a number of randomly oriented beryllium single crystals. At 90 at%Be, it is apparent from the micrographs, that the metastable weld microstructure, while still phase separated, no longer forms in the nucleation-free spinodal region, but rather in the binodal region of the miscibility gap throughout the weld cross-section. The resultant structure consists of large single crystals of beryllium, growing epitaxially from the base metal. These single crystals are interwoven with aluminum, but characterized by high angle boundaries between the different beryllium crystals.

Analysis of the dislocation distributions in the various microstructures indicates that when the microstructure forms from compositions in the spinodal region of the miscibility gap, the thermal strains from welding are randomly distributed throughout the aluminum matrix. However, under these conditions, the beryllium regions remains free of thermal strain induced dislocations. In contrast, transformation in the binodal region of the miscibility gap produces a less interconnected structure, characterized by high angle grain boundaries. Under these conditions, two notable changes occur: (i) dislocations are now present in some of the beryllium crystals, and (ii) large "crack-like" regions exist at some of the high angle beryllium crystal boundaries. These "crack-like" regions are typically backfilled with aluminum. As the amount of aluminum addition to the weld decreases below 20 at%Al, the number of dislocations and crack-like regions increases.

This paper will review the relationship between the weld microstructure of the weld and the crack susceptibility of electron beam welds in beryllium, as well as discuss the evolution of the microstructure as a function of composition and solidification conditions.

A Metastable Miscibility Gap Indicates the Possible Occurrence of a Liquid Phase Separated Structure

Beryllium-Aluminum Phase Diagram
(ref. J. Murray, NBS)



CHANGE IN PROOF STRESS VALUES OF CONSOLIDATED S65 BERYLLIUM POWDER SINCE 1979 .

by

P.A.Trimby (ROF Cardiff)

Summary

The 0.2% proof stress and UTS values of S65 powder, consolidated at ROF(C), have increased significantly since 1979 as shown by means of histograms. Possible reasons for these increases are discussed; particularly;-

1. Consolidating conditions.
2. Heat treatment.
3. Iron and Aluminium contents.
4. Silicon contents.
5. Beryllium oxide contents.
6. Particle size.

However, the 0.2% proof stress values, correlate with the grain size which suggests that the observed increase in proof stress derives from a reduction in the powder particle size.

INVESTIGATION OF THE CHANGE IN PROOF STRESS VALUES OF CONSOLIDATED S65 BERYLLIUM POWDER SINCE 1979

INTRODUCTION

During the latter part of 1986 a specification for the production of Trident weapon parts from consolidated beryllium powder was being formulated. During this process reference was made to the closely related specification for Chevaline weapon parts and also to actual recorded values obtained up to this period.

The existing Chevaline specification - "Material for Hot Pressed Beryllium Components" - required a 0.2% Proof Stress of 170 - 260MN/m², but it was noticed that reported values were approaching, and in one instance exceeding this upper limit. It was therefore decided to investigate the reasons for this apparent increase to enable a decision to be made on whether the Trident Proof Stress specification limit should be modified.

VARIATION OF MECHANICAL PROPERTIES

As the Brush Wellman powder batch numbers refer to the date on which the powder was produced, appendix 1 shows a histogram of Proof Stress values, supplied by Brush Wellman against the Brush Wellman powder batch numbers arranged in date order. Similarly it shows the Proof Stress values obtained by ROF(Cardiff) from hot pressed blocks and cold isostatically / hot isostatically pressed (CIP/HIP) logs for the same powders. In addition the Ultimate Tensile Strength (UTS) values from the Cardiff hot pressed blocks are also displayed.

It can readily be observed that the Proof Stress (and UTS) values have all generally increased.

POSSIBLE REASONS FOR INCREASE

There can be many reasons why mechanical properties of test pieces produced from consolidated beryllium powder can vary. Those considered in this exercise are:-

(1) Consolidating conditions.

(2) Heat treatment.

(3) Iron and aluminium content.

(4) Silicon content.

(5) Oxide content.

(6) Particle size.

Consolidating Conditions.

Those consolidating conditions likely to affect the mechanical properties are :-

(a) Temperature.

(b) Pressure.

(c) Time.

These consolidating conditions are closely controlled at ROF(Cardiff) and it seems unlikely that the gradual increase in Proof Stress values, since 1979, could be caused by these variations; especially as similar increases are observed for blocks produced by the Hot Press and CIP/HIP routes.

Heat Treatment

It has been shown many times that the heat treatment of Beryllium can significantly effect the mechanical properties; but as with the connsolidating conditions, the heat treatment process is also closely controlled. Therefore it would seem unlikely that the variation in the mechanical properties of both Hot Pressed and CIP/HIPed blocks could be caused by variations in the heat treatment conditions.

Iron and Aluminium Content

It has been demonstrated that free aluminium at the grain boundaries has a significant detrimental effect on the mechanical properties of beryllium. Iron, however, has the ability, under the right heat treatment conditions, to form a compound with aluminium ($AlFeBe_4$) and in effect remove free aluminium from the grain boundaries, and hence its deleterious effect. It is therefore standard practice, when procuring powder, to specify that the concentrations of iron and aluminium fall within an acceptability window. This window is such that the Fe/Al ratio is always better than 1.3 to 1.0. and as the stoicheiometry of these elements in the $FeAlBe_4$ compound is 2:1, it can be seen that this acceptability window will limit the concentration of free aluminium that can be present.

Appendix 2 shows the iron, aluminium and Fe/Al ratio for all the powder batches under consideration, and shows that the Fe/Al ratio never falls below the 1.3 to 1.0 specification limit.

Silicon Content

Silicon is a grain boundary constituent in beryllium and is known to aid the sintering of beryllium powder, particularly when using the Hot Press route. It is therefore conceivable that a variation in the silicon level could affect the mechanical properties, especially of Hot Pressed Components.

Appendix 2 shows the silicon concentrations of the powders and appendix 3 shows these silicon levels plotted against the proof stress values. These plots do not show a correlation between proof stress and silicon content.

Oxide Content.

It has been suggested that BeO particles can serve as sites of stress concentration, embrittling the Beryllium to premature fracture. Indeed this was shown by R.E.Evans, M.E.Faiers and D.Roberts [1] who demonstrated an increase in both 0.2% Proof Stress and UTS with increasing BeO content.

Appendix 4 fails to demonstrate a similar relationship between the Proof Stress values of Hot Pressed blocks and BeO content.

Particle Size.

It has been shown from dislocation theory that 0.2% proof stress should vary with grain size (a decrease in grain size resulting in an increase in Proof Stress). It is also known that powder particle size is the primary factor in determining final grain size. Consequently changes in the particle size of powders should manifest themselves in changes in the consolidated blocks proof stress figures.

A direct comparison was not possible, however, as the complete particle size distribution, of all the powders under consideration, was not available. Only the less than 45µm and less than 10µm figures were recorded for all powders, but as these results are rather imprecise it is not surprising that no correlation is observed.

A correlation does exist, however, between the grain size with the Proof Stress, and UTS as shown in appendix 5. The best fit to these coordinates is a curve, but the mechanical properties would be expected to vary with grain size according to the Petch- Hall relationship:-

$$\sigma = \sigma_o + Kd^{-\frac{1}{2}}$$

where σ = 0.2% Proof Stress

σ_o & K = Constants

d = Grain Size.

Therefore appendix 6 shows the relationship between $d^{-\frac{1}{2}}$ and the 0.2% Proof Stress and UTS.

DISSCUSION

The data expressed in appendix 6 shows a significant amount of scatter. It must be remembered, however, that this data was collected from averaging results from production quantities of powder. Consequently control of other parameters that are known to effect mechanical properties - such as aluminium or oxide content - was not possible.

CONCLUSIONS

1. A direct practical confirmation of the Petch Hall relationship has been established.

2. Although correlation between 0.2% Proof Stress and particle size was not possible from the data available, theoretical connections between particle size and grain size indicate strongly that the increase in 0.2% Proof Stress since 1979 can be attributed to a decrease in particle size over the same period.

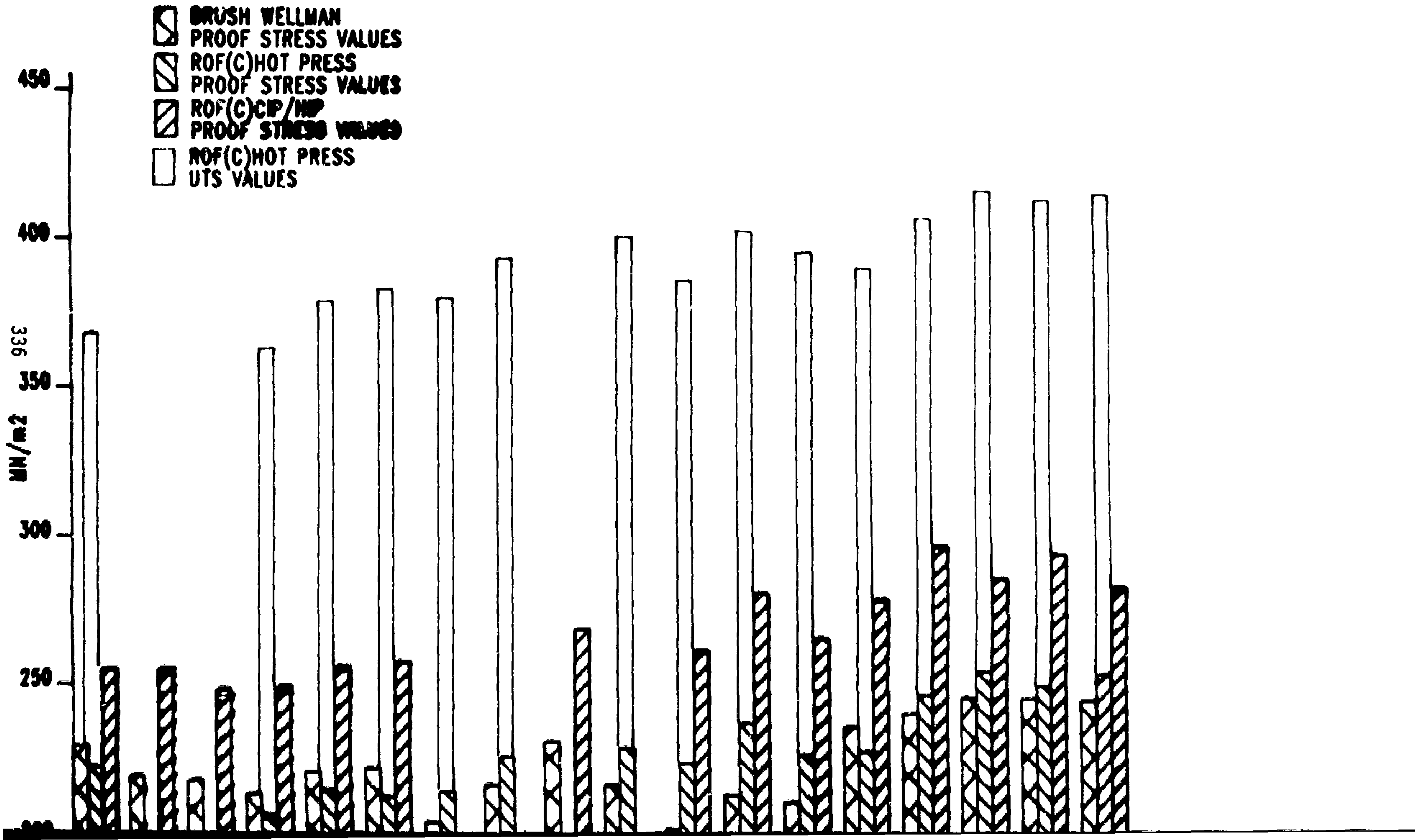
P.A.Trimby
Royal Ordnance Factory (Cardiff)
June 1987

jowog-ps

REFERENCES

[1] Effects of HIP Parameters on the Tensile Properties of P1, P10, Commercial Grade and S65 Beryllium Powders.
By R.E.Evans, M.E.Faiers and D.Roberts - AWRE.
From JOWOG 22/M19 - 8 to 11 September 1980.

MECHANICAL PROPERTIES OF BRUSH WELLMAN CONSOLIDATED POWDERS



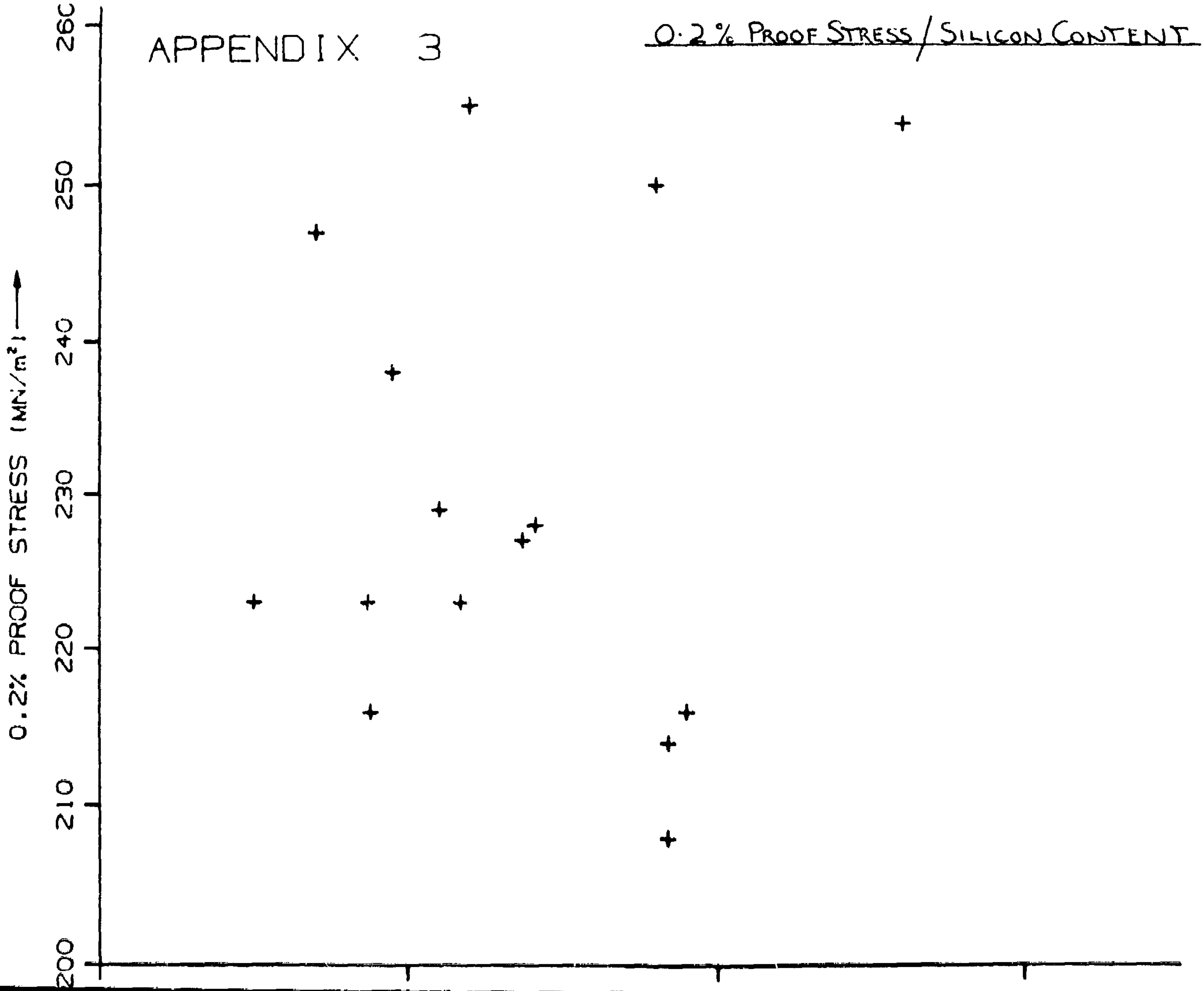
APPENDIX 2

A SUMMARY OF BRUSH WELLMAN 0.2% PROOF STRESS VALUES, CARDIFF CHEMICAL ANALYSIS OF POWDERS, HOT PRESSING AND CIP/HIP PRESSING MECHANICAL RESULTS

BRUSH WELLMAN		CARDIFF HOT PRESSING			CIP/HIP PRESSING		CARDIFF ANALYSIS OF POWDER					
BATCH No	PROOF STRESS MN/m2	AV GRAIN SIZE	PROOF STRESS MN/m2	UTS	PROOF STRESS MN/m2	UTS	Fe ppm	Al ppm	Fe/Al RATIO	Si ppm	PART SIZE 10 μm	%less than 45 μm
790809	230	14.7	223	369	255	435	630	300	2.1	150	7.0	100
791102	220	–	–	–	255	423	587	240	2.4	174	6.0	98
791202	219	–	–	–	248	414	622	200	3.1	201	8.0	98
800203	215	14.7	208	364	249	416	591	183	3.2	284	9.0	100
800303	222	–	216	380	256	429	594	183	3.2	290	10.0	100
800410	223	–	214	384	258	424	580	156	3.7	284	9.0	100
800512	206	17.2	216	381	–	–	570	250	2.4	188	10.0	98
800613	218	15.7	227	394	–	–	539	240	2.3	237	9.0	98
800715	232	–	–	–	269	446	546	250	2.1	182	8.0	98
800823	218	14.0	230	401	–	–	510	230	2.2	187	10.0	98
800919	204	15.6	225	387	262	437	576	220	2.6	217	13.0	98
801016	215	13.5	238	403	281	451	551	230	2.4	195	7.0	98
801113	213	15.0	228	396	266	445	541	230	2.4	241	11.0	99
840421	237	14.2	229	391	279	447	590	150	3.9	210	15.0	100
840617	241	13.8	247	407	297	463	550	380	1.4	170	9.5	98
841201	246	12.9	255	416	286	465	650	300	2.2	220	15.0	99
850427	246	13.4	250	413	294	461	630	350	1.8	280	12.0	100
850917	245	17.9	254	415	283	459	730	190	3.8	360	10.0	100

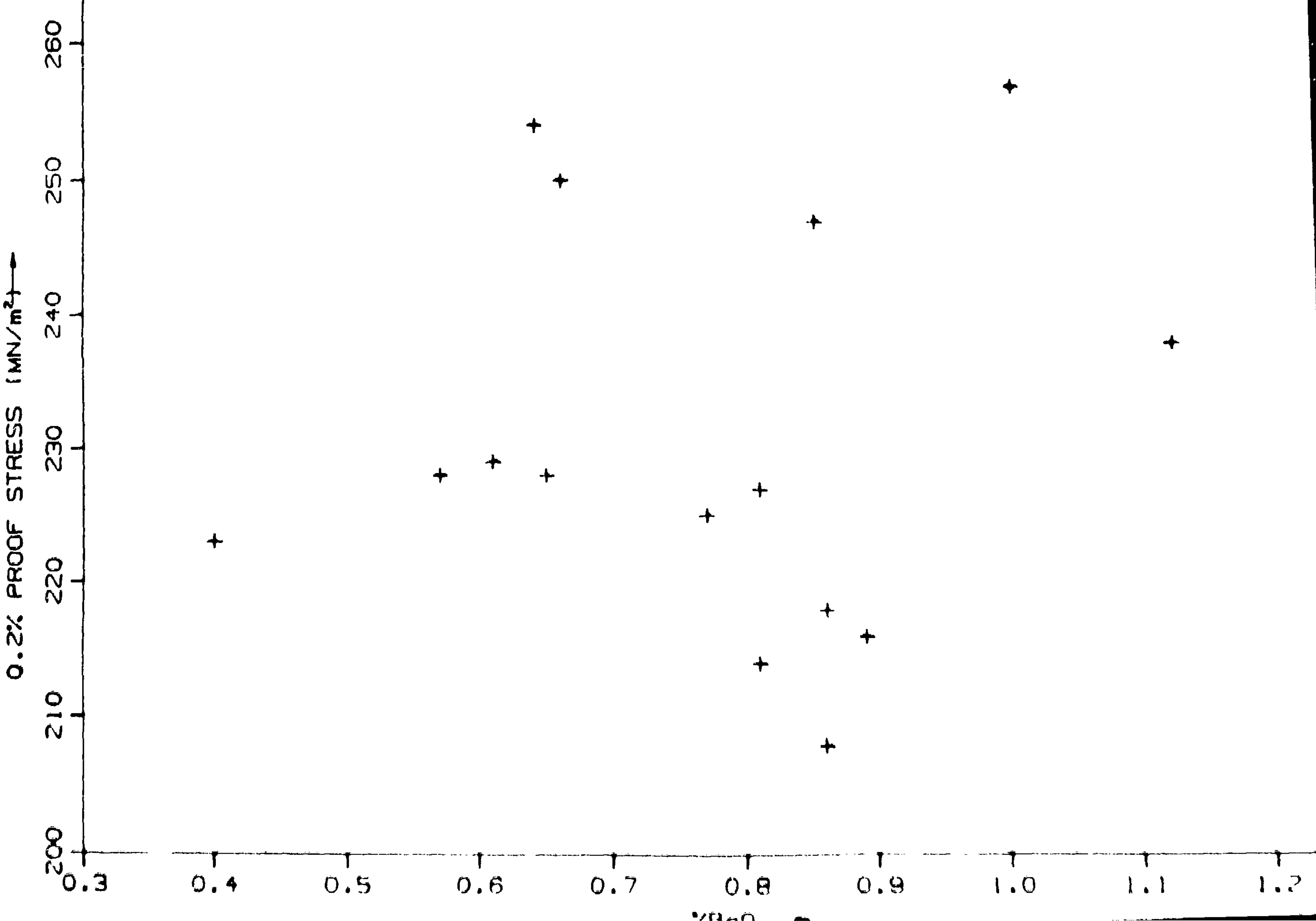
APPENDIX 3

0·2% PROOF STRESS / SILICON CONTENT



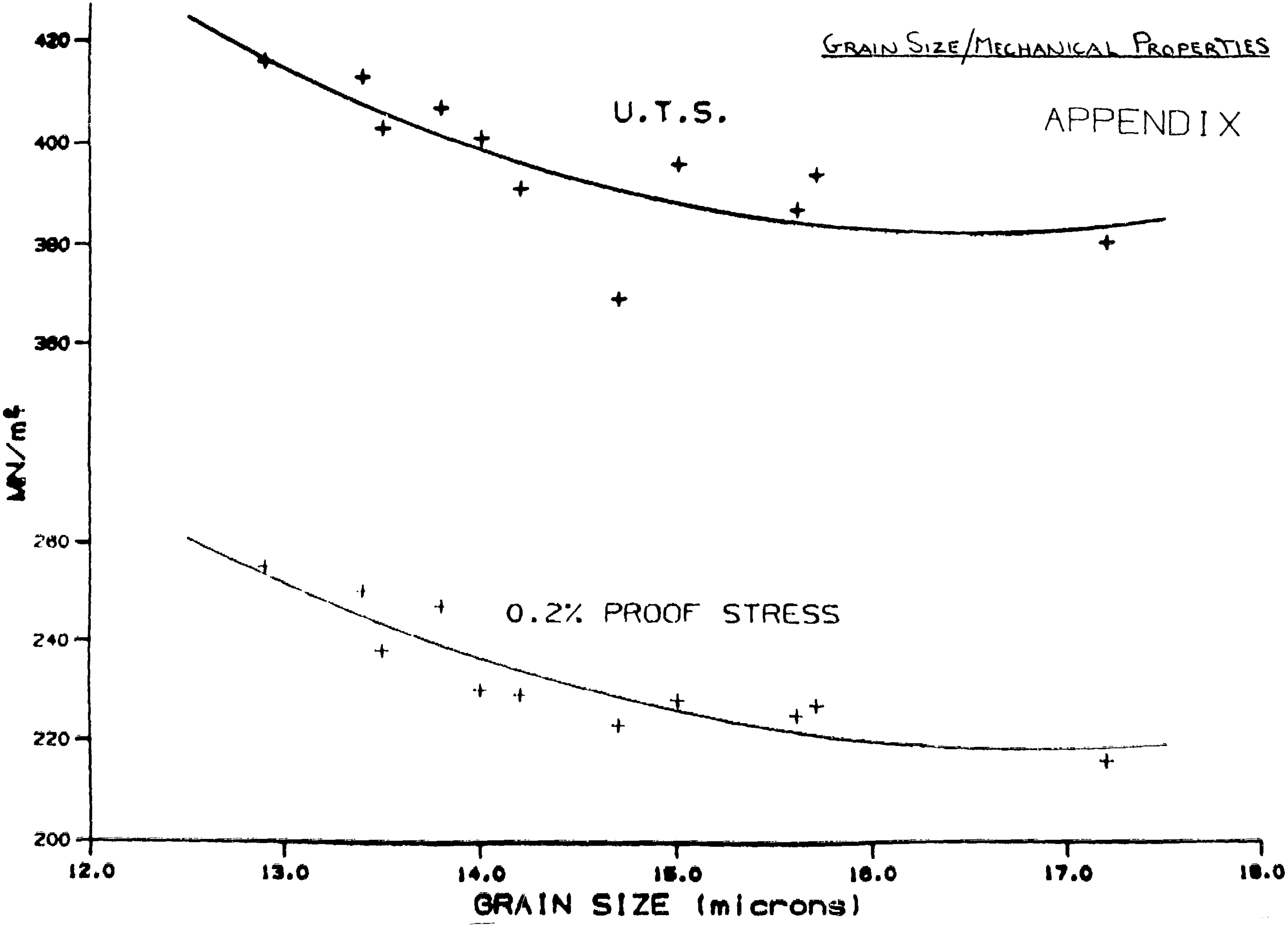
APPENDIX 4

0.2% PROOF STRESS/BeO CONTENT

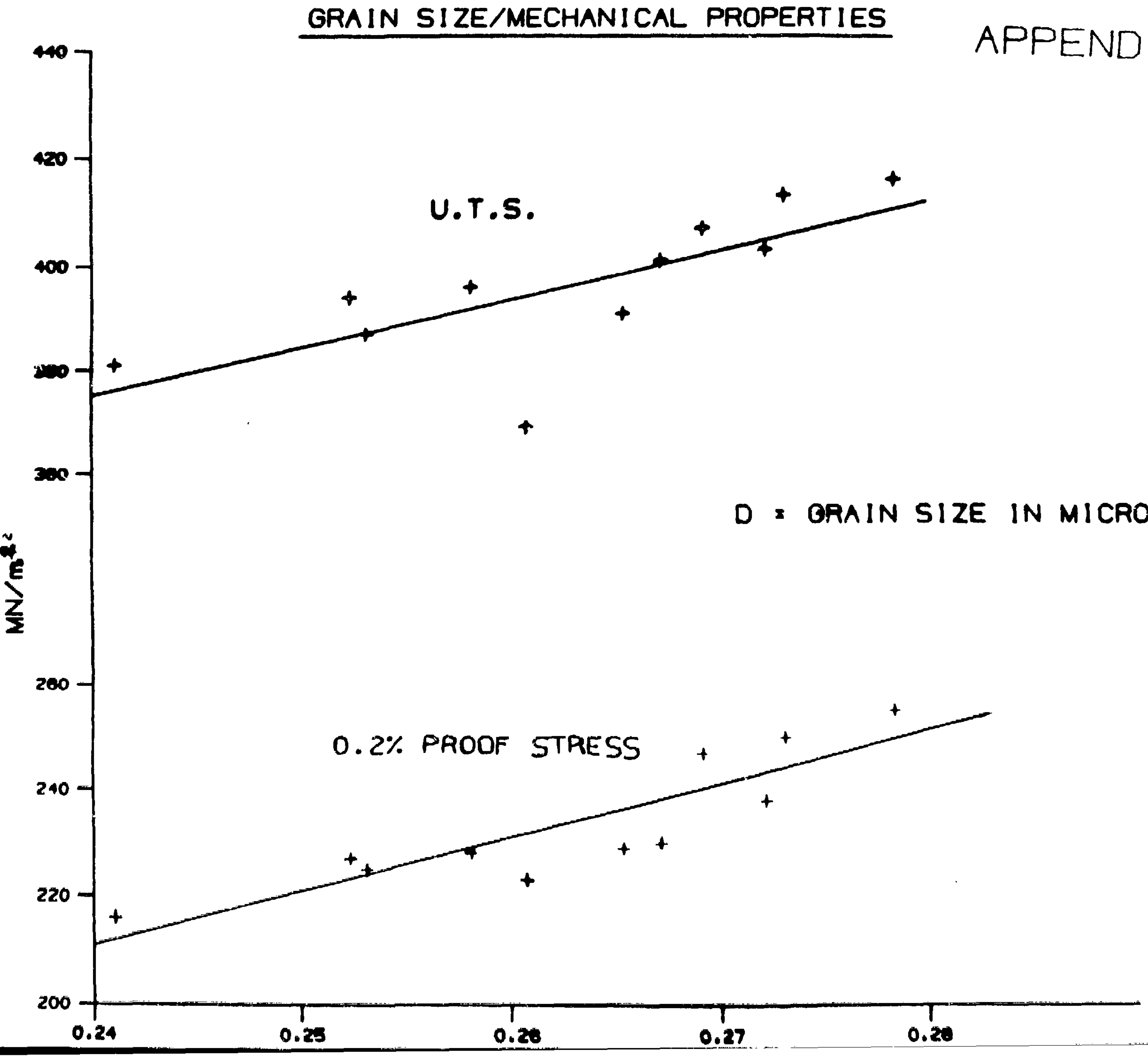


GRAIN SIZE/MECHANICAL PROPERTIES

APPENDIX 5



APPENDIX 6


GRAIN SIZE/MECHANICAL PROPERTIES
440
420
400
380
360
260
240
220
200
MN/m²
U.T.S.
0.2% PROOF STRESS
D = GRAIN SIZE IN MICRONS
0.24
0.25
0.26
0.27
0.28

A BERYLLIUM/STAINLESS STEEL COMPATIBILITY COATING

by

H.G.Rhodes and D.Roberts, AWRE

Summary

A compatibility coating consisting of a suspension of erbium oxide in an aqueous solution of erbium nitrate has been used to protect stainless steel tooling during the hot compaction of beryllium powder. The effectiveness of the coating has been evaluated at temperatures up to 1000°C in hot isopressing and 1050°C in direct hot pressing operations. In both cases it was found that the coatings were sufficiently durable to withstand three pressing cycles without intermediate repair.

COATED MANDRELS

SLIDE 1 (introduction)

THE AIM OF DEVELOPMENT WORK WAS TO REDUCE PREFORM WEIGHT BY PRESSING THE PREFORM CLOSER TO NET SHAPE

SLIDE 2 (problem and solution)

In die hot pressing with a graphite core the preform wall has to be a minimum thickness to resist contraction stresses and the present size is about optimum therefore there was little possibility of improving the process

If a graphite core was replaced with a metal mandrel then the preform after pressing would shrink less than the mandrel and the minimum wall thickness limit would not apply

A stainless steel mandrel would be ideal but pressing at above about 1000 c reaction between beryllium and stainless would destroy the mandrel

The possible isopressing routs uses a stainless mandrel but reaction between beryllium and bare stainless steel limits the use of mandrel to about three pressings after which the mandrel becomes rough and can be locked in the mandrel

SLIDE 3 (used mandrel with reaction layer)

The solution to both these problem would be to use a stainless steel mandrel coated with a refractory oxide coating.

SLIDE 4 (reasons why plasma out verbal only)

using plasma and flame spraying mandrels have been coated with alumina have been tested for both die hot pressing and hot isopressing for hot pressing at about1050 c the reaction destroyed the mandrel for isopressing at about 950 1000 the reaction was less but after being used three times the mandrel was so rough that it was locked in the preform.

Carrying out this work showed that one problem was that if used for isopressing the mandrel would have to be recoated after each use .

If the coating was done by a specialised firm then the mandrel would require cleaning and decontamination after each pressing run . The time required for this would make the process unworkable

WHAT WAS REQUIRED WAS A SIMPLE PROCESS THAT COULD BE CARRIED OUT IN HOUSE.

SLIDE 5 (erbia coating method)

Work on plutonium casting had reported using a coating based on erbium oxide suspension in a saturated solution of erbium nitrate in water and the claim was that the coating could be used up to 100 times before it broke down

This coating and its method of applying appeared ideal if was compatible with beryllium.

SLIDE 6 (experimental work description)

The best method of finding if erbium oxide was compatible with beryllium was to test a stainless steel mandrel coated with the mixture. For the first test a stainless steel mandrel of same size as the conventional graphite core used for hot pressing a 110mm preform was prepared and used to Hot press a 110mm diam preform at 1050C and 0.5 tpi . After removal from the die it was found that the mandrel was loose in the preform and the coating was intact but had changed colour from pink to dirty gray

SLIDE 7 (hot pressed preform small mandrel)

A second and third pressing were then made with the same mandrel

After the third pressing the coating was beginning to break down

The second and third preforms show that the density was possibly low.

As the result of the success of the first test a second hot pressing mandrel was manufactures with dimensions about 0.25 " larger than the previous mandrel .

SLIDE 8 (hot pressed mandrel large mandrel)

The first hot pressing at 1050C and 0.5tpi produced a sound preform but the density in the wall area appeared low for the next pressing the load was increased to 0.75 tpi and to 1.0 tpi for the third pressing when 100% density was attained

After the third pressing the coating appeared to be breaking and the mandrel was cleaned for recoating. Trials with the recoated core are to be carried out.

SLIDE 9 (coated mandrels)

The initial work was carried out with probably poor coating and the typical coatings shown were prepared after the coating technique had been improved

The isopressing mandrel shown [redacted]e in slide was used in an hot isopressing assembly . This was again successful up to three cycles

SLIDE 10 Isopressed mandrel and
preform after three runs

The next slide shows a die hot pressing carried out with the same size core as the large mandrel used in the coating experiment. The preform wall was cracked and opened by about 1/4 " showing the magnitude of the differential strain between core and preform

SLIDE 11 Preform pressed on
large graphite core

SLIDE 12

CONCLUDING

It appears that Erbium oxide coating can be used for protecting stainless steel mandrels from reaction with beryllium up to 1050C The technique requires futher development These are now being looked at

THESE ARE

A MORE PROTECTIVE COATING

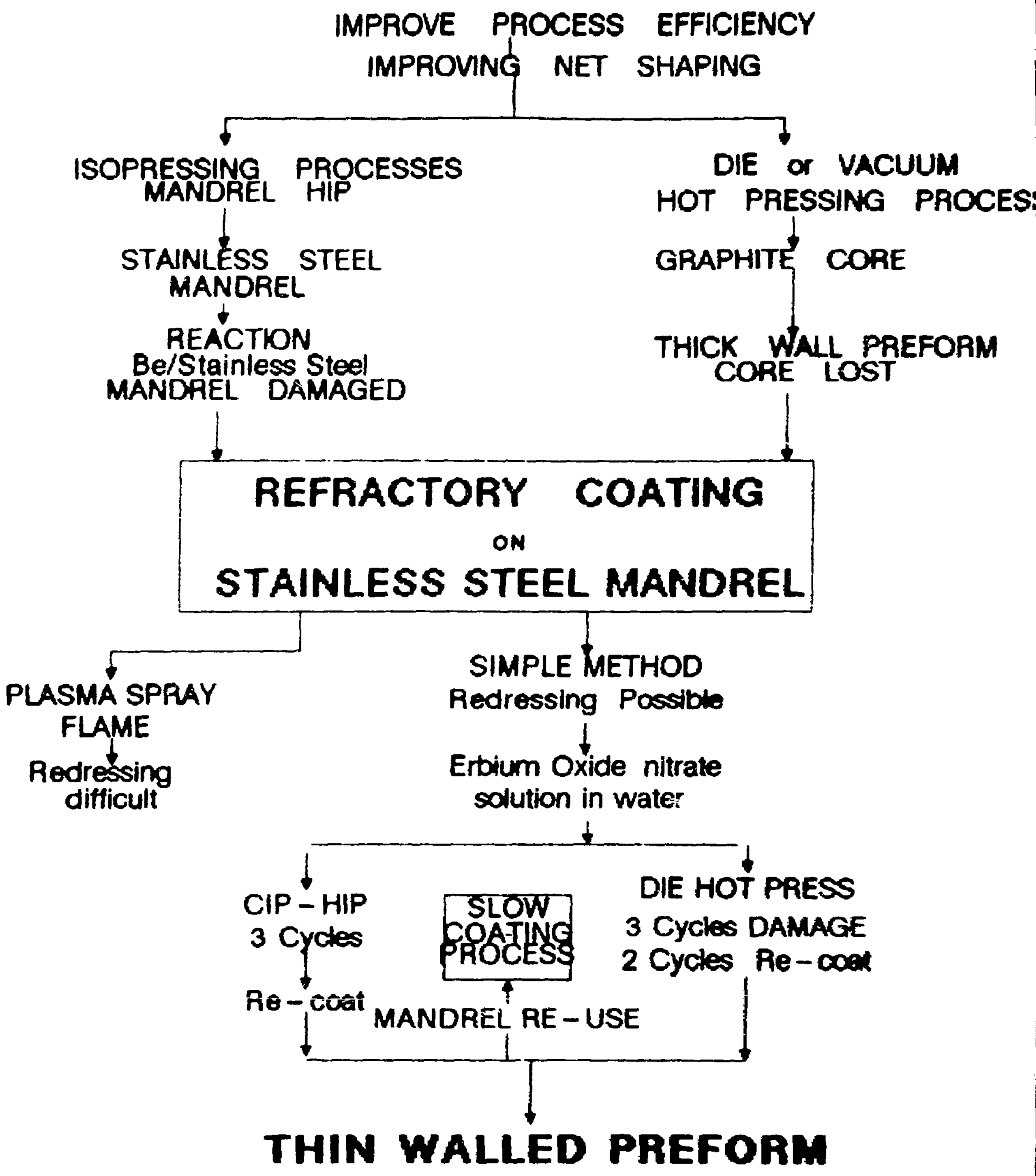
A METHOD OF APPLYING A POSSIBLY THICK COAT WHICH WOULD BE APPLIED IN A VERY SHORT TIME BUT WOULD ONLY LAST FOR ONE PRESSING

A CHEAPER COATING BASED ON THE SAME METHOD OF APPLICATION BUT BASED ON YTTRIA THE FIRST TESTS SEAM TO SHOW THAT THIS COAT MAY ACT BETTER THAN ERBIA

SLIDE 1

[redacted]

DEVELOPMENT OF A REFRACTORY COATING TO PROTECT STAINLESS STEEL MANDREL DURING THE HOT PRESSING OF BERYLLIUM POWDER



IMPROVE PROCESS EFFICIENCY
IMPROVING NET SHAPING

ISOPRESSING PROCESSES
MANDREL HIP

STAINLESS STEEL
MANDREL

REACTION
Be/Stainless Steel
MANDREL DAMAGED

DIE or VACUUM
HOT PRESSING PROCES

GRAPHITE CORE

THICK WALL PREFORM
CORE LOST

REFRACTORY COATING
ON
STAINLESS STEEL MANDREL

PLASMA SPRAY
FLAME

Redressing
difficult

SIMPLE METHOD
Redressing Possible

Erbium Oxide nitrate
solution in water

CIP – HIP
3 Cycles

Re – coat

SLOW
COATING
PROCESS

MANDREL RE – USE

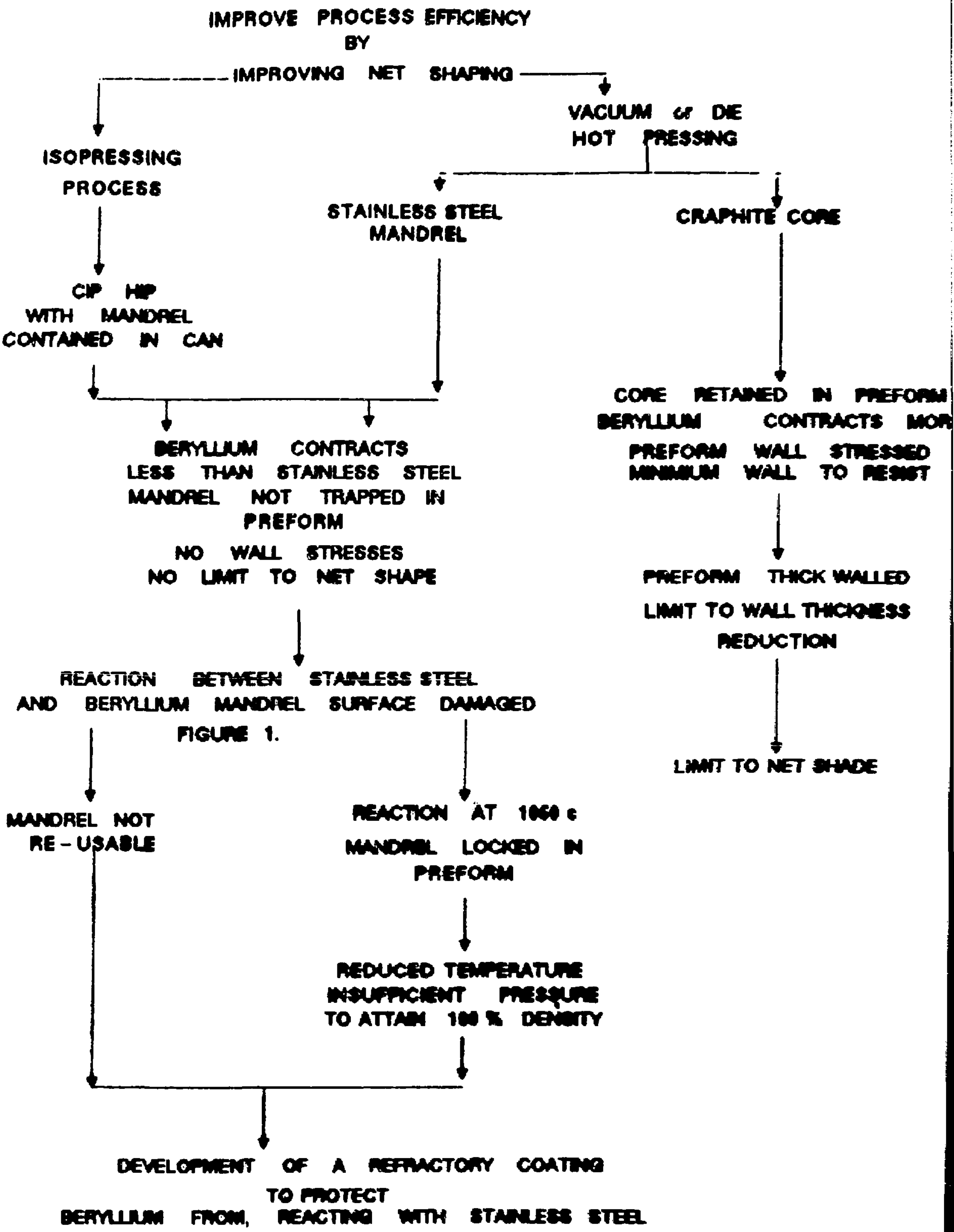
DIE HOT PRESS
3 Cycles DAMAGE
2 Cycles Re – coat

THIN WALLED PREFORM

SLIDE. 2

[redacted]

DEVELOPMENT OF A REFRACTORY COATING TO PROTECT STAINLESS STEEL MANDREL DURING THE HOT PRESSING OF BERYLLIUM POWDER



IMPROVE PROCESS EFFICIENCY
BY
IMPROVING NET SHAPING

ISOPRESSING
PROCESS

CIP HIP
WITH MANDREL
CONTAINED IN CAN

VACUUM or DIE
HOT PRESSING

STAINLESS STEEL
MANDREL

GRAPHITE CORE

BERYLLIUM CONTRACTS
LESS THAN STAINLESS STEEL
MANDREL NOT TRAPPED IN
PREFORM
NO WALL STRESSES
NO LIMIT TO NET SHAPE

CORE RETAINED IN PREFORM
BERYLLIUM CONTRACTS MOR
PREFORM WALL STRESSED
MINIMUM WALL TO RESIST

PREFORM THICK WALLED
LIMIT TO WALL THICKNESS
REDUCTION

LIMIT TO NET SHADE

REACTION BETWEEN STAINLESS STEEL
AND BERYLLIUM MANDREL SURFACE DAMAGED
FIGURE 1.

MANDREL NOT
RE-USABLE

REACTION AT 1060 c
MANDREL LOCKED IN
PREFORM

REDUCED TEMPERATURE
INSUFFICIENT PRESSURE
TO ATTAIN 100 % DENSITY

DEVELOPMENT OF A REFRACTORY COATING
TO PROTECT
BERYLLIUM FROM, REACTING WITH STAINLESS STEEL

SLIDE 3

[redacted]

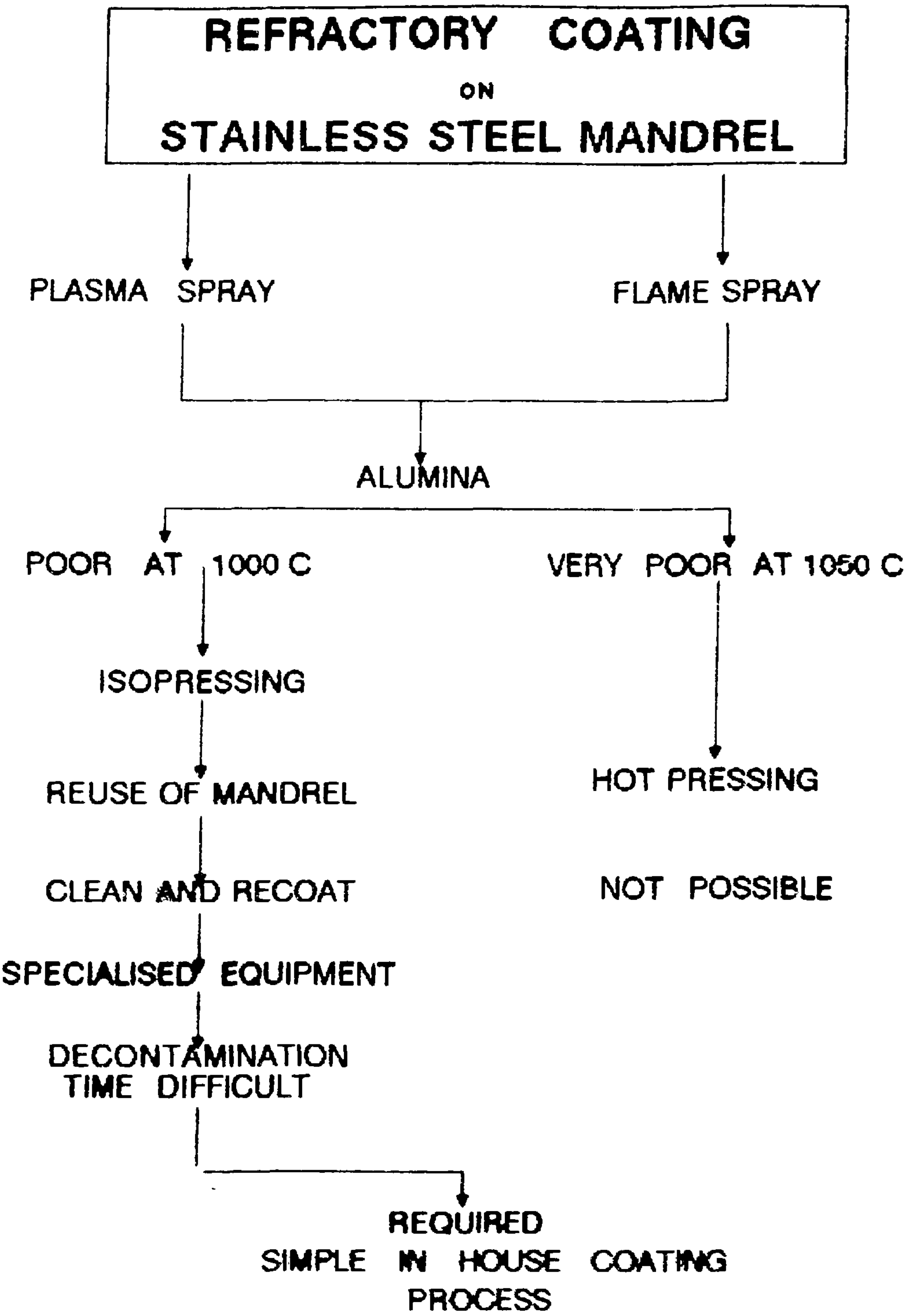
FIGURE 1.

Stainless steel mandrel (unprotected) after be used once for the hot isopressing of beryllium 1000°C and 1 kbar. Reaction layer visible surface.

SLIDE 4

[redacted]

DEVELOPMENT OF A REFRACTORY COATING TO PROTECT STAINLESS STEEL MANDREL DURING THE HOT PRESSING OF BERYLLIUM POWDER



REFRACTORY COATING
ON
STAINLESS STEEL MANDREL

PLASMA SPRAY

FLAME SPRAY

ALUMINA

POOR AT 1000 C

VERY POOR AT 1050 C

ISOPRESSING

REUSE OF MANDREL

CLEAN AND RECOAT

SPECIALISED EQUIPMENT

DECONTAMINATION
TIME DIFFICULT

HOT PRESSING

NOT POSSIBLE

REQUIRED
SIMPLE IN HOUSE COATING
PROCESS

SLIDE 5

[redacted]

DEVELOPMENT OF A REFRACTORY COATING TO PROTECT STAINLESS STEEL MANDREL DURING THE HOT PRESSING OF BERYLLIUM POWDER

COATING REPORTED FOR USE
TO PROTECT CASTING MOULDS
FOR RADIO-ACTIVE METALS

↓

ERBIUM OXIDE SUSPENDED IN
AN AQUEOUS ERBIUM NITRATE SOLUTION

↓

APPLIED TO A SHOT BLASTED SURFACE
BY
WIPING SURFACE WITH A RAG DIPPED
IN THE SUSPENSION

↓

MANDREL FIRED AT 560 C

↓

SECOND COAT OF ERBIUM NITRATE WIPED ON

↓

PROCESS REPEATED TO BUILD UP COAT
PROCESS SLOW BUT SUITABLE FOR
IN HOUSE USE

↓

COATING LIFE CLAIMED 100 CYCLES

SLIDE 6

[redacted]

DEVELOPMENT OF A REFRACTORY COATING TO PROTECT STAINLESS STEEL MANDREL DURING THE HOT PRESSING OF BERYLLIUM POWDER

COATING REQUIRED FOR
HOT ISOPRESSING

↓

SLOW PROCESS

↓

RAPID EVALUATION BY TESTING
ON HOT PRESSING MANDREL

↓

EVALUATE COATING BY AMOUNT OF REACTION
ON COATED MANDREL AFTER VACUUM HOT PRESSING
AT A RESTRICTED TEMPERATURE OF 1050 C
DENSITY OF PREFORM NOT IMPORTANT

↓

COATED MANDRELS ISOPRESSING AND HOTPRESSING
TO PRODUCE 110mm PREFORMS

↓

110mm PREFORM PRESSED FROM
POWDER AT 1050 C and 7.7N/mm
BREAKDOWN OF COATING AFTER THIRD CONSECUTIVE PRESSING
PREFORM DENSITY LOW (FIGURE 2)

↓

HOT PRESSING EXPERIMENT REPEATED AT
MANDREL SIZE INCREASED
THREE CONSECUTIVE PRESSING BEFORE BREAKDOWN
PRESSING LOAD INCREASED TO 15.6 N/mm
PREFORM DENSITY 100%
FIGURE 3

↓

REPEAT WORK USING CIP/HIP PROCESS
THIRD PREFORM PRODUCED ON COATED PREFORM
PRESSING CONDITIONS 1 KBAR 950 - - 1000c
COATING BREAKDOWN EVIDENT
FIGURE 5

↓

PREFORM HOT PRESSED USING GRAPHITE CORE OF
SAME SIZE AS STAINLESS STEEL MANDREL FIGURE 3
FIGURE 6

SLIDE 7

FIGURE 2.

Second and third preforms (110mm diam) of a seri
of three produced by die hot pressing using
erbia coated stainless steel mandrel of same size
normal graphite core.
Note: Coating break-down visible on third prefo
and on mandrel.

SLIDE 8

[redacted]

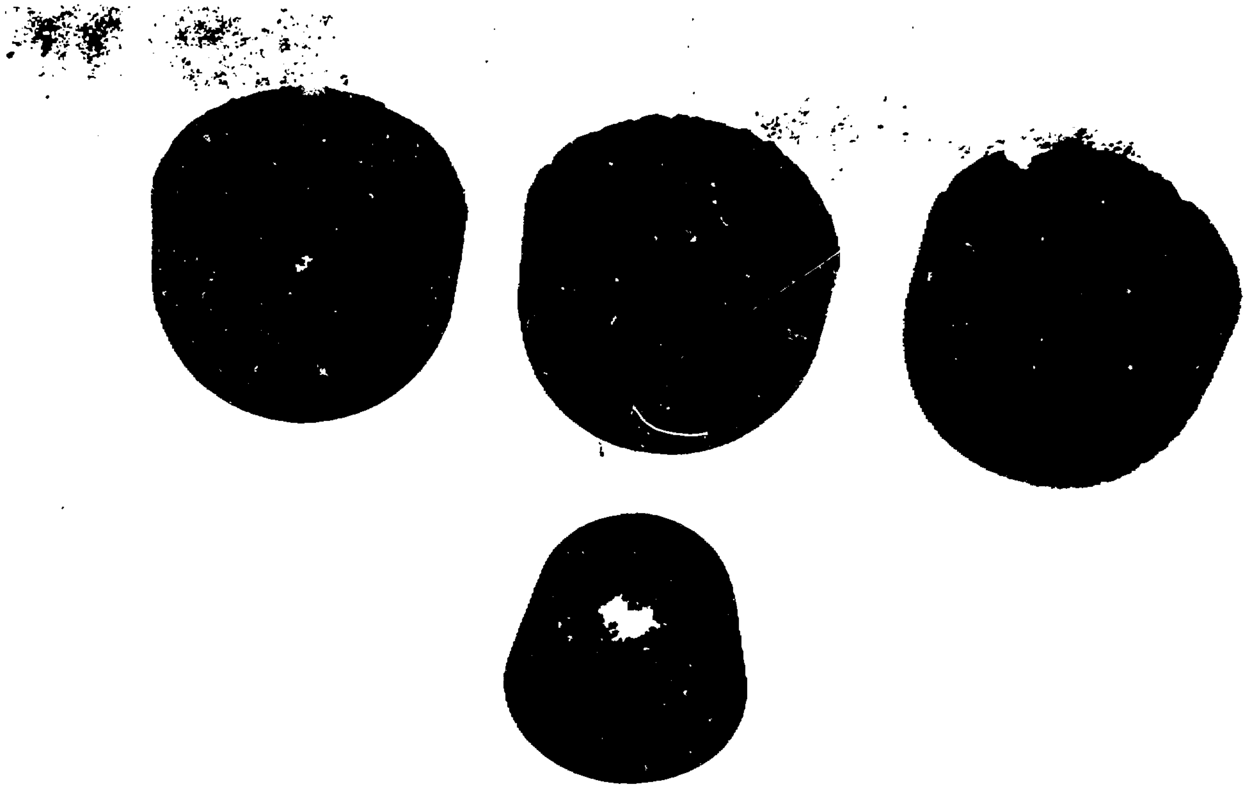


FIGURE 3.

A series of three preforms (110mm diam) produced by die hot pressing using an erbium coated stainless steel mandrel.

Preform wall thicknesss reduced by use of large mandrel. To attain good preform density the applied pressure was increased for each pressing

(a) 7.7 n/mm^2

(b) 11.5N/mm^2

(c) 15.5N/mm^2

SLIDE 9

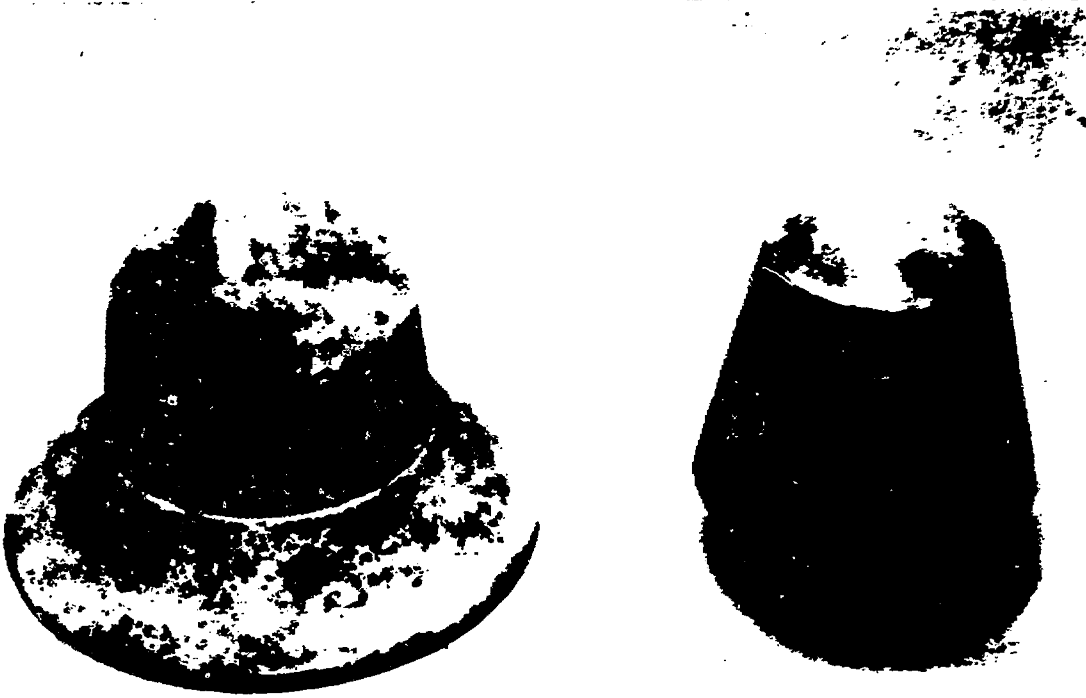


FIGURE 4.
Various stainless steel pressing mandrels
(110mm dia preform)
(a) Hot pressing mandrel
(b) Isopressing mandrel as coated (early development)

SLIDE 16

[redacted]

FIGURE 5.
Third isopressed preform produced on an erbia coatedstainless steel mandrel.
Note: Coating break-down visible.

SLIDE 11

[redacted]

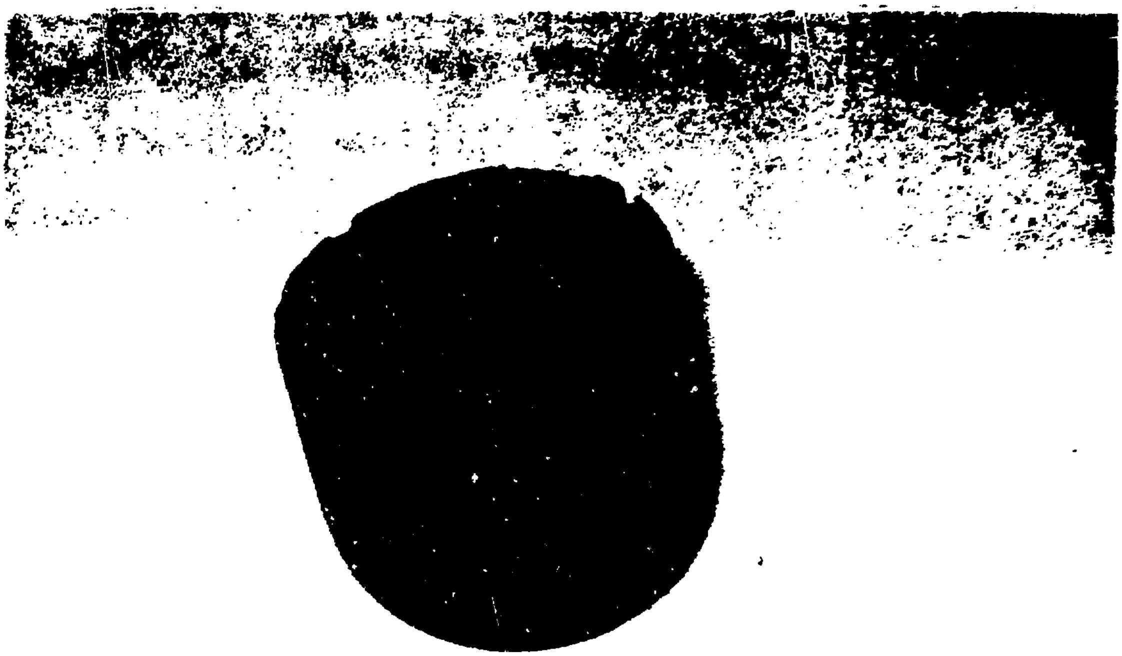


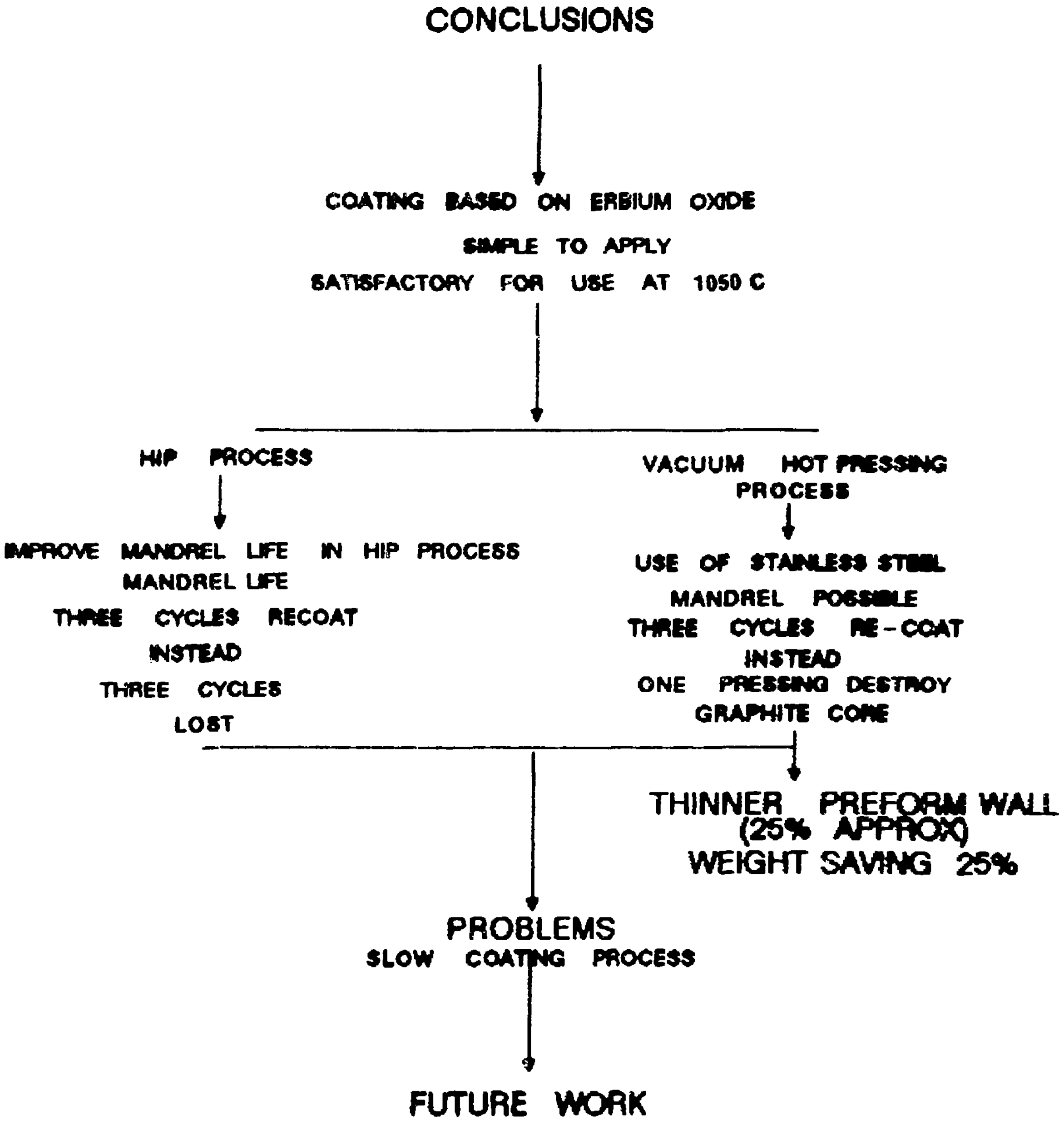
FIGURE 6.

Preform produced using a graphite core of similar size to stainless steel mandrel used for preforms displayed in FIGURE 3

SLIDE 12

[redacted]

DEVELOPMENT OF A REFRACTORY COATING TO PROTECT STAINLESS STEEL MANDREL DURING THE HOT PRESSING OF BERYLLIUM POWDER



CONCLUSIONS

COATING BASED ON ERBIUM OXIDE

SIMPLE TO APPLY

SATISFACTORY FOR USE AT 1050 C

HIP PROCESS

IMPROVE MANDREL LIFE IN HIP PROCESS
MANDREL LIFE
THREE CYCLES RECOAT
INSTEAD
THREE CYCLES
LOST

VACUUM HOT PRESSING PROCESS

USE OF STAINLESS STEEL
MANDREL POSSIBLE
THREE CYCLES RE-COAT
INSTEAD
ONE PRESSING DESTROY
GRAPHITE CORE

THINNER PREFORM WALL
(25% APPROX)
WEIGHT SAVING 25%

PROBLEMS

SLOW COATING PROCESS

FUTURE WORK

TEST FULL SIZE 200mm PREFORM

DEVELOP QUICK COATING TECHNIQUE

EXAMINE POSIBILITY OF OTHER COATING OXIDES SIMILAR SYSTEMS

YTTRIA