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METALLURGY OF URANIUM AND ITS ALLOYS

Edited by

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Classification cancelled (or changed to unclassified)
memo from Branch, dated 9-29-64
by J.C. Ridenour, date 4-28-65

Oak Ridge, Tennessee
United States Atomic Energy Commission
Technical Information Service
1953

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Copy Numbers

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Battelle Memorial Institute	36-40
Brookhaven National Laboratory	41-50
Brush Beryllium Co.	51
Bureau of Ships	52
California Research and Development Company	53-54
Carbide & Carbon Chemicals Company (K-25 Plant)	55-59
Carbide & Carbon Chemicals Company (ORNL)	60-74
Carbide & Carbon Chemicals Company (Y-12 Area)	75-82
Chicago Patent Group	83
Commonwealth Edison Co.	84
Curtiss-Wright Corp., Wood-Ridge, N. J.	85
Detroit Edison Co., N. Y.	86
Dow Chemical Company (Rocky Flats)	87-89
Du Pont Company, Augusta	90-96
Du Pont Company, Wilmington	97
Foster Wheeler Corp.	98
General Electric Company (ANPP)	99-103
General Electric Company, Richland	104-118
Hanford Operations Office	119
Idaho Operations Office	120-123
Iowa State College	124-128
Knolls Atomic Power Laboratory	129-136
Los Alamos Scientific Laboratory	137-144
Mallinckrodt Chemical Works	145-148
Massachusetts Institute of Technology (Kaufmann)	149
Materials Laboratory (WADC)	150
Monsanto Chemical Co.	151
Mound Laboratory	152
National Advisory Committee for Aeronautics, Cleveland	153
National Bureau of Standards	154

(Continued on Page 2)

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METALLURGY AND CERAMICS
(Continued)

Special Distribution	Copy Numbers
National Lead Company of Ohio	155-158
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North American Aviation, Inc.	171-173
Patent Branch, Washington	174
Sylvania Electric Products, Inc.	175-176
U. S. Bureau of Mines, Albany, Ore.	177
University of California Research Laboratory, Berkeley	178-181
University of California Research Laboratory, Livermore	182-185
Walter Kidde Nuclear Laboratories Inc.	186
Westinghouse Electric Corporation	187-190
Technical Information Service, Oak Ridge	191-254

2
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NATIONAL NUCLEAR ENERGY SERIES
Manhattan Project Technical Section
Division IV—Plutonium Project Record
Volume 12A

METALLURGY OF URANIUM AND ITS ALLOYS

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3

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FOREWORD

Since the discovery of practical means of utilizing the energy of the atomic nucleus, a large and complex atomic energy industry has begun in the United States. As a result of conditions in the world, external to the United States, the requirements of national security have been paramount in our development of this industry thus far. Constant and increasing attention, however, has been given to the problems of economic nuclear power and to the medical and industrial applications of radioactive materials with a view toward "improving the public welfare, increasing the standard of living, strengthening free competition in private enterprise, and promoting world peace." To this end the Atomic Energy Commission has sought the most effective means to accelerate the practical exploitation of nuclear data by American science and industry. The National Nuclear Energy Series is designed to provide for scientists and engineers as comprehensive a source of such data as is possible. The scope of the information presented in these volumes is a measure of American achievements to date in the field of atomic science.

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Lewis L. Strauss, Chairman
U. S. Atomic Energy Commission

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32

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2
3
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PREFACE

This volume is one of a series which has been prepared as a record of the research work done under the Manhattan Project and the Atomic Energy Commission. The name Manhattan Project was assigned by the Corps of Engineers, War Department, to the far-flung scientific and engineering activities which had as their objective the utilization of atomic energy for military purposes. In the attainment of this objective, there were many developments in scientific and technical fields which are of general interest. The National Nuclear Energy Series (Manhattan Project Technical Section) is a record of these scientific and technical contributions, as well as of the developments in these fields which are being sponsored by the Atomic Energy Commission.

The National Nuclear Energy Series, when completed, is expected to consist of approximately 100 volumes. These will be grouped into ten divisions, as follows:

- Division I—Electromagnetic Separation Project
- Division II—Gaseous Diffusion Project
- Division III—Special Separations Project
- Division IV—Plutonium Project
- Division V—Los Alamos Project
- Division VI—University of Rochester Project
- Division VII—Materials Procurement Project
- Division VIII—Manhattan Project
- Division IX—Thermal Diffusion Project
- Division X—Centrifuge Project

Soon after the close of the war the Manhattan Project was able to give its attention to the preparation of a complete record of the research work accomplished under Project contracts. Writing programs were authorized at all laboratories, with the object of obtaining complete coverage of Project results. Each major installation was requested to designate one or more representatives to make up a committee, which was called first the Manhattan Project Editorial Advisory Board and later simply the Project Editorial Advisory Board. This group was planned to coordinate the writing programs at all the installations and to act as an advisory group in all matters affecting the Project-wide writing program.

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The names of the Board members and of the installations which they represented are as follows:

Atomic Energy Commission Public and Technical Information Service	Alberto F. Thompson
Technical Information Division, Oak Ridge Extension	Brewer F. Boardman
Office of New York Operations	Charles Slessor, J. H. Hayner, W. M. Hearon *
Brookhaven National Laboratory	Richard W. Dodson
Carbide & Carbon Chemicals Corporation (K-25)	R. B. Korsmeyer, W. L. Harwell, D. E. Hull, Ezra Staple
Carbide & Carbon Chemicals Corporation (Y-12) †	Russell Baldock
Clinton Laboratories ‡	J. R. Coe
General Electric Company, Hanford	T. W. Hauff
General Electric Company, Knolls Atomic Power Laboratory	John P. Howe
Kellex Corporation	John F. Hogerton, Jerome Simson, M. Benedict
Los Alamos	R. R. Davis, Ralph Carlisle Smith
National Bureau of Standards	C. J. Rodden
Plutonium Project Argonne National Laboratory	R. S. Mulliken, H. D. Young
Iowa State College	F. H. Spedding
Medical Group	R. E. Zirkle
SAM Laboratories §	G. M. Murphy
Stone & Webster Engineering Corporation	B. W. Whitehurst
University of California	R. K. Wakerling, A. Guthrie
University of Rochester	D. R. Charles, M. J. Wantman

* Represented Madison Square Area of the Manhattan District.

† The Y-12 plant at Oak Ridge was operated by Tennessee Eastman Corporation until May 4, 1947, at which time operations were taken over by Carbide & Carbon Chemicals Corporation.

‡ Clinton Laboratories was the former name of the Oak Ridge National Laboratory.

§ SAM (Substitute Alloy Materials) was the code name for the laboratories operated by Columbia University in New York under the direction of Dr. H. C. Urey, where much of the experimental work on isotope separation was done. On Feb. 1, 1945, the administration of these laboratories became the responsibility of Carbide & Carbon Chemicals Corporation. Research in progress there was transferred to the K-25 plant at Oak Ridge in June, 1946, and the New York laboratories were then closed.

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Many difficulties were involved in preparing a unified account of Atomic Energy Project work. For example, the Project Editorial Advisory Board was the first committee ever organized with representatives from every major installation of the Atomic Energy Project. Compartmentation for security was so rigorous during the war that it had been considered necessary to allow a certain amount of duplication of effort rather than to permit unrestricted circulation of research information between certain installations. As a result, the writing programs of different installations inevitably overlapped markedly in many scientific fields. The Editorial Advisory Board has exerted itself to reduce duplication in so far as possible and to eliminate discrepancies in factual data included in the volumes of the NNES. In particular, unified Project-wide volumes have been prepared on Uranium Chemistry and on the Analysis of Project Materials. Nevertheless, the reader will find many instances of differences in results or conclusions on similar subject matter prepared by different authors. This has not seemed wholly undesirable for several reasons. First of all, such divergencies are not unnatural and stimulate investigation. Second, promptness of publication has seemed more important than the removal of all discrepancies. Finally, many Project scientists completed their contributions some time ago and have become engrossed in other activities so that their time has not been available for a detailed review of their work in relation to similar work done at other installations.

The completion of the various individual volumes of the series has also been beset with difficulties. Many of the key authors and editors have had important responsibilities in planning the future of atomic energy research. Under these circumstances, the completion of this technical series has been delayed longer than its editors wished. The volumes are being released in their present form in the interest of presenting the material as promptly as possible to those who can make use of it.

As many as possible of the volumes have been declassified for general distribution. Reproduction of this classified edition has been approved only for limited circulation within the Atomic Energy Project, and circulation of this edition must be closely limited to facilities and personnel definitely associated with work done for the Atomic Energy Commission. The negatives from which the limited edition is prepared will be preserved and will be available for publication of a public edition at such time as such action is determined to be consistent with the national security.

The Editorial Advisory Board

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The Manhattan Project Technical Section of the National Nuclear Energy Series is intended to be a comprehensive account of the scientific and technical achievements of the United States program for the development of atomic energy. It is not intended to be a detailed documentary record of the making of any inventions that happen to be mentioned in it. Therefore, the dates used in the Series should be regarded as a general temporal frame of reference, rather than as establishing dates of conception of inventions, of their reduction to practice, or of occasions of first use. While a reasonable effort has been made to assign credit fairly in the NNES volumes, this may, in many cases, be given to a group identified by the name of its leader rather than to an individual who was an actual inventor.

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PLUTONIUM PROJECT RECORD FOREWORD

Discontinued

This report is a technical account of information collected while developing methods for producing plutonium. Some of the information deals directly with nuclear physics and chemistry. Most of it is related rather to technical processes that needed to be performed in preparation for making the plutonium. These publications represent selections from the great mass of current reports, made on the basis of their value to basic science and technology.

The current technical reports, written during the war years, were essential to the active work of the plutonium project. They supplied needed data and calculations to those who were planning the new processes. Selecting from this mass of records the most reliable data and presenting them in a useful form has been an enormous task, for which the writers and editors of these volumes deserve the sincere thanks of their scientific colleagues. Many fields of science and technology will develop more rapidly because of this knowledge.

The efforts of the men who did this research resulted in the successful production of atomic bombs, which shortened the war and saved the lives of many of their comrades. But in the long view of history it is probable that the major human heritage from their work will not be this quick victory. It may not even be the useful applications of atomic energy, which was first presented as a Promethean gift to man. It is not unlikely that the scientific information in these pages may be the starting point to new reaches of knowledge, which will give to man an understanding that will truly enrich his life.

Arthur H. Compton

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PUBLISHER'S NOTE

Although every effort has been made to ensure accuracy in references, at the time of publication of this book some of the other volumes of the Series had not been completed. It is therefore possible that some of the references to other volumes are in error. It is hoped that the extensive cross checking which has been done in the preparation of this volume has resulted in keeping such errors to a minimum.

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INTRODUCTORY NOTE ON THE PLUTONIUM PROJECT RECORD

Organization and Record of the Metallurgical Project. The Plutonium Project Record, which forms Division IV of the National Nuclear Energy Series (NNES) is the scientific and technical record of the former Metallurgical Project. The project had its origin in work carried on in 1940-1941, mainly at Columbia and Princeton on the development of the chain-reacting pile and at the University of California at Berkeley on the production and chemistry of transuranic elements. In January 1942 this work was concentrated in the newly organized Metallurgical Laboratory at Chicago under the leadership of A. H. Compton. The Metallurgical Project grew out of the Metallurgical Laboratory. The initial objectives of the Metallurgical Laboratory were (1) to develop chain-reacting piles to produce plutonium and (2) to develop fission bombs. Major associated units were organized in 1942 at Iowa State College at Ames, Iowa (chemistry and metallurgy) under F. H. Spedding; at the University of California at Berkeley, Calif. (chemistry) under W. M. Latimer and E. D. Eastman, continuing the previous work there; and at Massachusetts Institute of Technology (metallurgy) under J. Chipman and later M. Cohen. Early in 1943 the work on fission bombs was transferred to an independent project at Los Alamos.

After the successful demonstration of a nuclear chain reaction in the West Stands pile at Chicago in December 1942, the Argonne Laboratory with its experimental pile was built west of Chicago, and the Clinton Laboratories with their pilot-plant pile were built at Oak Ridge, Tenn. — both in 1943. The three major laboratories at Chicago, Argonne, and Clinton, the associated laboratories at Ames, Berkeley, and M.I.T., and some seventy other cooperating groups then constituted the Metallurgical Project, under A. H. Compton as Project Director. Closely cooperating in the transition from laboratory and pilot-plant to large-scale operation was E. I. du Pont de Nemours & Company, which was made responsible for the design and construction of the Clinton pile and for the design, construction, and operation of the Hanford Plutonium Plant. The Project continued as such until June 30, 1945, when it was dissolved.

The Plutonium Project Record (PPR) covers most of the scientific and technical work of the Metallurgical Laboratory and the Metallurgical Project up to the date of the dissolution of the Project, and also

the continuation of this work in the successor laboratories up to approximately Jan. 1, 1946, or in some cases to a later date. In addition, the PPR covers in part the pre-1942 work at Columbia, Princeton, and Berkeley. The record of the work directly leading up to the Los Alamos Project, however, is omitted. Nevertheless the PPR and the Los Alamos Technical Series (Division V of the NNES) cover closely related and in part overlapping subject matter in some of their volumes, particularly in nuclear physics and in chemistry and metallurgy of plutonium.

Important phases of the work of the Metallurgical Project that are not reported in the PPR but will be reported elsewhere in the NNES are as follows: (1) Division VII, the report of the Materials Procurement Project, includes certain early work on process metallurgy, which was initiated largely by the Metallurgical Laboratory. (2) The Division VIII NNES volumes on Analytical Chemistry, which developed from two volumes originally planned as part of the PPR, contain much Metallurgical Project work, including one complete Collected Papers volume. (3) The Division VIII NNES volumes on Uranium Chemistry, which were planned and carried out under the supervision of the PPR editorial group, likewise contain much Metallurgical Project work, including one complete Collected Papers volume.

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History and Plan of the Plutonium Project Record. During the war years the scientific and technical work of the Metallurgical Project and its associated laboratories was described currently in a series of reports called the "C reports." The work up to July 1, 1945 was described in some 3,000 reports. After that date the Clinton Laboratories reports became a separate series, but reports of the other units of the former Metallurgical Project continued to be issued as C reports. Most of the C reports were preliminary or semifinal reports. The main consideration during the wartime development was speed of issue and distribution.

As the mass of scientific and technical knowledge obtained on the Project piled up, an increasing need was apparent for its digestion into survey or summary form. In partial answer to this need, an editorial group was set up in the spring of 1943 to organize a Project Handbook. Although never fully completed because of the engrossment of authors in immediately urgent tasks, and because of the transfer of many of them to other sites, enough of the Project Handbook was finished to be of real value.

By the summer of 1944, the Metallurgical Project had largely concluded its major task, that of providing the scientific and pilot-plant know-how for the design of the large-scale Hanford Plutonium Plant. The time seemed ripe to plan a series of volumes in which the Project's fund of accumulated scientific and technical knowledge would be

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recorded. These would replace the often sketchy and sometimes mutually contradictory C reports and fill many gaps of unwritten knowledge. In the early planning, Laurence L. Quill as Chief of the Editorial Section of the Project Information Division during the summer of 1944, Eugene Rabinowitch, and H. H. Goldsmith made important contributions. After several committee meetings, a plan for the preparation of a Metallurgical Project Record was approved by the Project Director in the fall of 1944. Later, in 1945, the name was changed to Plutonium Project Report or Record (PPR).

When the PPR was organized, rigid compartmentation was still in effect between the Metallurgical Project and the other Manhattan District projects. Members of each project were in general not supposed to know even the major objectives or main outlines of the other projects. The PPR had therefore to be planned as an independent entity. Nevertheless, at its inception the idea was firmly held that later on the Record should become part of a larger series covering the work of all the atomic energy projects. This idea was repeatedly advocated and led in late 1945 to the plan for the Manhattan Project Technical Series (MPTS), a name which was finally revised to the present designation of National Nuclear Energy Series (NNES).

The general plan of organization of the PPR was that of a series of some twenty Survey volumes, called "A volumes," each documented by a like-numbered Collected Papers volume (or volumes); these were called "B volumes." In general, following somewhat a pattern set by the Project Handbook, a Survey volume was planned for each scientific or technical subject to which the Metallurgical Project had made sufficiently major contributions. Each Survey volume was intended to be a fairly complete review or monograph (or else a collection of review chapters) on the subject field. It was planned to cover work done both within and outside the Metallurgical Project, though with primary emphasis on the former, outside work being included only for the sake of accuracy and completeness.

In contrast to the Survey volumes, each Collected Papers volume was designed to consist of individual papers, mostly from individual laboratories and more or less similar to articles in the scientific journals; they were to include only work done within the Project. In planning the PPR, it was realized that some of the Survey volumes would overlap with possible volumes of other projects, but because of compartmentation restrictions, it was decided to proceed in general with the plan as outlined. An exception was the field of uranium chemistry, where it was obvious that all the major projects were making important contributions. In this field, a Handbook of Uranium Chemistry was planned early in 1944, to be edited and written at the Metallurgical Laboratory at Chicago, but as a cooperative effort of all the

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projects, and based on a full interchange of information among them. When the Record was organized, this volume was tentatively included as one of the PPR Survey volumes, to be accompanied by a corresponding Collected Papers volume covering Metallurgical Project work only. Later, when the MPTS (now NNES) was organized, these volumes, with the addition of Collected Papers from the other projects, were transferred to the over-all Division (Division VIII) of the technical series. In the field of analytical chemistry, a Survey volume and a Collected Papers volume were planned for the PPR and were well on their way toward completion. When the MPTS was organized, the content of these volumes was pooled with the work of other projects of the Manhattan District to form Survey and Collected Papers volumes of Division VIII of the MPTS. In certain other fields, pooling of material from the different projects was also considered but was felt to involve too large a task of reorganization.

Because of the wide variety of subject matter, the organization of the PPR into Survey volumes, each accompanied by one or more Collected Papers volumes, is not always consistently followed. There are a few Collected Papers volumes without corresponding Survey volumes, and the converse is also true. Furthermore, the form of organization varies considerably from one volume to another because of varying subject matter and the preferences of the different volume editors and committees.

When the PPR plans were approved toward the end of 1944, the completion deadline for the manuscripts was set for June 30, 1945, the date of dissolution of the Metallurgical Project. Most of the PPR volumes were organized into three groups: (1) chemistry and metallurgy; (2) physics and related engineering; (3) biology and medicine. The first task was to obtain volume editors and editorial committees for the various volumes, to plan the contents, and to find authors. John C. Warner, as chemistry editor of the PPR and Chief of the Editorial Section of the Project Information Division from December 1944 to June 30, 1945, made decisive contributions to the chemistry and metallurgy volumes and to the general planning of the PPR.

The organization of the volumes on physics and on biology and medicine went more slowly, partly because the subject matter was then less ripe for writing than was that on chemistry and metallurgy, partly because of the demands for continuing research and, in the field of instrumentation, for production of instruments to be used at Los Alamos, Hanford, and other sites. Eugene P. Wigner, Frederick Seitz, and H. H. Goldsmith took an active part in the early organization of the physics volumes. Plans for the volumes on biology and medicine were very effectively organized by Raymond E. Zirkle as PPR editor for these fields, with the backing of Robert S. Stone as Associate

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Project Director for Health. Hoylande D. Young entered the PPR program as Technical Editor in charge of final editing and processing of manuscripts, and after June 30, 1946, became General Editor.

After the organization of the PPR, steady progress was made in the work of writing and editing, but at a slower pace than was originally hoped. The dissolution of the Project on June 30, 1945, with the readjustments and administrative problems involved in a 50 per cent cut of total personnel; the end of the war after the bomb was dropped in August and the subsequent deep preoccupation and extensive activities of Project personnel in connection with the social and political implications of atomic energy and atomic warfare; new research and planning directed toward the postwar continuation of the atomic energy program; all these slowed the progress of the PPR writing program. During this difficult period, invaluable encouragement and support of the PPR program came from, among others, Norman Hilberry, Associate Director of the Metallurgical Project up to the time of its dissolution, and Farrington Daniels, Director of the Metallurgical Laboratory in 1945-1946.

Meantime, other projects in the Manhattan District group began the preparation of final accounts of their work. In particular, the Los Alamos Technical Series was begun in 1945. Finally, the MPTS (now the NNES) was organized under the Manhattan District Editorial Advisory Board late in 1945. Under the chairmanship of Alberto F. Thompson, as Chief of the Publications Section of the Research Division of the District, this group began the task of coordinating existing writing activities and filling the gaps in these, with the objective of producing a reasonably well-rounded series of volumes covering the work of the entire District. During early 1946, rules for declassification were set up, and the editors of the MPTS volumes faced the difficult task of dividing the subject matter of their volumes into declassifiable parts, publishable immediately, and classified parts, for which publication must be deferred. In June 1947 the completion of the editorial work of the PPR, as part of the NNES, was taken over by the Technical Information Division of the Atomic Energy Commission, at Oak Ridge, Tenn.

In addition to those named above, many other project members worked together in planning the PPR. After the general plans were made, the actual work of preparing the various volumes was in the hands of the volume editors, volume editorial committees, and authors, as described in the prefaces of the individual volumes.

Robert S. Mulliken
Editor-in-Chief,
Plutonium Project Record

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[The page contains several lines of handwritten notes in cursive script, which are mostly illegible due to blurring.]

VOLUME EDITOR'S PREFACE

This volume reports the most significant part of the work on the metallurgy of uranium and its alloys which was carried out during World War II in the various laboratories of the Manhattan Project. Although much research in this field has continued since the war, this account serves to emphasize how little was known about the metallurgy of this element before the war and how rapidly knowledge can be accumulated by American scientists and engineers in times of emergency.

It is to be regretted that so much time elapsed before this material was put in shape for final publication. Most chapters were written in draft form in the latter days of the Manhattan Project. However, extensive revisions became necessary because of changes in the plans for the Project Record. By this time the authors were widely scattered and were burdened with other duties. Much credit for the final completion of the volume goes to Dr. Hoylande Young for her persistence and help.

J. C. Warner

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CONTENTS

Page

Foreword	7
Preface	9
Plutonium Project Record Foreword	13
Introductory Note on the Plutonium Project Record	15
Volume Editor's Preface	21

CHAPTER 1

Small-scale Production, Melting, and Casting of Uranium	25
By J. C. Warner	

CHAPTER 2

Fabrication of Uranium	42
By H. W. Russell, H. R. Nelson, J. M. Simmons, and A. Van Echo	

CHAPTER 3

The Physical and Mechanical Properties of Uranium	73
By Frank G. Foote	

CHAPTER 4

Metallographic Technique for the Study of Uranium and Its Alloys	114
By Frank G. Foote	

CHAPTER 5

Binary Alloys of Uranium	118
By R. W. Buzzard and H. E. Cleaves	

CHAPTER 6

The Mechanical Properties of Alloys of Uranium	153
By A. B. Greninger	

Index	205
-----------------	-----

SECRET

1. 1990년대 초반부터 시작된 '신자유주의' 정책의 영향으로, 국내 경제는 급속도로 개방화되고, 외국 자본의 유입이 증가하였다. 이로 인해 국내 산업은 경쟁력을 잃어가고, 실업률이 상승하는 등 경제 위기를 겪었다.

2. 1997년 IMF(IMF)에 가입한 이후, 정부는 IMF의 요구에 따라 경제 구조조정을 실시하였다. 이는 대규모 기업 구조조정, 노동 유연화, 공공부문 개혁 등을 포함하였다. 그러나 이러한 조치들은 사회적 불평등을 심화시키고, 국민들의 생활고를 악화시켰다.

3. 1998년 발생한 아시아 외환 위기(Asian Financial Crisis)는 한국 경제에 심각한 타격을 주었다. 이로 인해 수출이 급감하고, 외환보유고가 바닥나자 IMF에 가입하게 되었다. IMF의 처방에 따라 실시된 구조조정은 경제를 안정시켰지만, 사회적 갈등을 심화시켰다.

4. 1990년대 후반부터 2000년대 초반까지는 '신자유주의' 정책의 영향으로, 국내 경제는 급속도로 개방화되고, 외국 자본의 유입이 증가하였다. 이로 인해 국내 산업은 경쟁력을 잃어가고, 실업률이 상승하는 등 경제 위기를 겪었다.

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Chapter 1

SMALL-SCALE PRODUCTION, MELTING, AND CASTING OF URANIUM

By J. C. Warner

The large-scale commercial production of uranium by the reduction of uranium tetrafluoride with magnesium in refractory-lined steel bombs and the foundry practice for remelting the bomb-produced massive metal and casting it into ingots are described elsewhere in the National Nuclear Energy Series.¹

A historical account of the work on the preparation of metallic uranium leading up to the present large-scale production methods has also been prepared.² This account covers the investigations made from the time the element was discovered up through the early days of the Manhattan Project.

This chapter gives an account of various small-scale experiments which were conducted on the Project for the purposes of (1) producing exceptionally pure uranium, (2) casting shapes from uranium with precision, and (3) using uranium as a stand-in for the development of successful small-scale production methods for other important active metals.

1. PRELIMINARY EXPERIMENTS ON THE PRODUCTION OF URANIUM METAL ON THE MICROGRAM TO MILLIGRAM SCALE

In an effort to develop a method whereby microgram and milligram amounts of active metals could be produced from their compounds, the reduction of UF_4 by sodium and potassium was attempted on this scale. A pyrex apparatus was designed in which UF_4 could be brought into reaction with sodium, potassium, or other alkali metals that could be distilled in an evacuated glass system. Essentially the apparatus consisted of a series of glass bulbs that permitted the repeated distillation

SECRET

of the alkali metal for purification and its final distillation into a capillary tube containing the small sample of the uranium salt to be reduced. After the reaction between the uranium salt and the alkali metal had taken place, the excess of alkali metal was distilled away, and the glass capillary tube was sealed off under vacuum. The resulting product within the glass tube could be examined microscopically and later could be removed for chemical tests. Many samples of UF_4 , ranging from 1 to 50 μg in size, were reacted with sodium in this apparatus to produce a black powder that dissolved in HCl with the liberation of hydrogen. On the basis of this evidence, the product was believed to be uranium metal, but this conclusion was never established with certainty.

In order to carry out the reduction at higher temperatures, the pyrex apparatus was replaced by a very small refractory crucible which contained both the halide to be reduced and the active-metal reductant. The charged crucible, covered with a tightly fitting lid, was placed within a tungsten-wire resistance furnace, the tungsten wire being coated with BeO or ThO_2 as a conducting refractory. The furnace with its crucible and contents was heated to about 1500°C in vacuo, whereupon the reducing agent vaporized, reducing the metal halide, and the excess reductant was volatilized through the pores of the crucible. By the use of refractory crucibles of BeO and this technique, it was possible to reduce small quantities (1 to 50 μg) of UF_4 , ThF_4 , LaF_3 , CeF_3 , Fe_2O_3 , and MnO_2 to the corresponding compact metals, using Mg , Ca , Ba , and Li as reducing agents. The form of the metals produced indicated that solidification from the molten state had occurred.

Although these experiments were interesting as an indication that active metals could be produced on a very small scale, the product metal was not obtained in sufficient quantity to give reliable results concerning yields and purity. As a consequence, experiments were next undertaken on a somewhat larger scale (50 mg to 1 g) with the object of obtaining a sound, massive metal as the product in sufficient quantity that yields could be more accurately determined and that adequate analyses of the metal could be made. To accomplish this purpose, it seemed evident that the reduction should be carried out at such a temperature that both metal and slag would be liquid and that heat losses from the reaction zone should be slow enough to permit the separation of metal and slag and the collection of metal into a single liquid mass before solidification occurred.

It was recognized that data on the thermochemistry of the reduction of uranium halides by active metals would be a useful guide for this work; therefore a comprehensive review of the subject was prepared.³

SECRET

Many of these reduction reactions are sufficiently exothermic that, once brought to the ignition temperature by some means, the heat of reaction is sufficient to bring metal and slag into the liquid state if the reaction is carried out in an isolated system or on a large scale. These same reactions, when carried out on a small scale, are subject to so much heat loss to the refractory and surroundings that auxiliary heat must be supplied to keep the products molten so that the massive metal will be produced. This need for auxiliary heat increases, of course, as the specific reduction reaction becomes less exothermic and as the melting points of the reaction products increase.

In the experiments which are described here, additional heat has been supplied by one or both of the following methods: (1) external heating of the reaction bomb by induction (this is accomplished either by using a resistance furnace surrounding the bomb or by placing the bomb in a gas-fired furnace) or (2) by the use of an excess of the active-metal reductant and the addition of a "booster" to the reaction mixture—a substance (iodine, potassium chlorate, etc.) which undergoes very exothermic reaction with the excess reductant and adds no undesirable impurity to the metal.

Other things being equal, high purity, high yields, and good aggregation of metal become more difficult to obtain as the size of operation is decreased, and there is a corresponding increase in the surface-to-volume ratio of the charge. One idea which was very successful in improving the yield and quality of metal in small-scale reductions was that of carrying out the reduction while the bomb was being rotated rapidly in a graphite centrifuge. This method and some of the results accomplished thereby are described in Sec. 2.

2. BOMB REDUCTIONS IN THE GRAPHITE CENTRIFUGE ON THE 50-MG TO 1-G SCALE

The reduction of uranium halides by active metals on a small scale in the graphite centrifuge was first carried out successfully in the Metallurgical Laboratory at the University of Chicago;⁴ later the method was applied extensively at other Project laboratories.

As has been previously indicated, in a reaction of the thermite type the yield in a coherent metal mass and the quality of the metal produced improve as the scale of operation is increased. Not only is the surface-to-volume ratio of the charge less, but there is proportionally less loss of heat to satisfy the heat capacity of the refractory liner and the bomb and less conduction to the surroundings. This allows the reaction to reach higher temperatures and cool less rapidly and affords more time for the collection of the metal into a single button.

SECRET

Better yields can be obtained if sufficient external heat or a booster is provided to maintain the reaction mass molten for prolonged periods and, if gravity is aided by centrifugal force, to cause aggregation and collection of the small droplets of metal. In the method being described the use of centrifugal force to throw down the molten metal into the tip of a cone was successful even in very small-scale reductions. This was accomplished by placing the reaction mixture in a cone-shaped refractory liner sealed inside a steel bomb placed in a graphite rotor which was heated rapidly to a high temperature while rotating. As reduction took place, the metal was thrown together in the tip of the refractory liner, thus producing a good yield of coherent metal. Application of this technique has given successful reductions even on the 50-mg scale.

The apparatus consisted of a graphite rotor, 6 in. in diameter, rotated inside a high-frequency coil. The lower end of the rotor shaft rested on a water-cooled ball bearing, and the upper end had a slot cut in it to permit its being driven by a pin and socket on a simple drill press. The rotor was constructed with four slots, 90 deg apart, which could hold four steel bombs containing the liner and reactants. When less than four reductions were made at one time, the rotor was balanced with dummy bombs. The loaded bombs were packed into the rotor with MgO, and this procedure prevented attack by the graphite on the steel bombs at high temperatures.

The charge was placed in a cone-shaped BeO crucible with the uranium or other halide on top, covering the reducing metal (Ca or Li). The crucible, covered with a lid of MgO, was placed inside the steel bomb, and the apparatus was then sealed by arc welding. To prevent heating of the charge and premature ignition, the body of the bomb was held in a water-cooled clamp in such a manner that only the actual edge to be welded was exposed. After the bomb had been packed tightly with MgO in the graphite rotor, the loaded rotor was placed inside a high-frequency coil fed by a 50-kw 3000-cycle 400-volt generator. A speed of 900 rpm (centrifugal force about 50 G) was used.

The temperature cycle during a reduction was an important variable. Rate of heating, maximum temperature achieved, time at maximum temperature, and rate of cooling all needed to be controlled, and optimum practice for each scale of operation for each specific reduction reaction had to be determined. It was found that temperature control could be accomplished by sighting an optical pyrometer on the hottest part of the rim of the rotor. These temperatures could then be correlated to the temperature in the bomb.

Some reductions on the 50-mg scale were made in a similar centrifuge about one-third the size of the one already described, with the

SECRET

speed of rotation increased to give an equal centrifugal force. However, it was found that more satisfactory results could be obtained on this small scale by using the large centrifuge, placing the small charge in the tip of the regular BeO cone, and placing a lid immediately above the charge. In practically all reductions the air inside the bomb was displaced with argon before the lid was welded on.

The reaction mixture for the 1-g-scale reductions consisted of enough halide to produce 1 g of metal, plus one of the reducing agents, Ca, Li, etc. When iodine was used as a booster, it was usually added in the ratio of 1 mole of iodine per 3 moles of halide. Enough reducing agent was added to give an over-all excess of about 20 per cent. Figure 1.1 is a longitudinal cross section of a bomb fired in the graphite centrifuge. This particular specimen is far from the best, but it clearly shows the layer of slag on top of the button of uranium metal in the tip of the cone-shaped crucible.

Approximately 250 reductions were made using uranium halides and various reducing agents. Uranium tetrafluoride, UF_4 , reduced with Ca plus I_2 as a booster always produced high yields of brittle metal which contained considerable amounts of entrapped slag and which had a high iron content. Uranium tetrafluoride reduced with lithium, on the other hand, gave very malleable metal, with yields of 94 to 99 per cent and a much lower slag and iron content. When Li was used, it was not necessary to add I_2 as a booster because of the lower melting point of the LiF slag compared to that of the CaF_2 slag. Uranium trifluoride, UF_3 , was successfully reduced with Li, and UCl_3 was reduced with Ca, Ba, or Li. Some reductions were done with mixed uranium and manganese halides to give a 95-to-5 uranium-manganese alloy with a melting point in the range 700 to 800°C. These mixed fluorides were satisfactorily reduced with Li or Ca; the chlorides, with Li, Ca, and Na.

To determine the general applicability of the graphite centrifuge, $NdCl_3$ and $CeCl_3$ were successfully reduced with Ca, and $LaCl_3$ and MnF_2 were reduced with Li. For small-scale reduction, the centrifuge method is much less sensitive to the small variations which may cause complete failure when the stationary-bomb method is used.

Although later developments showed that reductions on a somewhat larger scale could be carried out more economically and in an entirely satisfactory manner in stationary bombs, the graphite-centrifuge method played an extremely important part in the development of reduction methods to produce quite pure metal on a small scale. This method remains the most satisfactory one for reductions on the 50-mg to 1-g scale.

SECRET

Fig. 1.1—Longitudinal cross section of a bomb fired in the graphite centrifuge.

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3. REDUCTIONS IN STATIONARY BOMBS ON THE 1- TO 250-G SCALE

After the process for the production of a satisfactory grade of commercial uranium metal by the bomb reduction of UF_4 with magnesium was in successful operation, the need arose for producing at least small quantities of uranium of a considerably higher order of purity than was produced in the commercial process. Uranium free from, or containing extremely low concentrations of, certain light elements was especially desired. At this same time there was considerable interest in the development of a satisfactory method for the production of other active metals on a small scale. As a consequence several groups of investigators began studies to determine whether the stationary-bomb method, so successful for the large-scale production of uranium, could be scaled down and so modified as to produce small amounts of especially pure metal.

Significant results were obtained by Spedding, Wilhelm, Keller, and others at Iowa State College;⁵ by Derge and Martin at the Metallurgical Laboratory, University of Chicago;⁶ by Kraus and his coworkers at Brown University;⁷ and by Smith, Jette, and their associates at the Los Alamos Scientific Laboratory.⁸ Altogether these investigations covered studies of the effects of those variables which might be expected to influence the yield, coherence, and purity of the metal:

(1) The influence of the composition; the density as determined by particle size, method of formation, and prefiring; and the shape and the thickness of the refractory bomb liners were investigated. (2) The nature of the uranium halide: UF_4 , UF_3 , UCl_5 , UCl_4 , UCl_3 , UBr_3 , and UBr_4 were used as compounds to be reduced. Furthermore, the effects of particle size, apparent density, moisture content, and oxygen content of the halides were studied. (3) The nature of the active-metal reductant was studied (in these experiments Ca, Ba, Mg, Sr, Li, and Na were used), and for most of these metals the particle size and oxide content were studied as variables. (4) Various designs of steel and graphite bombs were tried, including different closures and various procedures for filling the bomb. (5) Ignition temperatures for various reaction mixtures were determined. (6) Heating cycles were investigated to determine the optimum cycle for a particular type of reduction. (7) The use of booster additions alone, or along with the application of external heat, was the subject of much study.

The original reports and papers which are given in the references must be consulted for the details of these investigations. It seems a safe conclusion, however, that any of the uranium halides can be re-

SECRET

Fig. 1.2—Design of the steel bombs and magnesia liners used in reductions of uranium tetrafluoride and uranium trichloride.

duced to metal of good purity on the 1- to 25-g scale with high yields if the optimum procedure for the particular reduction mixture is followed.

The reduction of uranium tetrafluoride and uranium trichloride with calcium metal were probably more thoroughly investigated than

Table 1.1—Typical Analyses of Uranium Produced by Stationary-bomb Reductions of the 100-g Scale*

Impurity	Sample 1	Sample 2
Mg	<10	10
B	<0.8	0.72
Cd	<0.2	<0.2
Be	<0.2	<0.05
P	<5	<10
Fe	90	80
Mn	6	2

*Analysis of uranium-metal biscuits produced by reduction of UF_4 (production grade) with calcium (production grade) using an I_2 (USP grade) booster. Values are in parts per million.

Table 1.2—Analysis of Uranium Produced by Stationary-bomb Reductions*

Impurity	Sample No.				
	1	2	3	4	5
F	2.7				
B	4	4	0.77	0.79	1.2
Si	50	20	1.08	72	74

*Analysis of cast uranium metal produced by reduction of UBr_4 with calcium (reduction grade) using an iodine booster. Vacuum-cast metal. Values are in parts per million.

any others. Successful techniques were worked out by which uranium of high purity could be prepared with yields of 90 to 99 per cent in these reductions on the 1-, 10-, 25-, and 250-g scales. The design of the steel bomb and the dense, highly fired magnesia liners used in these reductions is shown in Fig. 1.2.

Although reductions of the uranium bromides, UBr_3 and UBr_4 , were not extensively investigated before the program was discontinued,

SECRET

these reductions showed great promise. Without extensive study to determine optimum conditions, it was possible to obtain uranium of good purity in yields of 91 to 95 per cent by the stationary-bomb reduction of distilled UBr_4 with 30 per cent excess calcium and 0.67 mole of I_2 per mole of UBr_4 added to the reaction mixture as a booster. Bomb liners of either sintered CaO or MgO were used. Some typical analyses of good-quality uranium produced in stationary-bomb reductions on the 100-g scale are given in Tables 1.1 and 1.2.

4. REDUCTIONS IN OPEN CRUCIBLES IN INERT ATMOSPHERES

Eastman and his associates at the University of California, Berkeley, showed that uranium could be produced with high yields on the 1- to 50-g scale by carrying out reductions in open crucibles in inert atmospheres.⁹ Although work on this method was discontinued at the close of the World War II, it showed considerable promise as a means of producing uranium substantially free from light-element impurities.

This general method was first applied to the preparation of cerium metal,¹⁰ and the same apparatus was used for carrying out the uranium reductions. Reductions were carried out in molybdenum crucibles using helium as an inert atmosphere. Uranium trichloride was reduced with calcium on the 4- to 50-g scale with yields of compact metal ranging from 99.4 to 99.9 per cent, the product metal containing no more than about 0.10 per cent calcium. Small-scale reductions could be carried out by adding all the uranium trichloride to the crucible, then slowly adding calcium at about 900°C , and finally heating the reaction mixture to 1200°C to melt the uranium and produce a compact ingot. Larger scale reactions were carried out successfully by either of two procedures: (1) Both UCl_3 and Ca were added slowly to prevent overheating and consequent volatilization and spattering. (2) All the UCl_3 was placed in the crucible, but the Ca was diluted by fusing it with CaCl_2 before use.

Uranium trichloride also was reduced with magnesium with good yields, but magnesium is not so satisfactory a reducing agent as calcium because of its higher volatility and the fact that higher concentrations of the reductant (5 to 7 per cent) remain in the uranium metal.

Uranium oxide, U_3O_8 , was reduced with calcium on the 1- to 10-g scale. When pure Ca was used, the reduction was quite violent and much of the uranium was produced in a finely divided condition. However, successful reductions could be carried out using Ca diluted with CaCl_2 . With this procedure, yields of compact uranium in the range 92 to 99 per cent were obtained.

SECRET

It also was demonstrated that high yields of compact metal could be obtained by reducing mixtures of UCl_3 and U_3O_8 with Ca diluted with CaCl_2 .

5. THE HOT-WIRE METHOD APPLIED TO THE PRODUCTION OF URANIUM METAL

The successful use of the hot-wire method for producing such metals as Ti, Zr, Hf, Th, V, Nb, Ta, Cr, W, Mo, Cu, Fe, Pt, and Rh in a state of extremely high purity¹¹ led Chipman, Derge, Magel, Fine, and their associates at the Metallurgical Laboratory, University of Chicago,¹² to investigate this method and modifications thereof for the production of especially pure uranium. Although the project was discontinued shortly after this work was initiated, some very promising results were obtained, and it seems certain that the method could be developed to produce small amounts of very pure uranium.

In essence the hot-wire process may be described as follows: A volatile compound of the metal, usually a halide, is passed over a heated filament, where the compound is thermally decomposed to form metal. The metal is deposited, and, when halides are used, the halogen is recirculated to form more volatile metal halide for decomposition. Usually the volatile halide is made by reacting the halogen with a commercial grade of the metal. In the conventional processes the filament is operated at a temperature below the melting point of the metal, and the latter is deposited on the filament, which grows into a rod. Elaborate control is required to maintain the hot surface at a constant temperature. A filament of the metal being deposited is used when possible. The apparatus may be designed so that the halogen forms the volatile halide by diffusing to the surface of the crude metal contained in a low-temperature zone, or the halogen, with or without a carrier gas, may be circulated in a flow system.

Available information on the chemistry of uranium indicated that uranium tetraiodide, UI_4 , was sufficiently volatile and that, under proper conditions, it could be decomposed to metallic uranium and iodine. The following system therefore was selected for study in a flow apparatus: Hydrogen, purified by passage through a palladium thimble, and iodine vapor were brought at controlled rates of flow into contact with commercial uranium metal at about 500°C in the low-temperature end of a quartz reactor tube. UI_4 formed and volatilized from the metal and was then passed over a hot filament operated at about 1500°C . The filament consisted of a tungsten coil made from 0.010-in.-diameter wire covered with a uniform layer of electrically fused thoria which had been fired to about 2200°C before

SECRET

use. Since the filament was operated above the melting point of uranium, molten metal was produced which collected in a crucible in a side arm under the filament.

Although a number of variables were explored, the work was discontinued before optimum conditions for operation were discovered. Nevertheless, a number of successful experiments were made in which 50 per cent of the commercial metal (3 to 4 g) was recovered as purified metal, and these samples were subjected to metallographic examination and chemical and spectrographic analyses. Nonmetallic inclusions were present in the purified metal, but these samples were much cleaner than commercial metal, and no identifiable metallic phase except that of uranium was observed. Spectrographic analyses of purified metal showed that all metallic impurities were absent down to the sensitivity of available analytical methods. Carbon was still present to the extent of 55 to 140 ppm, about one-half its concentration in commercial metal. It seems likely that a further reduction in the carbon content could have been achieved by a further investigation of the process. The oxygen content by vacuum-fusion analysis was in the range 0.009 to 0.02 per cent, but this was dependent on the surface-to-mass ratio of the sample taken. It seems likely that the most oxygen was present as surface oxide and that the true oxygen content of the bulk metal was very low.

A further study of important variables in the process and of modifications of the process was under way when the project was abandoned. The references cited must be consulted for a report on these unfinished investigations.

6. SMALL-SCALE PRODUCTION OF URANIUM BY ELECTROLYSIS OF FUSED SALTS

The general success achieved in the production of active metals by the electrolysis of fused salts led early investigators to study the applicability of this method to the production of uranium metal. Early work on this method and the successful production of uranium by the electrolysis of potassium uranium fluoride or uranium tetrafluoride dissolved in a fused mixture of 80 per cent CaCl_2 and 20 per cent NaCl in the Westinghouse process have been described elsewhere.² The success of this process led to a number of investigations in an attempt to devise a still better electrolytic process for metal production on a commercial scale. However, the rapid development and outstanding success of the bomb-reduction process caused electrolytic investigations to be abandoned. Later, when the search was on for methods for producing especially pure uranium on a small scale, interest was renewed in electrolytic processes, especially by Eastman and his

SECRET

associates¹³ at the University of California and by Smith, Jette, and coworkers at the Los Alamos Scientific Laboratory. The latter were able to obtain 50 per cent recoveries of metal on the 50-mg scale by the electrolysis of uranium trichloride, UCl_3 , in a fused mixture of 48 per cent BaCl_2 , 31 per cent KCl , and 21 per cent NaCl in small pyrex cells using an inert atmosphere of purified hydrogen or argon, an inert anode such as platinum, and a cathode of tungsten upon which the uranium was deposited.

Eastman's group studied the electrolytic production of uranium from solutions of uranium tribromide, UBr_3 , or uranium triiodide, UI_3 , in molten CaCl_2 , SrBr_2 , SrI_2 , BaI_2 , and mixtures of SrBr_2 and SrI_2 . Electrolyses were carried out in a helium atmosphere in the temperature range 500 to 770°C. Various electrodes and container materials were studied, the cathode being used as the container. On the 1- to 5-g scale, as much as 40 per cent of the uranium added to the bath could be recovered as a good crystalline cathodic deposit using solutions of UBr_3 or UI_3 in SrI_2 or mixtures of SrI_2 and SrBr_2 with iron or molybdenum cathode containers and strontium or tungsten anodes. When strontium anodes were used, the cell reaction was sufficiently spontaneous that electrolysis proceeded without the application of an external emf.

These investigators also found that uranium could be refined using uranium anodes with an electrolyte of UBr_3 dissolved in SrBr_2 . Current efficiencies of 53 per cent were obtained with cathodic current densities of 10 to 40 amp/cm² and temperatures in the range 680 to 700°C.

7. SMALL-SCALE REMELTING AND CASTING OF URANIUM IN SPECIAL REFRACTORIES

Uranium metal produced by any of the methods already described, except the hot-wire method, although intrinsically quite pure and in a coherent mass, contains slag or salts on its surface and as inclusions. Metal produced by one of the bomb processes usually contains some residual active-metal reductant. As a consequence considerable purification is accomplished by remelting the metal in vacuo or under an inert atmosphere to allow a separation of slag and a removal of various volatile impurities including the residual active-metal reductant. The molten metal is then cast to form an ingot.

In the large-scale production of uranium metal, the biscuit metal produced in the bomb-reduction process is remelted in vacuo in graphite crucibles and is cast into ingots in graphite molds. Considerable purification is accomplished in this process by removing

SECRET

the entrapped slag and volatilizing the residual magnesium. Although carbon pickup is substantial, it is kept within ranges which can be tolerated because of the relatively small surface-to-mass ratio for the metal being remelted and cast. Graphite is unsuitable as a refractory for the vacuum-melting and -casting of the metal produced in any of the small-scale experiments described in previous sections of the chapter because of the large ratio of surface to mass. Very small samples were converted completely to carbide when melting in graphite was attempted. Furthermore, it was one objective of the high-purity program to produce uranium much lower in carbon than the commercial metal.

A substantial research program was carried out on the Project for the purpose of developing refractories which could be used for melting and casting uranium in small quantities with high yields and without introducing undesirable impurities into the metal. Although the details of this work are reported in a separate volume,¹⁴ it seems proper to give an account of a few of the achievements of the program here: Spedding, Wilhelm, Gray, et al.,¹⁵ at Iowa State College, after demonstrating that high yields could be obtained in melting and casting uranium on the 50- to 100-g scale using graphite crucibles and molds, were able to remelt and cast without a carbon pickup by fitting the crucible and mold with a fired zirconia liner. These investigators also produced refractories of magnesia and calcium oxide which were fairly satisfactory for the small-scale remelting and casting of metal.

Eastman, Brewer, and coworkers at Berkeley developed some exceedingly interesting and important refractories made of cerium and thorium sulfides and subsulfides. They also carried out experiments on the remelting and casting of uranium on a small scale in these refractories.¹⁶ It was shown that the sulfur pickup by metal was very small, and they offered a means of remelting and casting without the introduction of oxygen or carbon. These sulfide refractories found much application in the remelting and casting of small amounts of high-purity metals.

The most extensive program for the production of new types of refractories and the preparation of better refractories from previously used materials was that carried on by Norton, Chipman, et al., first at the Metallurgical Laboratory, University of Chicago, and later at the Massachusetts Institute of Technology (M.I.T.). Almost all oxides that are very stable at high temperatures were investigated. In addition, this group produced refractories of the nitrides of uranium, titanium, and zirconium. The group also manufactured many refractories of the sulfides of cerium and thorium by the process discovered at Berkeley.

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8. PRECISION CASTING OF URANIUM

The final step in the metallurgy of uranium is the fabrication of the metal into pieces of a specified geometrical shape, usually with close dimensional tolerances. Applications of the conventional methods of extrusion, rolling, machining, grinding, and drawing to the fabrication of shapes from uranium ingots are discussed in Chap. 2. In some circumstances these usual methods are undesirable because of the chemical reactivity of the metal, its toxicity, and the desire to prevent the introduction of additional impurities. Because of these considerations it was thought desirable to investigate the possibilities of precision casting as an alternative to other methods of fabrication. This investigation was undertaken by Chipman, Marshall, et al. at M.I.T.¹⁷ When the Project closed, this investigation had not developed beyond small-scale experimentation, and a number of promising procedures had not been explored in any detail. Nevertheless, considerable success had been achieved.

A preliminary study led to the conclusion that the only known method for making precision castings worthy of consideration was that making use of the "lost-wax" technique. This method had been used for many years in the jewelry and dental trades for casting gold alloys and other low-melting-point metals, but more recently mold materials had been so improved that a number of high-melting-point, nonforgeable, and nonmachinable metals were being cast with precision. The method utilizes refractory molds, usually of silica, which are caused to set, in the manner of plaster of Paris, around a wax duplicate of the metal piece to be produced. When setting is complete, the wax is removed by firing in a furnace so that a cavity (which later is to be filled with metal) results in the refractory body. Careful control of the wax-pattern dimensions results in precise metal castings. In the standard practice the metal is usually cast with the aid of centrifugal force or air pressure in order that sound castings with good reproduction of fine details may be produced. For the same reason the molds are frequently used at elevated temperatures.

Several methods were investigated for making precision castings of uranium in molds prepared by the technique just described. In a parallel study a search was made for investment materials which could be used under the conditions necessary for casting, without contaminating the metal with undesirable impurities. The best results were obtained by gravity casting into an evacuated stationary mold; fairly good results were obtained in a centrifugal casting device in which a moderately good vacuum was maintained; but, melting under a barium

SECRET

chloride flux and casting in air or in an atmosphere of argon or nitrogen did not produce satisfactory results. Of a large number of investment materials studied, the simplest and most generally satisfactory was made of silica bonded with ethyl silicate.

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Chapter 2

FABRICATION OF URANIUM

By H. W. Russell, H. R. Nelson, J. M. Simmons, and A. Van Echo

1. EXTRUSION OF URANIUM

By H. W. Russell and H. R. Nelson

1.1 General Principles. Extrusion has been used extensively for the fabrication of rods of uranium. In the extrusion process, heated cylindrical billets are placed in the container of a press and are forced out through a die of suitable shape by the pressure exerted by a moving ram. This process will produce a rod of the desired size in a single operation with a minimum of exposure to the atmosphere. The extruded rod has a surface which, although not smooth, is free from seams and similar defects, but porosity at or near the center is not uncommon. Attempts to increase the yield by operating so as to leave only a thin extrusion butt in the container result in unsoundness known as the "extrusion defect."

It is common in extrusion to heat the billet to decrease its resistance to plastic flow. Uranium starts to soften at about 500°C and is significantly softer as the alpha-beta transformation at 662°C is approached. The beta phase from 662 to 772°C is relatively hard and brittle, whereas the gamma phase, above 772°C, is quite soft. Extrusion has been done at high alpha temperatures¹⁻³ and throughout the gamma range.⁴⁻⁶ The effect of temperature on the extrusion pressure is shown in Fig. 2.1. These pressures were measured in the laboratory, using a container which was maintained at the billet temperature, while extruding a $\frac{3}{8}$ -in. rod from a $1\frac{1}{4}$ -in. billet. The yield pressure is derived from the load required to start the extrusion of an uncoated billet. The extrusion pressure is the value required to maintain flow of the rod at a speed of about 9 ft/min, and it increases only slightly with increasing speed.

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Fig. 2.1—Effect of billet temperature on extrusion pressures.
(a) Alpha extrusion. (b) Gamma extrusion.

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It will be noted that the pressure for alpha extrusion even at 650°C (1200°F) is much higher than for gamma extrusion at 800°C (1470°F) or above. A few trials of alpha extrusion have been made on a commercial press, some of which were successful. The pressure available was not sufficient for practical operation.

At gamma temperatures the pressures required are within the range of the presses designed for use with aluminum. In fact, uranium is so soft that it tends to deform under its own weight. In extrusion vertically downward the rod leaves the die undersize when its length exceeds about 3 ft, whereas in horizontal extrusion, friction on the table causes upsetting; so the butt end of the rod is slightly oversize. The gamma-extrusion temperature is above that to which the container of the press can commonly be heated. With billets large enough to hold heat and a quick-acting press, however, the container need not be fully up to temperature if the billets are slightly superheated. The extrusion die is under severe conditions because it is heated practically to billet temperature by the metal flowing over it.

At high temperatures the die must have good strength and resistance to wear and chemical attack (alloying) by hot uranium. Ordinary hot-die steels alloy readily with uranium under the conditions of temperature, pressure, and flow in gamma extrusion. High-chromium steels show resistance to alloying but last for no more than four or five extrusions commercially, failing by wear. Chromium-plated die steels give similar results. A commercial hard facing material, Tantung, has been tried with very satisfactory results. Stoodite hard facing, which is equally satisfactory, has been used for all commercial extrusion.⁶⁻⁸

Dies for commercial extrusion have been made from chromium-tungsten hot-die steel containing from 10 to 15 per cent tungsten. These have been hard-faced by the application of Stoodite 63 to a thickness of $\frac{1}{32}$ to $\frac{1}{16}$ in. In early production runs such a die would extrude 200 bars about 8 ft long and finally fail by chipping out. Radial cracks appear in the facing material, but they do not seem to have any effect on the die life or the quality of the surface of the bars.⁹

A shear type die, consisting of a cylindrical hole in a flat-faced die plate, is considered most satisfactory for gamma extrusion. Such a die tends to hold back surface oxide, and the restricted flow of the hot metal over its face probably results in a lower die temperature. Differences in extrusion pressure between shear dies and flow dies are not important with the soft-gamma uranium.

The dummy block, a short cylinder interposed between the hot billet and the ram of the press, is subject to considerable hot battering

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and some alloying action by the billet. At moderate press speeds, requiring about 1 min for the extrusion of an 8-ft bar, a cast high-chromium high-carbon steel was fairly satisfactory.⁷⁻⁹ In the higher speed operation to be described, a high-tungsten hot-die steel hard-faced with Stoodite was much more satisfactory.

1.2 Commercial Extrusion Practice. Commercial extrusion of uranium has been done at several plants, using somewhat different procedures.^{10,11} A practice that was used at a plant where a large number of bars were fabricated has been selected for description. In this plant it was necessary to start production with the available equipment, or that which could be secured with a minimum of delay. It provides an example of a satisfactory, but not necessarily the best possible, procedure.

Billets 4.25 in. in diameter and 10 to 12 in. long were heated in a twin-muffle gas-fired furnace similar to that shown schematically in Fig. 2.2. For high rates of production, the furnace was about 14 ft long and about 7 ft wide and held 60 billets in the two long muffles. Each muffle extended the full length of the furnace and was 15.5 in. wide by 10.5 in. high inside. They were constructed of Carbofrax silicon-carbide slabs and rested on brickwork flues. Premixed gas and air were supplied to 14 burners through manifolds on each side of the furnace. The temperature was controlled by the action of a potentiometer controller and of thermocouples embedded in the hearth of each muffle near the exit end of the furnace.

To minimize oxidation of the billets, natural gas was introduced into each muffle through water-cooled pipes entering through the top near the middle. From 220 to 250 cu ft/hr of raw natural gas was used on the 14-ft furnace. Air entering when the furnace doors were opened caused some oxidation, but the total oxidation loss has been estimated to be not over 1 per cent.

Billets were rolled through the furnace, a slight slope from the charging end to the exit end being provided to aid in this. A continuous line of billets was maintained in each muffle, and the furnace temperature and rate of movement of the charge were adjusted so that the billets at the exit end were between 1600 and 1650°F (870 and 890°C). Billets high in iron were heated to only about 1500°F in order to avoid melting the eutectic.

The hot billets were rolled out onto a table; the oxide was brushed off; the temperature was measured with an optical pyrometer; and then the billets were transferred as rapidly as possible to the extrusion press.

A horizontal-extrusion press using hydraulic accumulators was used. The ram was capable of exerting a force of 1000 tons and had a

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SECRET

Fig. 2.2—Schematic diagram of billet-heating furnace.

stroke of 36 in. The extrusion cylinder was about 15 in. long and about 24 in. O.D. Replaceable liners of hot-die steel (0.35 wt. % carbon, 4 wt. % chromium, 9 to 10.5 wt. % tungsten, and 0.25 wt. % vanadium) initially $4\frac{5}{8}$ in. I.D. were fitted inside the container. The temperature of the liner was maintained at from 900 to 950°F (480 to 510°C) by heating the container externally with gas. After each billet was pushed, the layer of metal and oxide was cleaned out with a rotary wire brush. Some scoring occurred, and, after 1500 to 2000 pushes, it was necessary to rebore the liner, which increased its diameter by $\frac{1}{8}$ in.

Dummy blocks were 2.5 in. thick but were relieved to present only 1 in. of bearing surface to the liner. The diameter corresponded to that of the liner, with a 0.005- to 0.010-in. clearance. They were made of the same die steel as the liners and were hard-faced with $\frac{1}{16}$ in. of Stoodite 6 on the face and sides. The blocks were heat-treated to 45 to 50 Rockwell C, hard-faced, and then stress-relieved at 1200°F (650°C). Such a dummy block would serve for about 600 pushes on the average.

The dies were of hot-die steel, containing about 15 wt. % tungsten, heat-treated and hard-faced with $\frac{1}{10}$ in. of Stoodite 63. The die was 1 in. thick. To obtain a rod of 1.44 in. in diameter, the opening was made 0.030 to 0.050 in. larger. There were a $\frac{1}{8}$ -in. bearing and a 7-deg relief angle. The shear edge was relieved by a $\frac{1}{32}$ -in. radius. The die was removed and cleaned after 75 pushes and had a total life of several hundred extrusions.

Extrusion was done at the maximum speed of the press, requiring 5 to 6 sec, and the bar left the die at a speed of about 1.5 ft/sec. To obtain the maximum yield of rod, all billets were extruded until only a $\frac{1}{2}$ -in. butt remained.

The pressure required for extrusion varied somewhat, depending on the temperature and type of billet, but averaged 19,000 psi of billet area. Just before extrusion was stopped, the pressure rose to approximately 30,000 psi.

The extruded bars were run out by the press onto a table, were cut off with a hydraulic shear, and were straightened by hand while hot. Four bars were kept on the table, the coolest being quenched in water on reaching a black heat, estimated to be about 900°F (480°C). Cold straightening was done in a roller straightener. The as-quenched bars were commonly straight enough to permit outgassing and were roller-straightened after this operation.

1.3 Quality of Extruded Rod. If the extrusion billet is porous, some porosity may be expected in the extruded bar. Figure 2.3 shows an exaggerated case of this caused by shrinkage pipe in the billet.⁷

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Fig. 2.3—Extrusion butt and extruded bar ends from billet with shrinkage cavity showing sponginess at butt end of bar. A, leading end. B, butt end of bar showing decrease in porosity during cropping $2\frac{1}{2}$ -in. lengths. C, extrusion butt.

Holes, more or less spherical in shape, are frequently encountered. These are believed to be caused by small gas pockets in the cast billets, which reopen as a result of the gas pressure on the soft metal after it passes the die.

A defect, known as the "extrusion defect," can occur with sound billets. As the last portion of the billet is being extruded, abnormal flow occurs, and surface oxide is drawn into the bar. Figure 2.4 shows a $\frac{3}{4}$ -in.-thick extrusion butt sectioned to reveal the defect extending from the face of the billet and just entering the bar.^{7,11} The defect is noticeable at the butt end of the bar. The yield of bars has been maintained in the neighborhood of 90 per cent of the billet weight. To attain this, butts only $\frac{1}{2}$ in. thick were left. As a result of this procedure about 2 ft of the butt end of the bar was subject to the extrusion defect.

Fig. 2.4—Section through $\frac{3}{4}$ -in. extrusion butt showing origin of extrusion defect and appearance of defect in butt end of bar. A, leading end. B, butt end. C, butt.

The surface quality of extruded bars is largely dependent on the condition of the die and the amount of oxidation during cooling. Surface cracking may appear on bars containing iron exceeding about 0.05 wt. %. Lowering the extrusion temperature has been found beneficial.

2. OUTGASSING OF URANIUM

By H. W. Russell and H. R. Nelson

The hydrogen content of extruded uranium bars is generally too high for certain applications, and an outgassing treatment is required.

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In the case of slugs sealed in protective aluminum jackets or other impervious coatings, the evolution of hydrogen at elevated temperatures may cause swelling of the jackets or failure of the coatings. For such applications, the hydrogen content must be reduced to harmless values before the jackets are applied. A heat-treatment for several hours in an inert atmosphere that is maintained low in hydrogen has been found to be the most practical method of outgassing.

2.1 General Principles. In order to produce swelling of jackets, it is necessary that evolved hydrogen build up to gas pressures exceed-

Fig. 2.5—Solubility of hydrogen in uranium in equilibrium with hydrogen at atmospheric pressure.

ing 1 atm. From the hydrogen-solubility curve,^{12,13} Fig. 2.5, it is apparent that uranium containing more than about 2 ppm hydrogen may evolve hydrogen into a restricted space to pressures exceeding 1 atm when the temperature of the metal is maintained in the range 440 to 660°C. For complete safety, therefore, the outgassing treatment should reduce the hydrogen content to 2 ppm or less.

Since the removal of hydrogen in the outgassing operation is a diffusion process whose rate increases rapidly with increasing temperature, it is desirable to utilize as high a temperature as possible in order to shorten the time. Experience has shown that 620 to 660°C is a

SECRET

satisfactory and practical working range. At these temperatures ordinary black-iron pipes can be used as containers to protect the uranium from air and other active furnace gases. Moreover, this temperature range avoids both the higher hydrogen solubility of the beta phase and the possibility of undesirable properties resulting from heating into the beta region.

The length of time the bars are held at temperature depends on the diameter of the bars and the degree of outgassing that is required. Since extruded bars of uranium have been found to contain as much as

Fig. 2.6—Theoretical rate of outgassing of rods at 640°C. Upper curve is for 1.44-in. diameter rods; lower curve is for 1.25-in.-diameter rods.

8 ppm hydrogen, it has been common practice to hold them at temperature for a period long enough to reduce the hydrogen to 20 to 25 wt. % of the original content. Figure 2.6 shows the theoretical rates of outgassing at 640°C for rods with diameters of 1.25 and 1.44 in. These curves were calculated on the assumption that initially the hydrogen is uniformly distributed over the cross section of the rod, and the hydrogen-diffusion coefficient at 640°C was taken as 0.0125 sq in./hr, a value obtained experimentally.^{13,14} The curves show that 6 hr is a sufficiently long time for the 1.25-in. rods, whereas a period of 8 hr is required for the larger diameter rods. The time required to outgas other sizes varies directly as the square of the rod diameter.

SECRET

The atmosphere surrounding the rods during outgassing must be inert to uranium in order to prevent excessive scaling, and its hydrogen content must be maintained at a low value to provide the necessary diffusion gradient. Common bright-annealing furnace atmospheres fail in one or both of these requirements. Air, carbon dioxide, and nitrogen cause rapid scaling. Natural gas has been found to be ineffective, probably because the nitrogen present produces a scale through which hydrogen diffuses rather slowly.¹⁵ Vacuum outgassing is quite satisfactory as a laboratory technique, but it has not been used commercially for obvious reasons. Any of the rare gases may be used, but argon has been chosen because of its availability at high purity.

The atmosphere must be changed periodically or continuously to keep the hydrogen content of the gas reasonably low. It has been found that an average hydrogen content of 10 vol. % in the argon atmosphere is satisfactory as a working maximum. Since 1 cu ft of extruded bar stock is unlikely to contain more than 2 cu ft of hydrogen, it is necessary to pass a total of 20 cu ft of argon per cubic foot of uranium (35 cu ft/ton) over the bars during the outgassing period to maintain the average hydrogen content of the gas below the 10 vol. % limit.

2.2 Commercial Outgassing Practice. Outgassing of extruded uranium rods using the process described below has been carried out extensively at two plants. This process has proved completely satisfactory, although it is probably not the best or the cheapest that could be devised.

In the outgassing of 10- to 15-ton lots of uranium rods in one operation, the rods are placed in black-iron pipes 8 in. in diameter by 10 ft long. Each pipe holds 15 to 18 rods 1.49 in. in diameter. The ends of the pipes are closed by welding on $\frac{1}{2}$ -in. mild-steel plates that carry inlet- or outlet-gas connections. Four loaded pipes are placed on each of four furnace pans, and gas connections are made between the pipes with $\frac{3}{8}$ -in. soft-steel tubing, two groups of eight pipes being connected in series. All joints are welded to ensure gastight seals. The four pans of pipes are placed on the charging table in front of the furnace, and the argon-supply and -discharge lines are connected, as shown in Fig. 2.7.

The 16 pipes are purged of air by passing argon through them until the exhaust gas analyzes less than 1 vol. % oxygen. After purging, the gas lines are disconnected for a short time while the four pans are pulled into the furnace, the temperature of which, at the time of charging, is generally at 1200°F (650°C). A hand-controlled oil-fired furnace, whose chamber dimensions are 3 ft high by 7.5 ft wide by 28.5 ft long, has been used in most of the outgassing treatment. The furnace temperature is indicated by four thermocouples spaced equally along

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SECRET

Fig. 2.7—Arrangement of iron pipes and interconnecting argon lines for outgassing uranium bars.

the furnace length. The metal temperatures are measured by thermocouples at the centers of two pipes at opposite ends of the furnace, as shown in Fig. 2.7. About 2.5 hr is required to bring all the rods to temperature, after which the metal temperature is held in the range 1150 to 1220°F (620 to 660°C) for a soaking period of 9 hr. At the end of the soaking period, the gas-supply and -exhaust lines are cut, crimped, and sealed with a weld bead before the charge is pulled out of the furnace. After a cooling period of about 3 hr, one end of each pipe is sawed off, and the rods are removed.

Argon, 99.6 per cent pure, is obtained in bottles containing approximately 300 cu ft of gas compressed to 2800 psi. Gas from four of

Table 2.1—Gas Consumption for a Typical Outgassing Treatment of 10.5 Tons of Metal

Operation	Approximate time, hr	Gas required, cu ft	Drop in gas pressure, psi*
Purging	0.5	128	300
Heating	2.5	156	360
Soaking	9	562	1300
Total	12	846	1960

*Four bottles.

these bottles is passed through the pipes at a rate of 6 cu ft/hr per ton of metal during the heating and soaking periods. The rate of flow is measured on flowmeters and is manually controlled with needle valves.

The gas consumption for a typical outgassing treatment of 10.5 tons of metal is given in Table 2.1.

Figure 2.8 gives a record of the hydrogen content of the exhaust gas and the temperature of the metal at the center of one tube during a typical outgassing treatment of 1.25-in. rods.¹⁵ The hydrogen-content curve is similar for the 9-hr soak that is used in outgassing 1.49-in. rods. Although most of the hydrogen is removed early in the outgassing treatment, it has been found necessary to continue the high-temperature soak in order to remove hydrogen from those portions, particularly the butt ends of extruded bars, that tend to be high in hydrogen.

Table 2.2 gives typical hydrogen analyses of samples of extruded bars before and after outgassing.^{16,17} Most of the samples were taken

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Fig. 2.8—Hydrogen content of the exhaust gas and temperature at the center of the charge in one tube during a typical outgassing treatment of 1.25-in. rods.

Table 2.2—Hydrogen Contents of Samples of Extruded Bars

Bar No.	Diameter*	Section sampled	Hydrogen content, ppm	
			As-extruded	Outgassed
A-3230	X	Lead end	1.5	0.5
		Butt end	2.3	0.5
A-3241	X	Lead end	2.1	0.6
		Butt end	4.5	0.9
W-618	W	Lead end	1.5	1.1
		Butt end	5-6	1.4
G-29-A	W	Lead end	2.0	0.7
		Butt end	6-7	1.1
A-1318	X	Not given	4-8	
A-3230	X	Not given	1.5-2.3	
H-539	W	Lead end		0.8
D-371	W	Lead end		0.5
G-41-A	W	Lead end		0.7
D-392	W	Lead end		0.6
D-417	W	Lead end		0.5
BT-252	W	Butt end		2.1
H-656	W	Butt end		1.3
H-678	W	Butt end		2.1
W-522	W	Butt end		1.5

*X denotes 1.25-in. rods that were turned to 1.1-in. diameter before analysis, and W denotes 1.44- to 1.49-in. rods that were turned to 1.34-in. diameter before analysis.

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from near the ends of the bars, and therefore the analyses are not typical of the metal as a whole.

3. ROLLING OF URANIUM

By J. M. Simmons

The production of uranium in desired shapes by common commercial practices was a research problem of importance equal to that of casting the metal into massive ingots. Therefore studies on uranium fabrication were initiated as soon as enough of the metal became available. Based on a knowledge of industrial methods, rolling of uranium, either cold or at some elevated temperature to be determined by experiment, was judged to be a feasible method for large-scale production of semifinished material. Cooperating laboratories at Massachusetts Institute of Technology (M.I.T.), National Bureau of Standards (NBS), and Battelle Memorial Institute (BMI) and in Great Britain began studies on rolling almost simultaneously in early 1943, although a few small pieces of rolled uranium sheet had been produced by J. W. Marden of Westinghouse Electric Corporation several years before this project was set up.

3.1 Cold Rolling. Rolling at room temperature has been successful on thin sheets of uranium stock; short lengths of strip from vacuum-fused metal were given a 75 per cent area reduction without annealing.¹⁷ Stock rolled from 0.25 in. thick to 0.033 in. thick required an anneal at 600°C after each 10 per cent area reduction to prevent edge cracking, but this same material was cold-rolled from 0.033 in. thick to a 0.005-in. foil without annealing and was finished as sound metal.¹⁸ A 20 per cent area reduction of uranium by cold rolling has been obtained before surface and edge cracking occurred, but this is the maximum limit of reduction on massive sheets of the metal.²⁰ The pronounced work-hardening of uranium under cold working, its high resistance to deformation at room temperature, and its good workability at higher temperatures have been responsible for the lack of interest in further attempts at cold rolling.

3.2 Experimental Hot Rolling in the Alpha Range. Rolling of uranium at temperatures below the first transformation point (660°C) has been investigated thoroughly, both on an experimental and on a production scale. Cast cylinders of metal, 2 in. in diameter, were heated to 560°C and rolled into strips $\frac{3}{16}$ in. thick without further heating. The metal produced was sound, although edge cracking took place after a total area reduction of 37 per cent.²¹ Other samples of strip, 0.2 in. thick, were rolled at 300°C to 0.1 in. thick between rolls which were kept at 250°C. These samples were reheated between each pass, and seven passes were required to reach the finished thickness.²² On roll-

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ing 1.5-in.-square bars of metal to 1-in. rounds at an initial temperature of 540°C, a temperature rise in the bar was observed.²³ This rise in metal temperature during hot rolling has played an important part in determining the proper procedures to be followed in commercial rolling.

Uranium at 620°C has the same resistance to deformation as does steel at 1040 to 1100°C. Apparently the resistance to deformation increases with the carbon content of the metal; a bar containing 0.26 wt. % carbon required twice the roll pressure and power as did one having only 0.08 wt. % carbon.²⁴

The first experimental rolling on commercial equipment was conducted at The Joslyn Mfg. & Supply Co., early in 1943.²⁵ Three uranium ingots, cast at M.I.T., approximately 3 in. in diameter and 16 in. long, were preheated in a propane atmosphere and rolled to about a 1.32-in.-diameter rod. Billet 1 was preheated to 640°C and rolled into a rod; the surface of the finished rod was pitted, cracked, and uneven. Billet 2, preheated to 610°C, was rolled under different conditions; a heavy stream of water flowed over the rolls in each stand during rolling, and the billet was allowed to cool after each roll pass. The finished rod had a better surface, although pitting and unevenness persisted. Billet 3 was preheated to only 554°C, and an attempt to roll it was made; on the second pass through the 12-in. mill, one of the roughing rolls broke, ending the experiment.

After the first unsuccessful attempt at rolling uranium in commercial mills, arrangements were made to use Joslyn's stainless-steel mill located at Fort Wayne, Ind.^{26,27} Billets were rolled into acceptable rods on this equipment, but, because the extrusion process was by that time producing metal thought to be sound, further experimental work on this program was discontinued.

3.3 Commercial Hot Rolling in the Alpha Range. All commercial rolling has been carried out at The Joslyn Mfg. & Supply Co. Uranium rod, produced by rolling in the alpha range, is sound and free from internal defects. Its production is economical and simple, and it has no apparent defects except occasional surface seams. A rolling-mill practice has been worked out, which, when used on production runs, gives sound rods, with only slight oxidation loss and billet spoilage.^{28,29}

Rolling billets, 4 to $4\frac{3}{8}$ in. in diameter if round, sides $4\frac{1}{2}$ in. and $\frac{5}{8}$ in. if square, and 20 to 28 in. in length, are heated individually in electric furnaces with natural-gas atmosphere. The billets are heated to 590 to 615°C, allowed to soak at this temperature for a minimum time of 30 min, and then rolled. Since the metal heats up during rolling, the billet is held for a few seconds after each roll pass to allow cooling; the rolling cycle is about 15 min per billet.

SECRET

Two different types of roll-pass designs have been used with about equal success in the commercial production of uranium rod: (1) diamond-shaped breakdown and intermediate passes, with finishing to round accomplished through a series of rounded oblong and oval passes³⁰ and (2) "hand-round" passes,³¹ all of which are almost circular in section. For the diamond-pass rolling, three sets of mills are used: 18- and 12-in. roughing mills and a 9-in. finishing mill whose settings are similar to those used for stainless-steel rolling. The hand-round rolling is all done on the 18-in. mills, and billets are reduced from $4\frac{1}{8}$ in. to $1\frac{1}{2}$ in. in diameter in 25 passes at a roll speed of 22 rpm, which includes 4 rounding passes through the final hole. The normal reduction in area per pass for both of the above rolling procedures is 10 per cent, and the rolled rod size is 1.52 to 1.55 in. in diameter (cold) if the rod is to be finished by machining or grinding at 1.360 in. in diameter.

Rods produced by rolling are from 0.01 to 0.04 in. out of round; their surfaces are heavily oxidized, and the rods are usually quenched in water immediately after the last pass to minimize oxidation during cooling. "Seaming" of the rod is the most troublesome defect encountered in rolling. The roll design has been changed several times because it was thought that seams in rods might be eliminated by altering the shape of the finishing passes.³²⁻³⁴ This was only partially successful; it is highly probable that the working qualities inherent in the metal are responsible for most of the seaming.

Billet condition before rolling is important; cold shuts, scabs, and porosity in the casting will give rods of varying degrees of soundness.³⁵

Billet temperatures during rolling are difficult to measure. During one run, bar temperatures, measured with an iron-constantan lava-cap contact element, were found to be:³⁶

Mill	Temperature, °C
18 in.	488-523
12 in.	530-545
9 in.	482-566

The rolling temperature is, in actual practice, judged by eye.

3.4 Experimental Rolling in the Beta Range. Rolling of uranium within the beta range (660 to 770°C) apparently is feasible, but temperatures during rolling must be very closely controlled. However, experimental rolling within this range has failed to produce rods free

SECRET

from cracking or checking;³⁷ roll pressures in this range are higher than they are for rolling in the alpha range at about 550°C.

3.5 Experimental Rolling in the Gamma Range. Rolling at temperatures above the second transformation point (770°C) has been attempted at several laboratories. Cylinders of cast metal, 1 in. in diameter, after being heated in argon to 900°C were rolled to 0.5-in. squares;³⁸ square bars, 1.5 in. on the side, were rolled²³ to 1-in. diameter cylinders in five roll passes at bar temperatures of 870 to 930°C. Rolling at temperatures above 770°C produces bars of poor metal, checked and cracked, with high losses from scaling.³⁷ Two regular-size rolling billets were rolled in the gamma range on a commercial rolling mill;³⁹ both rods jammed in the 9-in. finishing mill because the metal was too soft to work at these sizes and temperatures.

3.6 Rolling of Uranium Alloys. Alloys of 95 wt. % uranium and 5 wt. % molybdenum have been rolled⁴⁰ into rods of various diameters at temperatures of 760 to 790°C. Alloys of uranium containing 2 to 4 wt. % columbium were rolled at 790°C, but alloys containing 6 to 8 wt. % columbium could not be finished successfully at this temperature.³⁹ Although small samples of alloys have been rolled on experimental mills, none, other than those listed above, have been rolled on a production rolling mill.

4. DRAWING OF URANIUM

By J. M. Simmons

Concurrent with the investigation of uranium fabrication by means of extrusion and rolling, a program dealing with the feasibility of drawing metal into semifinished rod and wire was undertaken on both an experimental and a commercial basis. It was desirable to produce as high a percentage of finished material as possible, and investigators believed that, by drawing rolled or extruded rods down to diameters just above the finished slug size before machining or grinding, a more economical production could be attained. Uranium wire of small diameter was required in experimental and production work on the project, and the large-scale fabrication of this material apparently was feasible only by drawing. Consequently wire drawing was investigated. The experimental work on drawing was initiated at the laboratories of BMI and NBS, and experimental drawing on commercial equipment was undertaken at The Joslyn Mfg. & Supply Co.

4.1 Experimental Cold Drawing. Uranium rod, 0.420 in. in diameter, produced by hot rolling, was cold-drawn to 0.394 in., but the rod fractured when an attempt was made to reduce it to 0.374 in. At the

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same time, hot-swaged material, 0.220 in. in diameter, fractured when a reduction to 0.185 in. in diameter was attempted.⁴¹ Drawing of as-extruded rod, approximately 1.200 in. in diameter, was successful up to an area reduction of 6 per cent, but any attempt to obtain a greater area reduction on an uncoated rod failed. The rod galled badly, stuck in the die, and snapped.⁴² Studies of surface preparation, lubricants, die materials, pickles, and methods of drawing were made to simplify the cold-drawing process. The as-extruded rod could not be cold-drawn without a preliminary surface treatment; its surface was full of slivers, scabs, and ridges which adhered to the draw die. Sandblasting of the rod was found to be a suitable preparation for drawing; therefore that treatment was adopted. No ordinary lubricant, such as Oildag, white lead, beeswax and castor oil, or metallic soap, was effective; the best results, although not good, were obtained by using a mixture of 50 per cent beeswax and 50 per cent glycerine as a lubricant. Using this lubricant, an area reduction of 16 per cent was made in two draw passes of a sandblasted rod.⁴³ Stoodite or Carboloy dies were used for drawing; preliminary pickling was not helpful.

A process was developed for cold drawing, which, under carefully controlled conditions, could be used quite effectively, giving a 36 per cent area reduction of the rod and increasing the output of material suitable for finished slugs by 18 per cent.⁴² Rods were copper-coated from a copper sulfate bath, this coating serving as a lubricant and preventing die scoring and rod galling. As-extruded rods were sandblasted, pickled in cold nitric acid (50 vol. % commercial nitric acid and 50 vol. % water), and washed in cold water. Immediately after washing, the rods were immersed in a bath made up of 40 g/liter of 37 per cent hydrochloric acid, 25 g/liter of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, and 300 g/liter of 95.5 per cent sulfuric acid; 30 sec after the rods were immersed, gas evolution began in the solution; 30 sec after gas evolution began, the rods were removed from the bath and were washed in cold water. A bright, uniform coating of copper covered the rod and, on passing through a die, acted as a lubricant. Longer immersion of the uranium in the bath gave a nonadherent coating; a shorter period produced a spotty coating.

Uranium wire, 0.012 in. in diameter, was first produced by drawing swaged wire, 0.030 in. in diameter, through sapphire dies, the wire having been precoated with a copperplate 0.0005 in. thick.⁴⁴ Success in drawing depended on die lubrication, this being achieved by copperplating the wire in an acid copper sulfate solution after first cleaning the wire anodically in a 50 per cent sulfuric acid solution. In later developments this practice was modified, after it was apparent that

SECRET

the major problem in wire production was the formation of an adherent, uniform coat of copper on a long thin wire.⁴⁵ Uranium wire, 20 mils or less in diameter, was cleaned in a 60 per cent nitric acid solution, and a passive film was produced on the wire by the bath. This film was removed by the immersion of the coiled wire in a bath of 250 g/liter of copper sulfate, which had been brought to a pH of 3.3 with cupric hydroxide; the wire was connected to a current source and was made anodic for 2 min. The polarity was then reversed, and the wire was made cathodic for 30 min (voltage, 1.1 to 1.2 volts; current density, 2 to 5 amp/sq ft). By this treatment a continuous, adherent, uniform film of copper, 0.0005 to 0.001 in. thick, was laid down, but the process was extremely difficult to control.

If its diameter were greater than 10 mils, plated wire usually could be drawn through several dies before replating was necessary. Wire 10 mils or less in diameter had to be replated after each pass. Annealing of the wire was necessary after a 10 to 15 per cent area reduction in wire of a diameter range of 30 to 10 mils. Annealing was carried out by passing the wire through a helium-atmosphere tube furnace held at 600°C. Wire 0.200 in. in diameter was annealed for 5 min, whereas wire of lesser diameter was annealed for 2 min.

4.2 Experimental Hot Drawing. Hot drawing of swaged wire at 250 to 300°C from 0.030 to 0.020 in. in diameter through sapphire dies was not successful.⁴⁶ Later experiments⁴⁷ in hot drawing proved that the process was feasible; 0.375-in.-diameter uranium rod was fluxed in a salt bath of sodium chloride, potassium chloride, and lithium chloride; dipped while hot into a molten lead bath; and drawn immediately at a temperature of 200 to 260°C. The diameter of the rod was reduced from 0.375 to 0.311 in. in three steps of 7, 17, and 12 per cent area reduction; an excellent rod surface was produced by this method of drawing. A flux, whose essential components were cryolite and lithium fluoride, was found to clean uranium much better than the chloride bath.

4.3 Commercial Drawing. Four rods, 1.190-in.-diameter extruded stock for Clinton-size slugs, were centerless-ground to 1.075 in. in diameter to remove any surface imperfections before being copper-coated by the experimental method developed by BMI.⁴⁸ These rods were cold-drawn through Carboloy dies, giving a 2 per cent area reduction, with no die scoring apparent. Three as-extruded rods, 1.190 in. in diameter, were pickled and then coated with copper, and attempts were made to cold-draw them through a Carboloy die. All the rods snapped after 4 to 6 in. had passed through the die; the drawn surface was badly galled.⁴⁹ Ten other as-extruded rods were reduced 0.065 in. on the diameter by centerless grinding; they were then pre-

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pared and drawn. Cold drawing, with 0.040 to 0.050-in. -diameter reduction, was possible on smooth, copper-coated rods; no rough-surfaced rods were drawn successfully.⁵⁰ Apparently the cold drawing of rough and as-extruded rods is not feasible. Rods which have been prepared by centerless grinding before coating and drawing can be reduced 0.040 to 0.050 in. in diameter; this reduction, however, work-hardens the rods and requires an anneal before additional working.

5. MACHINING AND GRINDING OF URANIUM

By J. M. Simmons

Investigation of the machinability of uranium began almost simultaneously with the production of the metal in shapes massive enough to be worked in lathes, grinders, drill presses, and other machine tools used in shops. It was apparent from the specifications and numbers of the cylinders requested for use that only by large-scale machine-shop production could this material be delivered at the speed and with the tolerances desired. Consequently, intensive work upon metal machinability was undertaken at a number of laboratories. Extruded rod, 1.1 in. in diameter, was found to machine somewhat like the austenitic stainless steels, work-hardening under tools, requiring a continuous high feed rate, even though tool failure came from wear at the tool point rather than from cratering and fracture behind the cutting edge.⁵¹ The type of tool failure indicated the presence of an abrasive substance in the metal which caused the wear, since the hardness of the metal itself did not explain the short tool life. Too, machinability varied from bar to bar, and even different sections of the same bar contained hard and soft spots.⁵² Cast billets were sectioned and cut off by a mechanical feed power hacksaw;⁵³ later an abrasive-wheel cutoff machine was used. On the basis of these few preliminary experiments, machine shops for the machining of uranium were put into production operation, and modifications in the methods of turning and cutting were made as soon as they were discovered; thus the best machining practice for working uranium was learned while the actual production of slugs was in progress.

5.1 Machinability. The term "machinability" refers to the speed with which metal can be removed by machining, and in judging this the following factors must be considered: speed of metal removal, tool life, character of the finished machined surface, heat evolved from the operation, and condition of the machines. Cutting tools must be shaped accurately; only a few degrees difference in the cutting-edge angle will make a great change in the speed of metal removal.

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Tools for cutting uranium are carbide-tipped because carbide is highly resistant to the abrasive action of the metal. Uranium distorts readily upon machining; therefore very light finishing cuts and a heavy flow of coolant are necessary to dissipate the heat generated by the tool cutting a pyrophoric metal.

For cutting rough stock (i.e., cast billets and heavy rods), an abrasive-wheel cutoff machine is recommended; a power saw is too slow. The work should be clamped securely, a heavy flow of coolant (Sun Oil emulsifying oil, a ratio of 20 parts water to 1 part oil is recommended) should be kept over the wheel and work, and a blower system must be used to draw off oxide fumes given off during the operation.

Tools tipped with tungsten carbide, either Carboloy or Kennametal, are used most successfully in turning uranium; high-speed steel tools have been employed less efficiently. The cutting speed for rough turning is 130 ft/min, with a feed of 0.010 in. per revolution and a maximum cut depth of 0.040 in.; for finish turning, the cutting speed is increased to 150 ft/min, and the feed is reduced to 0.003 in. per revolution. A heavy coolant flow, directed at the cutting point, is used in turning; chips from the work should fall into the coolant in the bottom of the lathe to prevent them from burning.

Carbide-tipped tools are used in boring and milling operations on uranium. In boring, a cutting speed of 100 to 120 ft/min, with a feed of 0.002 to 0.006 in. per revolution, and a rigid boring tool are recommended. Milling cutters tipped with carbide can be operated at a greater speed in milling uranium than can high-speed steel cutters; high-speed steel cutters are used at a speed of 30 to 50 ft/min, whereas carbide-tipped cutters operate at rates of 100 to 175 ft/min. Since high-speed steel dulls very rapidly from the abrasive action of uranium, its use as a tool is extremely uneconomical.

The metal can be drilled, threaded, and reamed with ease, but it is very difficult to tap. Carbide drills up to 1 in. in diameter and high-speed steel drills $\frac{1}{8}$ in. in diameter and less have been employed in drilling. In drilling small holes, $\frac{1}{8}$ in. and less, the uranium must be heated to 150°C, and a sensitive-touch drill press must be used without any lubrication on the drill point. For larger holes an ordinary drill press and a coolant flow just sufficient to prevent the drill from burning are used. Reaming and threading are straightforward operations, the cutting speeds being approximately one-half the speed for turning; however, tapping is difficult and must be done slowly by hand. A paste of acetylsalicylic acid and water is used as a lubricant; heating the uranium to 150°C makes the operation easier.

The Medart rough turner, a machine used in roughing down stainless-steel bars, has been used in making heavy, rough cuts on ura-

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ni-um bars of 9-ft or greater lengths.⁴⁸ This machine works on the principle of a firmly held fixed bar being fed slowly through a revolving cutter head; the cutter head is armed with one or two sets of tools, four tools to a set, 90 deg apart and takes a deep, rough cut of 0.050 to 0.065 in. on the radius of the bar as it passes through the machine at a feeding speed of 20 in./min. Tool wear is excessive on this machine when it is used on uranium since high-speed steel must be used. Carboly tips would be shattered by the shock of the deep cut employed.

5.2 Grinding. Centerless grinding, the grinding of rods and slugs not held between centers, has been used on uranium since the first rods were extruded. The first uranium bar to be finished by centerless grinding was ground at Globe Tube Company into an approximately 1.280-in.-diameter rod, 6.5 ft long, in January 1943.⁵⁵ By using a centerless grinder (all grinding has been done on Cincinnati centerless grinders), stock is removed evenly from a bar, out-of-round bars are rounded, and tool marks and taper are removed from semifinished slugs.

For bar grinding (lengths of 5 ft or more), the bar is cropped, straightened on a Medart straightener to remove all kinks and bends, and passed through the centerless grinder which removes 0.005 to 0.010 in. from the bar diameter on each pass.^{56,57} When the bar is 0.005 in. above the desired diameter, the roughing wheel is removed from the grinder, replaced by a finishing wheel, and finishing cuts of 0.001 to 0.002 in. are taken until the finished size is reached. Where highly polished surfaces are desired, the centerless grinder produces excellent material.

A heavy flow of coolant must be maintained over the wheel surfaces during grinding to prevent the uranium bar from sparking excessively and to prevent the finely divided grindings from burning. At the end of each mill shift, the coolant tank must be emptied, grindings must be removed and stored in an open metal container, which is kept outside of all buildings, and fresh coolant solution must be prepared. If the grindings and contaminated solution are not removed on this schedule, there is a real danger of fire and hydrogen explosions in the machine during operations. The wheels used successfully for grinding are 46/60 grit SiC bonded wheels for rough cutting and 80 grit SiC bonded wheels for finishing.

In cylindrical and surface grinding of uranium, surface speeds of 25 ft/min for rough grinding and 15 ft/min for finish grinding are suitable.⁵⁴ For rough grinding, a feed of one-half to three-fourths wheel width to each revolution of the work and a cut 0.001 to 0.004 in. in depth is used; for accurate finishes, a feed of one-fourth wheel

width to each revolution of the work and a cut 0.00025 to 0.0005 in. in depth is taken

5.3 Safety Precautions.⁵⁴ More precautions must be taken in working uranium than with most metals since it is both pyrophoric and poisonous. Finely divided particles will react with water, giving off hydrogen gas which explodes easily. It is imperative that the workers wear safety goggles or face masks since the heavy chips fly about readily. Also, the chips usually burn rapidly from the machining heat on exposure to air; so chip breakers should be fitted to each machine to break the chips into small pieces which will be carried by the coolant flow to the reservoir underneath the machine. All rooms must be ventilated to remove the oxide fumes coming from the metal as it is machined; individual suction pipes should be fitted to each machine. Reference has been made to the danger inherent in the grindings, even under a coolant flow. It cannot be emphasized too strongly that finely divided uranium particles ignite spontaneously and, in some cases, have caused fires and explosions.

6. METAL QUALITY

By A. Van Echo

Inspection of Hanford slugs disclosed that gamma-extruded metal contains many internal defects in the form of blow holes, porosity, and oxide seams. These defects do not ordinarily appear on the cylindrical surface. Cold-swaging gamma-extruded metal does not improve the quality of the metal as regards internal defects and often results in serious surface damage. Recently extruded metal is of higher quality than that extruded earlier but is still not entirely sound.

Alpha-extruded metal is much the same as gamma-extruded metal but contains fewer defects and has a lower hydrogen content.

Alpha-rolled metal is sound and practically free of internal defects and has a low hydrogen content. Surface seams are largely eliminated by new roll design and larger cleanups. The metal is of much higher quality than gamma-extruded metal.

6.1 Gamma-extruded Rod. Some type of defect is found in most gamma-extruded metal.^{58,59} The defects are internal and seldom appear on the cylindrical surface of the slug but often appear on the ends of 8-in.-long slugs. The most common defects are blow holes located near the axis of the rod. The blow holes are more or less spherical and apparently clean holes, ranging in size from small pores to large holes over $\frac{1}{8}$ in. in diameter. These blow holes occur more or less uniformly along the length of the extruded rod.

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Infrequent defects are seams running parallel to the axis and located at various distances from the axis. These defects appear to be dirty and are filled with nonmetallic material, presumably the oxide. Other infrequent defects are seams perpendicular to the axis of the rod.

Defects of the type and magnitude found to exist will have but little influence upon slug-surface temperature during operation.⁶⁰

6.2 Alpha-extruded Rod. The defects in alpha-extruded rod⁶¹ are similar to those found in the gamma-extruded rod. The quality of the alpha-extruded material is somewhat better than that of gamma-extruded material. The hydrogen content in the alpha-extruded metal is lower than that of gamma-extruded metal.

6.3 Gamma-extruded and Cold-swaged Rod. A rod reduced 40 mils in diameter by swaging^{62,63} and 87 mils by machining cleaned up satisfactorily. Rods that were reduced 70 mils in diameter by swaging and only 27 mils by machining did not clean up. Good quality extruded rod, lightly swaged, cleans up satisfactorily, but heavy swaging results in serious surface damage. Cold-swaging gamma-extruded metal does not improve the quality of the metal as regards internal defects.

6.4 Alpha-rolled Rod. Rods fabricated by rolling in the high-alpha region^{30,64-66} are sound and practically free of internal defects. Most of the surface seams arise from wrinkling of unsupported surfaces in the finishing mill. A new set of rolls was designed that gave almost complete contact between roll and rod surface. These new roll passes produced better rod surfaces than did the old roll passes. Rolled metal is of much higher quality than gamma- or alpha-extruded metal. The hydrogen content of as-rolled metal is 1.0 ppm or less and is as low as the hydrogen content of degassed gamma-extruded metal. The alpha-rolled metal would be usable without degassing.

Alpha-rolled metal is an excellent supersonic conductor and is much better in this respect than is degassed gamma-extruded metal. The supersonic reflectoscope readily distinguished sound rolled metal from porous metal. The density of the as-rolled metal is practically the same as that of gamma-extruded metal and probably depends more upon carbon content than upon porosity. Carbon affects the density at the rate of 0.02 g/cm³ per 100 ppm carbon.⁶⁷

6.5 Type of Defects. The types of defects observed in the metal from the various methods of fabrication can be summarized as follows:

1. Internal defects, including these flaws:

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a. Blow holes—spherical in shape, usually axial, and ranging in size up to $\frac{1}{8}$ in. in diameter—predominantly found in extruded metal.

(UNCLASSIFIED)

b. Seams of nonmetallic material parallel to the axis and off-center—observed in extruded metal.

(UNCLASSIFIED)

c. Striations of nonmetallic material at right angles to the axis—found occasionally in extruded metal.

(UNCLASSIFIED)

d. Small and numerous pores—found mostly in extruded metal.

(UNCLASSIFIED)

2. External or surface defects:

- a. Surface seams—longitudinal, found mostly on rolled material.
- b. Open blow holes on cut ends—noticed in extruded metal.

6.6 Methods of Inspection. For a study of defects in Hanford slugs, four methods of inspection were used for evaluation: supersonic-transmission measurements, Zyglo inspection, density measurements, and visual inspection.

(a) Supersonic-reflectoscope Tests.^{68,69} For a nondestructive test, F. A. Firestone, University of Michigan, has developed a method of measuring the supersonic transmission of metals. The method con-

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sists in sending a pulse of supersonic energy into one end of the slug and measuring the position, intensity, and number of reflections from the opposite end. In physically sound metal the initial pulse travels down the length of the slug, is reflected from the opposite end, and then travels back up to the starting end. Part of the energy is transmitted to the recording crystal, and part is internally reflected back toward the far end of the slug. The pulse travels back and forth within the slugs until its energy is dissipated. In sound Hanford slugs, 8 to 10 successive reflections, representing from 128 to 160 in. of travel, can be detected. Discontinuities in the metal will cause partial reflection at the interfaces and a rapid dissipation of the energy of the pulse. These effects will be detected in two ways: First, the intensity of the pulse reflected by the far end of the slug will be diminished, and the number of reflections of detectable energy will be fewer. Second, intermediate reflections representing distances of travel less than twice the length of the slug will be observed between the initial pulse and the first reflection from the opposite end. In general, sound metal will give a clean regular pattern repeated a number of times, whereas metal of low quality will give an irregular pattern showing many reflections representing short distances of travel.

(b) Zyglo Test. The slugs are given a nitric acid etch and then are tested for surface defects by the Zyglo method. This method is not too satisfactory, and a careful visual examination is just as good.

(c) Density. Density measurements were made by two methods, by determination of the mass-volume ratio and by immersion-weighing in monobrombenzene. Measurements were made on machined slugs and on ground slugs. Calibrated weights were used in making these measurements.

The density of Hanford slugs cannot be satisfactorily correlated with metal quality. Carbon affects the density at the rate of 0.02 g/cm³ per 100 ppm carbon. For density values to have any meaning, they would have to be corrected for the influence of carbon. Each rod would have to be analyzed for carbon, and samples should be taken from a number of locations along the length to check uniformity. Nondestructive grading by density measurement plus carbon analysis would appear to be impractical.

(d) Visual Inspection. Visual inspection of the ends of 8-in. slugs will give a general idea of the quality of the metal and will usually detect extensive defects such as longitudinal seams, but will only by chance reveal the existence of large blow holes or transverse seams. Visual examination of longitudinally sectioned slugs was used to evaluate other methods of testing. Sectioning the slugs is a destructive method and would be impractical for production inspection.

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Chapter 3

PHYSICAL AND MECHANICAL PROPERTIES OF URANIUM

By Frank G. Foote

This chapter gives an account of the large amount of information which has been obtained on the physical and mechanical properties of uranium metal during the past few years. A similar summary on the constitution and properties of uranium alloys will be found in Chaps. 5 and 6.

Uranium, with an atomic number of 92, the last element in the "natural" or "periodic" system of the elements, has a chemical atomic weight of 238.07. Natural uranium consists of three radioactive isotopes. The abundance and radioactive properties of each isotope are as follows:

U^{238} : 99.28%; α rays (4.23 mev), half life = 4.51×10^9 years

U^{235} : 0.71%; α rays (4.52 mev), half life = 8.5×10^8 years

U^{234} : 0.006%; α rays (4.78 mev), half life = 2.69×10^5 years

1. PHYSICAL PROPERTIES OF URANIUM

1.1 Allotropy, Crystal Structure, Atomic Dimensions, and Density.

Uranium forms three, or perhaps four, different solid phases. In the alpha phase, the metal crystallizes at room temperature in a lattice which is different from the usual closest packing arrangement in solid metals. In other words, the metal behaves as if its atoms were non-spherical. This orthorhombic structure (space group V^{17}) may be interpreted as a distorted-hexagonal closest packing. The unit cell contains four atoms, with the dimensions¹

$$a_0 = 2.852 \text{ \AA} \quad b_0 = 5.865 \text{ \AA} \quad c_0 = 4.945 \text{ \AA}$$

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Other investigators² have reported the values of the lattice parameters shown in Fig. 3.1. These data yield values at room temperature in good agreement with those above, namely:

$$a_0 = 2.852 \text{ \AA} \quad b_0 = 5.859 \text{ \AA} \quad c_0 = 4.944 \text{ \AA}$$

In this structure³ each atom has two nearest neighbors at a distance of 2.75 Å, two neighbors at 2.84 Å, four at 3.25 Å, and four at a distance of 3.34 Å. Thus the structure may be interpreted as a close packing of rotation ellipsoids with a small half axis of about 1.4 Å and a large half axis of about 1.65 Å.

The exact structure of the beta phase is unknown; several attempts to analyze its x-ray diagram have failed because of the weakness of the lines.⁴ According to one investigator⁵ this phase can be "frozen" from uranium-chromium alloys and proves to be a cubic phase with a giant unit cell (12.88 Å) containing 58 atoms.

The structure of the gamma phase, determined by means of the high-temperature x-ray camera, is a body-centered cubic lattice with a lattice constant of 3.48 Å at 785°C;^{4,6} extrapolation of the high-temperature measurements gives a lattice constant of 3.43 Å at room temperature. Extrapolation from the lattice constants of uranium-molybdenum alloys⁷ where the gamma structure can be preserved by quenching gives 3.48 Å for the lattice constant at room temperature. These constants correspond to an atomic radius of 1.49 or 1.51 Å.

The density of alpha uranium calculated from lattice constants¹ is 18.97 g/cm³, whereas calculations based on the data of Fig. 3.1 indicate a density of 19.00 at 25°C. The density of gamma uranium, obtained by extrapolating the high-temperature x-ray data to room temperature, is about 19.5. Direct measurements on commercial uranium metal give densities between 18.7 and 19.1, depending on the purity (especially carbon content) and the previous treatment of the specimen. *Typical results are given in Table 3.1. Do not lose*

The alpha form of uranium (orthorhombic) goes over upon heating, first into the beta form (cubic, exact structure unknown) and then into the gamma form (body-centered cubic). The alpha-to-beta transition is not strictly isothermal but extends over 10 or more degrees, perhaps due to nonuniform internal pressure.¹³ The transition temperatures are strongly affected by certain impurities; for example, 2.5 per cent molybdenum lowers the alpha-beta transition point from 760 to 650°C, eliminates the beta region altogether,¹⁴ and slows the gamma-alpha transition so much that the gamma phase can easily be preserved at room temperature by quenching.⁷ The presence of carbon, however, does not change the transition temperatures greatly.

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Fig. 3.1—The constants of alpha uranium as a function of temperature.³³

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With gamma-extruded metal, the alpha-beta transition begins at the same temperature in heating and in cooling; however, overheating and undercooling occur regularly with alpha-extruded material as well as with cast and biscuit metal.^{15,16} This hysteresis is illustrated in Figs.

Table 3.1—Density of Commercial Uranium

Specimen	Wt. % C	Density at 25°C, g/cm ³	Observer
99.9% cast metal		18.9	Driggs and Lilliendahl ⁸
Wire rolled to 1/5 its diameter		19.08	
MH* cropped ingot H74		18.93	Thompson ⁹
Rods cut from H77		18.95–19.01	
A* cropped ingots C733 and D733		18.89	
Rods cut from ingots C733 and D733		18.94–18.95	
Fourteen A* samples		18.92–19.01†	Peterson ¹⁰
Plant-cast metal	0	19.05‡	Kaufmann ¹¹
Plant-cast metal	0.01	19.02	
Plant-cast metal	0–0.24	19.05	
2-in. cast rod SF4	0.062	18.7	Seybolt and coworkers ¹²
2-in. cast rod SF4	0.066	18.9	
1 1/4-in. extruded rod RU677	0.051	18.9	
1 1/4-in. extruded rod RU677	0.055	18.7	

* MH = Metal Hydrides; A = Ames.

† Uncorrected values for samples, some with surface bubbles, at 23°C.

‡ Extrapolated.

3.2 and 3.3. The sudden decrease in emissivity¹⁷ that occurs at 1048 to 1050°C may indicate a third transformation, but this has not yet been thoroughly investigated. Typical data on the temperatures at which phase transformations occur are given in Table 3.2.

1.2 Thermal Expansion of Uranium Metal. The coefficient of linear expansion of uranium in the alpha region varies widely, depending on the nature and thermal pretreatment of the specimen. The reason for this is the anisotropic structure of the alpha phase, whose

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Fig. 3.2—Dilatometric curve of a longitudinal specimen of gamma-extruded uranium.¹⁵

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Fig. 3.3—Dilatometric curve of a transverse specimen of gamma-extruded uranium.¹⁵

expansion coefficients in the three main crystallographic directions are quite different²² (see Table 3.3). The measured linear-expansion coefficients of different uranium specimens and their changes with

Table 3.2—Transition Temperatures of Uranium

Material	Transition temperature, °C				Observer
	Heating		Cooling		
	$\alpha \rightarrow \beta$	$\beta \rightarrow \gamma$	$\gamma \rightarrow \beta$	$\beta \rightarrow \alpha$	
Forged 2 $\frac{1}{16}$ -in. round bars of cast uranium	673	775	770		Kaufmann ¹⁸
Uranium metal	662 $\pm$ 3	772 $\pm$ 3			Kelley and Moore ¹⁹
Uranium, containing 0.03% C			775	645	
Uranium-carbon alloy 0.33% C					
1.50% C			785	652	Carter ²⁰
Gamma-extruded rod, longitudinal specimen	660.5 $\pm$ 1.5		762	633	BMI ¹⁵
MH, A,* and Hanford ingots	665-670	766-776†	760-770†	643-651†	Thompson ¹⁶
ICI† Widnes metal (heated in vacuo)	650	764-771	752	629	Sykes ²¹

* MH = Metal Hydrides; A = Ames.

† Determined from conductivity curves (see Fig. 3.4).

‡ Imperial Chemical Industries.

Table 3.3—Expansion Coefficients of Alpha Uranium in the Main Crystallographic Directions*

Direction	Coefficient, 10 ⁻⁶ cm/(cm)(°C)	
	25 to 300°C	25 to 650°C
a	23 $\pm$ 3	28 $\pm$ 2
b	-3.5 $\pm$ 2	-1.4 $\pm$ 1
c	17 $\pm$ 2	22 $\pm$ 1

*See Fig. 3.1.

thermal treatment cannot yet be fully analyzed in terms of these three fundamental coefficients, but undoubtedly they are determined by the preferred orientation of the crystals in the polycrystalline material. In rolling or extrusion the individual crystals tend to orient

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themselves with the low-expansion axis parallel to the direction of flow. Consequently, longitudinal samples of alpha-rolled or alpha-extruded material have much smaller linear-expansion coefficients

Fig. 3.4—Electric resistance of uranium as a function of temperature.¹⁶

than transverse ones. The same difference appears, although less pronouncedly, even in gamma-extruded specimens. Heating above the alpha-beta transformation point and cooling back to room temperature tend to equalize the longitudinal and transverse coefficients

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and leave a permanent dimensional change, namely, a longitudinal contraction of the order of 0.5 per cent and a transverse dilatation of the order of 0.25 per cent—without appreciable change of density²³ (see Figs. 3.2 and 3.3).

Dilatometer curves of a specimen cut longitudinally from commercially extruded and outgassed bar are shown in Fig. 3.2. The first cycle gave a net shortening of the specimen and an increase in the coefficient of expansion as indicated on the curves.¹⁵ The second cycle, not shown, resulted in a further shortening and an increase in the coefficient of expansion. Note that the transformations in both directions started at the same temperature and that the change in length in the transformation varied widely. Expansions and contractions of this specimen during seven cycles are summarized in Fig. 3.5. Similar curves¹⁵ for a specimen cut transversely from commercially extruded and outgassed bar are given in Fig. 3.3. Each cycle resulted in an increase in length and a decrease in the first two cycles with a longitudinal specimen. This specimen was overheated and undercooled before transformation.

Some characteristic empirical expansion coefficients are given in Table 3.4.

Table 3.4—Linear Expansion Coefficients of Alpha-uranium Specimens

Specimen	Coefficient, 10^{-6} cm/(cm)(°C)	Observer
Light wire under stress ($2-4 \times 10^8$ dynes/cm ²)	5.95	Snyder and Kamm ²⁴
Rolled or extruded longitudinal alpha specimens	3 - 6	BMI ¹⁵
Gamma-extruded longitudinal commercial metal specimens	~ 15	BMI ²³
Gamma-extruded transverse specimens, commercial metal	Up to 26	BMI ²³
Specimens of types 3 and 4 after 1 to 2 heating cycles	~ 20	BMI ²³

The linear-dilatation coefficient of the beta region is nearly constant³ (about 22×10^{-6} cm/(cm)(°C). The alpha-beta and beta-gamma transformations are both accompanied by expansion. The extent of linear expansion caused by alpha-beta transition is variable, probably because of variations in the preferred orientation of crystals in the alpha phase. In repeated recycling of longitudinal specimens of

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Fig. 3.5—Changes in length caused by repeated cycling of longitudinal and transverse specimens of gamma-extruded uranium.¹⁵

gamma-extruded metal,¹⁵ the change in length associated with the alpha-beta transformation (extrapolated for isothermal transition at 661°C) varied between +0.2 and +0.6 per cent in heating and between -0.3 and -0.4 per cent in cooling. In transverse specimens the variation was between +0.2 and +0.35 per cent in heating and -0.2 and -0.25 per cent in cooling (see Fig. 3.5).

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The effects of repeated recycling¹⁵ are illustrated in Fig. 3.5. The first two cycles lead to longitudinal contraction and transverse expansion caused by increased isotropy of orientation of the individual crystals. The continued expansion resulting from repeated cycling of the longitudinal specimen can be explained in part by the roughening of the metal surface, but other unknown factors are also involved. Each pair of bars on the figure represents the change in length in mils per inch in a cycle, 25 to 750 to 25°C. Shaded portions indicate the changes in the transformation measured as if they were isothermal at 661°C.

Fig. 3.6—Specific heats and heat contents of uranium at elevated temperatures. —, specific heat. ---, heat content. (UNCLASSIFIED)

1.3 Specific Heat, Entropy, Heat Content, and Heats of Transition. The specific heats of uranium metal have been measured at low temperatures.²⁵ With these data in the region 15 to 300°K and the Debye function below 15°K, a value for the entropy of uranium at 25°C of 12.03 e.u. is obtained. It is interesting that the Dulong-Petit value of the atomic heat capacity is exceeded at temperatures as low as -73°C.

Specific heats in the region above room temperature have also been measured.²⁶ These, together with values of heat content, are given in Fig. 3.6 and Table 3.5.

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The alpha-beta transformation in uranium absorbs 665 cal/gram-atom, and the beta-gamma transformation absorbs 1165 cal/gram-atom;²⁶ the entropy of the first transformation is 0.71, and that of the second is 0.98 e.u.

Table 3.5—Specific Heats, Heat Contents, and Entropies of Uranium at High Temperatures

Temperatures		Specific heat, C_p , cal/(gram-atom)(°C)*	Heat content, ΔH , cal/gram-atom*	Entropy, ΔS , e.u.*
°C	°K			
25	298.1	6.6		12.03 ± 0.3
27	300	6.6	1,440	12.07
127	400	7.0	2,160	13.97
227	500	7.5	2,940	15.60
327	600	8.1	3,730	17.04
427	700	8.9	4,580	18.35
527	800	9.9	5,535	19.62
627	900	11.0		
662	935†	11.3	6,975	21.29
662	935‡		7,640	22.00
772	1,045‡	10.2	8,770	23.14
772	1,045§		9,940	24.12
827	1,100		10,440	24.55
927	1,200	9.2	11,350	25.38
1,027	1,300		12,260	26.12
1,102	1,375		13,000	26.8

* Values for ΔH are those measured by Moore,²⁶ with the addition of 1,440 cal as heat content at 300°K (calculated by integration of specific heat over the range 0 to 300°K, using Long's data.²⁵ The C_p and ΔS values were calculated by Simon.²⁷ There seems to be a slight discrepancy between the values for ΔH and for C_p at 300 to 500°C.

† Alpha.

‡ Beta.

§ Gamma.

1.4 Melting Point, Heat and Entropy of Fusion, Vapor Pressure, and Boiling Point. Prior to 1942 the melting point of uranium was thought to be about 1700°C,^{8,28} but the discovery that an oxide envelope forms around the molten metal led to the development of new techniques which give much lower values for the melting point.^{20,29-33} It seems generally agreed that the best value known for the melting point of uranium is $1132 \pm 1^\circ\text{C}$.

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It has been estimated²⁷ that the heat of fusion at the melting point is 2.5 to 3.0 kcal/mole, corresponding to an entropy of fusion of about 1.5 to 2.0 e.u.

Vapor-pressure measurements on uranium are of a preliminary character, and the experimental results scatter badly. Using a mean experimental value of 5×10^{-2} mm Hg determined by Creutz and Anderson^{34,35} at 2200°K and theoretical considerations, Simon²⁷ obtained the following equation for the vapor pressure of liquid uranium as a function of the temperature:

$$\log p \text{ (mm Hg)} = -\frac{100,000}{4.57T} - 2.04 \log T + 14.5$$

The boiling point, obtained by extrapolation, is about 3900°C, and the heat of vaporization at 1375°C is about 93 kcal/mole. Derge and Cefola³⁶ also made an experimental study of the vapor pressure of uranium by the orifice effusion method and obtained widely different results depending on whether a tantalum or a beryllia crucible was used as a container for the metal. Their results are summarized by the following equations:

$$\log p \text{ (mm Hg)} = -\frac{12,500}{T} + 3.3 \quad (\text{Ta crucible})$$

$$\log p \text{ (mm Hg)} = -\frac{30,000}{T} + 12.5 \quad (\text{BeO crucible})$$

The experimental results of all these investigations on the vapor pressure of uranium are summarized in Figs. 3.7 and 3.8.

1.5 Electrical and Thermal Conductivity of Uranium. Uranium is a poor conductor, its electrical conductivity being about one-half that of iron. Table 3.6 contains some typical data.

Because the alpha phase is anisotropic, its conductivity probably varies with crystallographic direction. This may account for some of the discrepancies in Table 3.6, although low purity undoubtedly is the main reason for the higher resistivity values found by several observers. Thompson^{16,39} plotted resistivity vs. temperature for the range 0 to 900°C. His curves, reproduced in Fig. 3.4, show sudden drops at both transformation points (alpha to beta and beta to gamma). The temperature coefficient of resistivity declines from 3×10^{-3} at room temperature to 0.5×10^{-3} at 650°C. It is as low as 0.14 to 0.18×10^{-3} (in the beta phase), but it increases again to 0.40 to 0.45×10^{-3} (in the gamma phase) at 300 to 900°C.

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Fig. 3.7—Vapor pressure of uranium at 1300 to 2300°C.^{27, 34-36} Sources:
 ○, Anderson. □, Creutz. ■, Simon. ▲, Derge and Cefola (tantalum crucibles).
 ●, Derge and Cefola (BeO crucibles).

Resistivity at low temperature has been discussed by Simon,²⁷ who estimated from the characteristic Debye temperature of uranium that the ratio of conductivities at 300 and 90°K should be 3 to 4, as against about 2 found in the earlier measurements, and he predicted that pure metal should have a conductivity of about 4×10^4 . This was confirmed by the measurements of Thompson. According to a more recent British investigation,⁴¹ between 13.9 and 373°K the resistivity of a uranium specimen can be represented by the equation

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Fig. 3.8—Vapor pressure of uranium.^{27,35,36} Sources: ■, Simon (semitheoretical values). ▲, Derge and Cefola (tantalum crucibles). ●, Derge and Cefola (BeO crucibles).

$$R_T = R_A + (1 - R_A) \frac{T}{273.2} G(\theta)$$

where R_T is the ratio of resistance at temperature $T(^{\circ}\text{K})$ to that at the ice point, and $G(\theta)$ is the Grueneisen function for a characteristic temperature of 175°K (as against $\theta = 162^{\circ}\text{K}$ derived from specific heat). The residual resistance for the sample investigated was given by $R_A = 0.194 + 0.000511T$. This resistance ($R_A = 0.19$) is much higher than can be accounted for by known impurities (0.3 per cent C, 0.6 per cent Fe, 0.19 per cent Cu, and 0.14 per cent Si) and must be

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attributed to oxygen (whose content was not determined) or to physical lattice disturbances.

Resistivity under high pressure was measured by Bridgman.⁴² Up to 12,000 atm it decreased at an average rate of 4.36×10^{-6} ohm/(ohm)(atm). The sample used was not particularly pure; its specific resistivity at 0°C was as high as 76×10^{-6} ohm-cm.

The thermal conductivity of uranium has been studied by a number of investigators, and selected data are reported in Table 3.7. In all probability thermal conductivity, too, depends on crystallographic

Table 3.6—Electrical Conductivity of Uranium

Specimen	Conductivity at 25°C, $10^4/\text{ohm-cm}$	Resistivity at 25°C, 10^{-6} ohm-cm	Temp. coef. of resistivity (0–100°C), 10^{-3} ohm/(ohm)(°C)	Observer
Wire	3.0 (23°C)	33		Driggs and Lilliendahl ⁸
Rolled wire, $\frac{1}{32}$ -in dia.	2.44	41		Snyder and Kamm ^{37,38}
Swaged wire, 1.5-mm dia., annealed in He for 10 min at 600°C	3.6–3.8	26.2–27.5	2.70–2.82	Thompson ^{16,39}
Cylinder, $2\frac{1}{4}$ -in. dia., annealed in He for 10 min at 600°C	4.0	25.0	2.73	Thompson ^{16,39}
Forges and turned bar of ICI metal	3.0 (23°C) 2.5 (100°C)	34 (20°C) 39.5 (100°C)		NPL ⁴⁰
ICI metal, rolled strip, 99.8–99.9% pure	3.1–3.2 (23°C)	31–32		Winnington ⁴⁰

direction. The temperature coefficient of conductivity is, according to Kratz,⁴⁶ 4.9×10^{-5} cal/(sec)(cm)(°C)².

The transfer of heat from the uranium slug to aluminum jackets or liners has been measured and has been found to be dependent on heat flux and thermal cycling of the slug assembly.⁵³

1.6 Thermoelectric Potential and Emissivity of Uranium. The thermoelectric potential between platinum and 99.9 per cent pure swaged uranium wire has been measured.⁵⁴ The wire was 0.033 in. in diameter and had been annealed for 10 min at 900°C. The results are given in Table 3.8. It is interesting that the emf-temperature curve, as contrasted to electrical resistivity, shows no discontinuities at the alpha-beta and beta-gamma transition temperatures.

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Table 3.7 — Thermal Conductivity of Uranium

Specimen	Temp., °C	Conductivity, 10^{-3} cal/(sec)(cm)(°C)	Observer
Cast metal	15–100	57	Snyder and
MH powder, sintered	35–75	37	Kamm ^{38,43}
Cast metal		58	Snyder and
			Kamm ^{38,43}
MH No. 1 sintered, not cold-pressed	60	57.4(±1.4%)	Plott and
			Raeth ^{44,45}
MH No. 2, (200 tons), cold-pressed	60	43.8(±1.1%)	
Fused cylinder			Plott and
13-cm dia.	63	45.7(±1.8%)	Raeth ^{44,45}
2.5-cm dia.	62	48.3(±0.4%)	
Commercially pure metal	50	57.5	Kratz et al. ⁴⁶⁻⁴⁹
	100	58.5	
	150	61.5	
	200	64.5	
	300	66.5	
	400	69	
Radial conductivity of extruded metal	84	62.3	Raeth and
	127	62.6	King ⁵⁰
	183	65.7	
	241	68.6	
Rolled, heated 2 hr at 850°C, water, quenched	31	62	Raeth ⁵¹
	47	65	
	66	67	
As-rolled	30	66	Raeth ⁵²
	59	68	
Rolled, heated 2 hr at 850°C, furnace-cooled to 200°C, quenched	28	66	Raeth ⁵²
	60	68	

Table 3.8 — Thermal Emf of Uranium Against Platinum

°C	mv	°C	mv
0	0.00	500	10.54
100	1.19	600	13.90
200	2.87	700	17.51
300	5.03	800	21.35
400	7.59	900	25.50

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The emissivity of uranium has been calculated¹⁷ by comparing the apparent temperature of the metal surface with the true temperature, the latter being determined by the emission from a hole in the metal which served as a black body. The emissivity was found to be 0.453 in the range 907 to 1050°C and 0.415 in the range 1052 to 1097°C. The sudden change between the two regions may indicate a crystal transformation. The low values of both emissivity coefficients are not consistent with attributing one of them to an oxide.

2. THE MECHANICAL PROPERTIES OF URANIUM METAL

During the past few years many of the mechanical properties of uranium have been investigated, together with the effect of previous treatment and alloying additions on these properties. Although these properties are well enough known for many practical applications, it would be desirable to repeat many of these investigations with uranium of greater purity and alloys of more definitely known compositions.

2.1 Hardness. The hardness of uranium is dependent on its previous heat and mechanical treatment; it also is influenced by the presence or absence of other elements, although this factor has not been studied extensively. Table 3.9 reports macrohardness values for differently treated specimens. Uranium cast at 1200°C may have a Rockwell B hardness as high as 100 on the surface, but the hardness declines to 84 Rockwell B at a depth of 0.05 in. (Example 2). The maximum hardness (100 to 115, Rockwell B) is obtained by quenching from 900°C in cold water, after keeping the metal at 900°C for 5 hr or more (Example 5). It has been suggested, but not proved, that this hardening may be associated with the solution of carbide.

Cold working by swaging or rolling increases the hardness, for example, from 91 to 114 Rockwell B (Example 7). Most of the hardening occurs while the thickness is reduced by the first 20 per cent; further rolling has comparatively little effect.

The influence of initial condition on the annealing of uranium has been studied in some detail.⁶⁷ In general, annealing reduces the hardness of cold work-hardened metal, beginning at 150°C; complete softening is achieved at 650 to 700°C (see Examples 9 to 12). Annealing in the beta region (greater than 660°C) approximately equalizes the properties of gamma-extruded and cold-worked metal, and it was therefore suggested²³ that the beta-annealed state should be considered as the "standard" state of uranium at room temperature.

Microhardness determinations on uranium by the methods of Knoop and Eberbach and their comparison with the results on the

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Table 3.9 — Hardness of Uranium

Example No.	Specimen and conditions	Rockwell				Brinell	Vickers Diamond Pyramid	Observer
		A	B	C	G			
Cast, Quenched, or Hot-worked								
1	0.04% Fe, 0.01% Al		95					Highriter and Lilliendahl ⁵⁵
2	Westinghouse metal cast at 1200°C							Creutz and Simmons ⁵⁶
	Slow-cooled 1/2 hr to 100°C							
	At surface		88.6					
	At 0.05-in. depth		87.5					
	Quenched 3–5 min to 100°C							
	At surface		100					
	At 0.05-in. depth		84					
3	Cast at Ames foundry		88–90					Wilhelm and Baker ⁵⁷
	Cast in BeO		91					
	Cast in alundum			28				
4	Cast metal, 1% Si					190–240	240	British observers ⁵⁸
	Cast metal, 1% Si, vacuum-annealed						300	
5	Gamma-extruded bar, 0.1% C, quenched in H ₂ O at 900°C after							Seybolt and Jette ⁵⁹
	2 hr	66–67						
	5 hr	70						
	8 hr	66–70						
	3/16-in. rods, 1 1/4-in. dia., quenched in H ₂ O at 900°C after	68–71						
	16 hr							

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Table 3 9—(Continued)

Example No	Specimen and conditions	Rockwell				Brinell	Vickers Diamond Pyramid	Observer
		A	B	C	G			
6	Two rods (see No 8)		92, 100					Van Echo and Foote ⁶⁰
	Quenched from							
	570°C		89; 98.5					
	645°C		91.5; 97					
	690°C		88.5					
	750°C		91					
	800°C		94.5					
	Furnace-cooled from 800°C		87.5					
Effect of Cold Working								
7	Cold-rolled							BMI ^{61,62}
	Before rolling	57	91					
	After scalping		101					
	Reduced by							
	9.3%		104					
	20.8%		107					
	46.5%		111					
	73.3%	71	114					
8	Rods, alpha-rolled at 620–650°C	54–63	92–102	10.5–25*	72–88	201–269		Van Echo and Plott ^{60,63}
	Same, at bottom of Brinell impression (apparent work hardness)				90–98			

Effect of Annealing

9	Cold-worked, heated to			BMI ⁶¹
	93°C	71	114	
	260°C	69		
	482°C	61	100	
	760°C	56	90	
10	927°C	56		
	Castings forged at 560°C,			Kaufmann ⁶⁴
	hot-rolled, pickled, and			
	cold-rolled (37% reduc-			
	tion). Annealed 1 hr in			
	vacuum at			
	115°C		36.6	
	310°C		36.6	
11	490°C		24	
	600°C		22	
	720°C		9	
	770°C		0	
	Alpha-rolled at 425°C	66		Kaufmann ⁶⁵
	Annealed 1/2 hr at 650-720°C	60		
	Alpha-rolled at 550°C	63		
12	Annealed at 650°C	57		
	Alpha-rolled at 640°C	56		
	Annealed at 650°C	55		
	Swaged disks about 0.034-in. dia.			Thompson ⁶⁶
	Transverse	100 ± 2	280	
	Longitudinal		325	
	Same, annealed at 625 or 830°C			
	Transverse	90 ± 2	220	
	Longitudinal		240	

*7.5-14 Rockwell F.

macrohardness numbers on the Rockwell, Vickers, and Brinell scales are reported in Table 3.10. The hardness values obtained in high-temperature tests are summarized in Table 3.11.

2.2 Elastic Deformation. Uranium is semiplastic, and its stress-strain curves often show definite curvature even at very low loading, as shown in Figs. 3.9 to 3.13, making the determination of elastic constants practically impossible. On the other hand, some curves have been obtained with a proportional limit as high as 50×10^3 psi, but they lead to a very low elastic modulus (about 5×10^6 psi), and it seems likely that the sensitivity of the apparatus used for these measurements was insufficient to reveal the curvature of the stress-strain curves at low stresses. This applies particularly to measurements at elevated temperatures (Table 3.14 and Fig. 3.14). Most investigations gave results intermediate between these two extremes, with a proportional limit of 10 to 15×10^3 psi (Figs. 3.9, 3.10, and 3.15); the elastic modulus derived from curves of this type is 20 to 25×10^6 psi, as shown in Table 3.12.

It is known that alpha uranium is anisotropic; thus its elastic constants probably are different in different crystallographic directions. This should be of importance, for example, for the propagation of high-frequency vibrations in uranium metal.

Selected values of the elastic modulus (Young's modulus) for uranium are given in Table 3.12. As is apparent from the data in Table 3.13, cold working does not change the elastic modulus very strongly.

The results of elasticity tests on uranium at elevated temperatures are given in Table 3.14.

Only a few values of the shear modulus of uranium are available. Creutz⁸⁷ reported 6.7×10^6 psi on material of uncertain previous history, and Snyder and Kamm^{38,43} reported values of 6.85×10^6 and 6.62×10^6 psi for cold-worked and annealed wire, respectively.

Poisson's ratio, the ratio of the contraction in the direction of stress to the dilatation perpendicular to the stress, can be calculated from two elastic constants, for example, from Young's modulus and the shear modulus. This ratio can, in turn, be used for the calculation of the bulk modulus which is the reciprocal of the volume compressibility. The results of such calculations are shown in Table 3.15. The high values obtained for Poisson's ratio may be another indication of the semiplastic nature of uranium metal.⁷

As a result of studies on the volume compressibility of uranium at pressures up to 12,000 kg/cm² (about 100,000 psi), Bridgman⁸⁸ reported 9.7×10^{-7} cm/kg as the average coefficient of compressibility.

2.3 Plastic Deformation. As is evident from Figs. 3.9 to 3.13, the stress-strain curves for alpha uranium bend gradually, and only

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Table 3 10—Uranium Hardness on Different Scales

Example No.	Material	Hardness											Observer
		Rockwell		Vickers		Brinell	Knoop				Eberbach		
		B	G	10-15 kg	30-45 kg		100 g	200 g	500 g	1000 g	~ 250 g	~ 630 g	
13	Hot-rolled rod Transverse Longitudinal							250 300					Thompson and Gray ⁶⁸
14	Disks from Hanford slug, extruded, outgassed	98		258	360	259	345			312	314	283	Thompson (NBS), Van Echo, and Foote ⁷⁰
15	Rod, alpha-rolled* (620-650°C)	92-102	71 5-88			201-269							Van Echo and Plott ⁶³
16	Original rod, cross section Swaged disks 0 375-0 017-in dia , annealed at 625°C after each 15% reduction, parallel and transverse						280 ± 100 380 ± 100						Thompson ⁶⁹
17	Alpha-rolled bar Transverse Longitudinal	97 98	80 5 83	220 240	250	240† 260‡		260 320	250 300	240 320	270 300	260 290	Thompson ⁷¹
18	Alpha-rolled bars§ Transverse Longitudinal	93 6-99 9 97 4-100.3	76-85 84-87 5	199-226 243-271		241-264 252-301					155-248 199-279		Gordon ⁷²

* 54-63 Rockwell A; 10.5-25 Rockwell C; 7.5-14 Rockwell F.

† Calculated from Rockwell B; 250 if calculated from Rockwell A

‡ Calculated from Rockwell B, 280 if calculated from Rockwell G.

§ Some were machined and others were annealed 12 hr at 300 to 600°C, but no systematic effect of annealing is recognizable, probably because the annealing temperature was not higher than the temperature of rolling

Table 3 11 — Uranium Hardness at High Temperatures

Example No.	Material and method	t_0 , °C	Test temp., °C	Brinell hardness	Observer
19	Alpha-extruded metal, heated to test temp., held for 15 min		20	252	BMI ⁷³
			200	144	
			400	66	
			500	33	
			600	19	
			650	13	
			690	36*	
			700	32*	
20	Alpha-extruded metal, heated to t_0 (β range) for 15–30 min, cooled to t , measured at once	700	600	19	BMI ⁷³
			735	18	
			735	21	
			735	24	
			700	49*	
21	Gamma-extruded metal		500	25	BMI ⁷³
			600	16	
			650	14	
			670	36*	
			690	31*	
			700	47*	
22	M.I.T. cast metal		500	50	BMI ⁷³
			600	28	
			650	22	
			670	31*	
			690	52*	
23	Mutual indentation method in argon		604	19	BMI ⁷⁴
			654	25	
			702	44*	
			753	16*	
			772	(-)†	
24	ICI Widnes metal		20	188	Sykes ²¹
			200	196	
			300	150	

* Beta.

† Gamma.

those for beta uranium show a definite yield point (18×10^3 psi at 700°C and 13×10^3 psi at 750°C). Because of the semiplastic nature of alpha uranium, the yield strength (namely, the stress at which the deviation of the strain from proportionality reaches 0.2 per cent, determined from the stress-strain curves) is not only low but also is not very easily reproduced. The value obtained is dependent upon the duration of the measurement and the whole previous history of the

Fig. 3.9—Tensile test of alpha-rolled uranium rods.⁶⁰ A, annealed for $\frac{1}{2}$ hr at 800°C and cooled down with furnace. B, annealed for $\frac{1}{2}$ hr at 800°C and water-quenched.

specimen. Furthermore, the unloading curves are not linear (see Fig. 3.11). It therefore is not surprising that the type of scattering indicated by the values reported in Table 3.16 is observed.

The plastic deformation of uranium under high compression has been observed.⁹² It was found that uranium cylinders could be com-

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Fig. 3.10 — Transverse and longitudinal tension stress-strain curves of gamma-extruded uranium.¹⁵ A, transverse contraction. B, longitudinal elongation.

Fig. 3.11 — Loading and unloading curves of gamma-extruded uranium.¹⁵ A, lateral contraction. B, longitudinal extension.

SECRET

pressed by 35 per cent without shattering; plastic deformation sets in very early, but no definite yield point could be observed. Similar results obtained by other investigators^{76,86} are given in Table 3.17 and Fig. 3.16.

Preliminary studies on the creep testing of uranium have been reported.⁹³⁻⁹⁵

Fig. 3.12—Stress-strain curve for swaged uranium rod annealed at 600°C.⁷⁵

2.4 Ultimate Strength. The ultimate tensile strength of uranium is of the same order of magnitude as that of steel. It varies between 25 and 100 tons/sq in., depending upon the amount of cold-work and previous heat-treatment of the specimen. Some typical results are given in Tables 3.18 to 3.21. Annealing in the alpha region (Examples 2, 3, 4, 8, 10, and 20) does not reduce the tensile strength to the same low value as is reached by annealing above 800°C.

Cast and gamma-extruded specimens (Examples 6, 11, and 13) have almost as low a tensile strength as those which were annealed and are not much affected by further annealing. The dependence of tensile strength on direction in rolled or extruded metal has not yet been investigated.

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Fig. 3.13—Tension stress-strain curves for cast and extruded uranium.⁷⁶
 —, cast. ---, extruded.

Some measurements of the tensile strength of uranium at elevated temperatures have been made:⁸⁹ Cast metal which had a tensile strength of 52.5×10^3 psi at 20°C showed strengths of 27.1×10^3 and 117×10^3 psi at 150 and 600°C , respectively. Hot-rolled material

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which possessed a tensile strength of 86.3×10^3 psi at 20°C showed a strength of only 12.2×10^3 psi at 600°C .

Values for percentage elongation and reduction in area obtained in ultimate-tensile-strength tests are given in Table 3.21.

Fig. 3.14—Compression tests of alpha-rolled uranium.⁸⁶ The samples were 1 in. in length by 0.505 in. in diameter. The test speed was 0.05 in./min.

The Charpy impact strength of uranium has been measured⁸⁷ on specimens cut from three ingots. Specimen sizes conformed to ASTM specifications as closely as was possible considering the comparatively poor machinability of the metal. On these three specimens impact strengths of 16.4, 12.9, and 14.0 ft-lb were obtained. It is in-

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interesting to note that British observers⁵⁸ have given 5 ft-lb as the impact strength of Widnes cast uranium.

2.5 Coefficient of Friction. The friction between machined graphite and scaly oxide-coated uranium and between machined graphite

Fig. 3.15—Compression stress-strain curves for cast and extruded uranium.¹² —, cast. ---, extruded.

and machined uranium has been measured.⁹⁸ The coefficient of friction was assumed to be equal to the tangent of the angle at which the metal piece begins to slide on the graphite surface (static friction), or at which it does not stop if brought to sliding (sliding friction). For unmachined uranium on graphite, the static coefficient was 0.556 and the sliding coefficient was 0.530. For machined uranium on graphite, coefficients of 0.211 and 0.200, respectively, were obtained.

SECRET

Table 3.12—Elastic Modulus of Cast Hot-worked, or Annealed Uranium*

Example No.	Material	Stress, psi	Elastic modulus, 10 ⁶ psi	Proportional limit, 10 ³ psi	Observer
1	Biscuit metal (as-cast), compression tests		22–25	15–18	Seybolt and Jette ⁷⁷
2	2-in.-dia. cast rod, compression tests		20–22	8–10	Seybolt ¹² (see Fig. 3.16)
	1 1/4-in.-dia. extruded rod, compression tests		22–26	8–10	
3	1-in.-dia. cast bar		20–21	14–15	Seybolt ⁷⁶
	1 3/16-in.-dia. extruded bar		21–23	13–15	
	1 1/2-in.-dia. extruded bar		22–25	13–15	
4	Bar forged at 815°C, quenched in H ₂ O		26(±5%)		Kaufmann ⁷⁸
5	UH metal, cast (25°C)		5.53		BMI ^{78,80}
	UH metal, hot-rolled (25°C)		5.39		
6	Gamma-extruded	5,000	24; 27†		BMI ⁸¹
	Outgassed	10,000	19; 23†		
	6 hr at 600°C	15,000	14; 18†		
7	Gamma-extruded, outgassed		25		BMI ⁸²
	Annealed 1 hr at 725°C and slow-cooled		23–25		
8	99.4% cast	20	20	11	Greenwood; ⁸³ British observers ⁵⁸
	ICI crude cast metal	26	26	11	
9	Annealed		23		Thompson ⁸⁴
	Swaged rods, annealed at 600°C		22–25	< 0.4	

* Tensile tests (if not otherwise indicated).

† Tangent values.

Table 3.13—Elastic Modulus of Cold-worked Uranium (Tensile Tests)

Example No.	Material	Elastic modulus, 10 ⁶ psi	Proportional limit, 10 ³ psi	Observer
10	Work-hardened wire (1/32 in. sq.)	15.8		Snyder and Kamm ^{56,43,85}
	Annealed wire	17.4		
11	Alpha-rolled (0.079% C)	19		Kaufmann ⁶⁵
	Alpha-rolled (0.26% C)	15		
12	Alpha-rolled, annealed 1/2 hr at 570°C, quenched	19	8	Foote and Van Echo ⁶⁰ (read values from Fig. 3.9)
	645°C, quenched	25	10	
	690°C, quenched	25	9	
	750°C, quenched	22.5	9	
	800°C, quenched	24.5	12	
	800°C, furnace-cooled	21	13	

SECRET

Table 3.14—Elastic Modulus of Uranium at Elevated Temperatures

Example No.	Material	Temp., °C	Elastic modulus, 10 ⁶ psi	Proportional limit, 10 ³ psi	Observer
13	MH cast metal	25	5.53		BMI ⁸⁰
		150	4.21		
		600	1.21		
14	Alpha-rolled metal	25	5.2	50	Van Echo ⁸⁶ (see Fig. 3.14)
		200	2.8	40	
		400	2.3	29	
		500	0.83	15	
		650	0.55	4	
		700*	1.30	17	
		750*	0.95	12	

* Beta.

Table 3.15—Poisson's Ratio and Bulk Modulus of Uranium

Material	Stress, psi	Poisson's ratio	Bulk modulus, × 10 ⁶ psi	Observer
Cold-worked wire		0.195	8.9	Snyder and Kamm ^{88,43}
Annealed wire		0.31	15.5	
Gamma-extruded	5,000	0.38; 0.38		BMI ⁸¹
Outgassed	10,000	0.39; 0.43		
(60°C, 6 hr)	15,000	0.39; 0.43		BMI ⁸²
Gamma-extruded, outgassed bar		0.35; 0.38		
Same, annealed 1 hr at 725°C, slow-cooled	5,000	0.35; 0.32		

SECRET

Table 3.16 — Yield Strength of Uranium

Example No.	Material	Yield strength, 10^3 psi		Observer
		0.1% offset	0.2% offset	
1	2-in.-dia. cast rod*		30.0	Seybolt ¹²
	1 $\frac{1}{4}$ -in.-dia. extruded rod*		32.7	
2	Commercially pure 1-in. cast rod		32.8; 33.8	Seybolt ¹⁶
	Commercially pure 1 $\frac{3}{16}$ -in. extruded bar		30.4; 34.6	
	Commercially pure 1 $\frac{1}{2}$ -in. extruded bar		33.8; 36.3	
3	Cast, measured at 20°C		43.0	BMI ⁸⁹
	Cast, measured at 150°C		Brittle	
	Cast, measured at 600°C		8.9	
4	As-extruded	48.8	57.4	BMI ⁹⁰
	Same, annealed 1 hr at 600°C	52.5	62.5	
5	Hot-rolled 1-in. bar from 40-lb UCC ingot, as-rolled	16.5		BMI ⁹¹
	Same, quenched from 750°C	47.5		
	Same, quenched from 1000°C	56.9		
	Same, annealed 3 hr at 600°C	38.8		
6	Gamma-extruded, outgassed bar		10.5–12.0	BMI ⁹²
	Same, annealed 1 hr at 725°C, slow-cooled		7.7–12.5	
7	ICI crude cast metal	35.0		British ⁵⁸
8	Two rods alpha-rolled at 600°C			Van Echo ⁶⁰
	Quenched from 570°C		31.8; 33.5	
	695°C		35.8	
	690°C		39.5	
	750°C		38.5	
	800°C		42.0	
	Furnace-cooled from 800°C		24.5	

* 15.0–17.0 $\times 10^3$ psi for 0.02 per cent offset.

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Table 3.17 — Compression Tests on Cylinder from Commercial Alpha-rolled Uranium

Temp., °C	Permanent compression*	
	By 10%, 10 ³ psi	By 20%, 10 ³ psi
25	110.5	
200	68.5	98.5
300	55.0	69.0
450	29.5	38.0
500	21.5	35.0
650	7.2	8.2
700†	17.0	
750†	12.0	13.5

* Permanent compression is caused by specimens becoming barrel-shaped.

† Beta.

Fig. 3.16 — Compression flow curve of commercially pure cast uranium.⁷⁶

SECRET

Table 3.18 — Effect of Cold-work and Annealing on Ultimate Tensile Strength of Uranium

Example No.	Specimen	Ultimate strength, 10 ³ psi	Observer
1	Before rolling	111–146	Kaufmann ⁶⁵
2	Alpha-rolled (50% flattening) at 800°F	205	
3	Alpha-rolled (50% flattening) at 920°F	196	
4	Alpha-rolled (50% flattening) at 1100°F	184	
5	Hard-rolled	192	Kaufmann ⁶⁴
6	Hot-rolled, annealed at 770°C	82	
7	Cold-swaged	160	Thompson ^{75,84}
8	Cold-swaged, annealed at 600°C	122–150	
	Hot-rolled 3/8-in. rod, swaged to 0.017 in. with intermediate anneals at 600°C		Thompson and Gray ⁶⁸
9	After swaging (20–25% reduction after last anneal)	170	OK
10	After annealing at 625°C	135	
11	After annealing at 830°C	65	
12	Cold-rolled (75% flattened) strip of Widnes metal, 0.015–0.020 in. thick	183	ICI ⁹⁶
13	Same strip, annealed 15 min at 800°C	70–78	
14	Alpha-rolled at 600°C	105	Van Echo and Foote ⁶⁰
	Heated to 800°C and furnace-cooled	57.5	

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Table 3.19 — Ultimate Tensile Strength of Hot-worked and Quenched Uranium

Example No.	Material	Ultimate tensile strength, 10^3 psi	Observer
15	Two alpha-rolled rods		Van Echo and Foote ⁶⁰
	As-rolled	105; 144.8	
	Quenched from		
	570°C	97.1; 118.4	
	645°C	96.9; 103.6	
	690°C	94.0	
	750°C	94.6	
	800°C	92.6	
16	Bar forged at 815°C and quenched in H ₂ O	98	Kaufmann ⁷⁸
17	Hot-swaged	125	Thompson ⁷⁵
	Hot-rolled 1-in. bar from UCC ingot		BMI ⁹¹
18	As-rolled	129	
19	Quenched from 1000°C	107	
20	Annealed 1 hr at 600°C	110	

Table 3.20 — Ultimate Tensile Strength of Cast and Gamma-extruded Uranium

Example No.	Material	Ultimate tensile strength, 10^3 psi	Observer
21	Commercially pure 1-in.-dia. cast	74; 82	Seybolt ⁷⁶
22	Gamma-extruded $1\frac{3}{16}$ -in. bar	66.5; 63.8; 67.1	
23	Gamma-extruded $1\frac{1}{2}$ -in. bar	76.8; 82.1	
24	Winnington and Widnes crude cast	64-90	ICI ⁹⁶
25	Gamma-extruded, outgassed bar	64-68	BMI ⁸²
26	Same, annealed 1 hr at 725°C, slow-cooled	61-64	
27	As-extruded	73; 80	BMI ⁸⁰
	Extruded, annealed 1 hr at 600°C	85

SECRET

Table 3.21 — Percentage Elongation and Reduction in Area in Ultimate-tensile-strength Tests

Example No.	Material (2-in. specimens)	Elongation, %	Area reduction, %	Observer
21	Commercially pure 1-in.-dia. cast	5; 10	9.5; 15	Seybolt ⁷⁶
22	Gamma-extruded 1 $\frac{3}{16}$ -in. bar	4.5; 4.5; 4.0	2.4; 5.8; 4.0	ICT ⁸⁶ <i>\$ory/4c</i>
23	Gamma-extruded 1 $\frac{1}{2}$ -in. bar	4.5; 4.5	2.4; 3.2	
12	Cold-rolled strip	0-3		
13	Cold-rolled strip annealed 15 min at 800°C	1-3		
24	Winnington and Widnes crude cast	0-3	0-4	BMT ⁸²
25	Gamma-extruded, outgassed	1.5	Difficult to observe be-	
26	Gamma-extruded, outgassed, annealed	5.0-6.0	cause of slip bands	
15	Two bars alpha-rolled (at 600°C) Heated and quenched from	19.0; 17.5	15.0; 13.0	Van Echo and Foote ⁶⁰
	570°C	22.2; 13.8	21.8; 12.7	
	645°C	20.6; 16.4	18.3; 16.6	
	690°C	10.3	9.6	
	750°C	9.8	8.7	
	800°C	8.6	8.6	
	Furnace-cooled from 800°C	7.0	10.6	

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Chapter 4

METALLOGRAPHIC TECHNIQUE FOR THE STUDY OF URANIUM AND ITS ALLOYS

By Frank G. Foote

The metallographic examination of specimens has always been an important technique in studies on the physical metallurgy of metals and alloys. Suitable techniques for the study of uranium and its alloys were not available at the time of the advent of the tremendous wartime interest in this metal. Consequently some of the early investigations of the physical metallurgists on the Project were directed toward finding suitable techniques for preparing specimens of uranium for metallographic examination. These techniques have been improved gradually during the past few years, and this chapter gives a brief account of the polishing and etching techniques which have been found most suitable.

1. POLISHING OF URANIUM AND ITS ALLOYS

Both mechanical and electrolytic polishing have been found useful. Satisfactory procedures are described in the following sections.

1.1 Mechanical Grinding and Polishing.¹ The grinding of uranium metallographic specimens may be carried out wet on "Wet-or-dry Tri-M-ite" (Minnesota Mining and Mfg. Co.) abrasive papers. The successive grit sizes used are 240, 400, and 600. The mechanical polishing of uranium metallographic specimens is best accomplished in two stages. Preliminary polishing is carried out with 600-grain carborundum on Selvyt polishing cloth at a wheel speed of 875 rpm, and final polishing is done with magnesium oxide and distilled water. Distilled water must be used to prevent the formation of basic magnesium carbonate. The procedure used is to wet the cloth with distilled water, apply the dry abrasive from a salt-shaker type of con-

tainer, and then apply more distilled water to form a paste with the abrasive. Still more water is applied at frequent intervals during polishing. The magnesium oxide recommended is Merck's Heavy, USP.

1.2 Electrolytic Polishing in Chromic Acetic Acid.² Unmounted uranium specimens are prepared for final electrolytic polishing by grinding through No. 180 grit and then wet-grinding by hand through No. 600 grit. Specimens are always freshly abraded on No. 600 grit just before electropolishing. The electropolishing solution consists of 45 g of chromic anhydride, 50 ml of water, and 350 ml of glacial acetic acid. The chromic anhydride is first dissolved in the water, and the solution is then added to the acetic acid. The proportions of the constituents are not critical and may be varied within ± 5 per cent without seriously affecting the results. For polishing, a current density of 10 to 15 amp/sq in. is applied for 5 to 10 sec, with the temperature of the solution being maintained in the range 60 to 80°F (15 to 27°C). A stainless-steel cathode rests on the bottom of the beaker, and the specimen, which is the anode, is suspended face down about 1 in. above the cathode. The potential drop across the cell is 60 to 80 volts. Inclusions are well retained during electropolishing in this bath.

1.3 Electrolytic Polishing in Phosphoric Acid.³ In preparation for electropolishing in this solution, the specimen is given a preliminary wet grinding through No. 400 grit paper. The mounted samples are drilled, and molten solder is poured into the hole to establish electrical contact between the specimen and the excess solder on the bottom of the mount. The polishing solution has the following composition: 85 per cent phosphoric acid, 5 parts; ethylene glycol, 5 parts; and ethyl alcohol, 8 parts. The cell is set up in a crystallizing dish provided with a glass cooling coil. A plate-graphite anode is placed on the bottom of the dish, and the specimen is placed on this plate with the surface to be polished face up. The cathode is another graphite disk placed about 1 cm above and parallel to the surface of the specimen. Satisfactory polishing may be obtained by the application of a current of 2.5 to 3.0 amp for 8 to 10 min. The potential required is about 24 volts, and the temperature of the solution should be kept below 35°C.

Alternate mechanical and electrolytic polishing may be used. In this procedure the specimen is first ground through 4/0 paper; it is then partially polished electrolytically in orthophosphoric acid-water or in orthophosphoric acid-glycerol solutions. The final polish is then obtained by 30 min of mechanical polishing with rouge.

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2. ETCHING OF URANIUM AND ITS ALLOYS

A variety of procedures for the chemical and electrolytic etching of uranium and its alloys have been developed. The exact procedure selected must depend somewhat on the type of specimen and the structural features which are to be developed by the treatment. Several studies³⁻⁵ have shown that the grain structures of electropolished and electroetched uranium specimens are well revealed by examination in polarized light.

Nitric acid (70 per cent) and glacial acetic acid are suitable reagents¹ for the chemical etching of uranium. Best results are usually obtained with an etching time in the range 3 to 6 min.

Hydrogen gas may be used as a chemical etchant.⁶ It readily reacts with uranium at 250°C but does not usually react with uranium compounds. A free-uranium phase can therefore be identified by observing the reaction of a specimen with hydrogen gas. A hot stage for hydrogen etching of metallographic samples has been described.⁷

Electrolytic etching has been applied successfully under a variety of circumstances. For example, the specimen may be made the anode in an electrolyte of 10 per cent oxalic acid in water.⁸ The current and time should be adjusted to give best results for the particular sample, but the use of a current density of 20 to 50 ma/cm² and an etching time of 5 to 10 min is typical. The substitution of a 10 per cent solution of citric acid for the oxalic acid solution yields similar results.⁹

Good grain-boundary delineation has been obtained¹⁰ by etching electrolytically at 200 ma/sq in. in an electrolyte consisting of 1 part ammonium hydroxide, 2 parts 30 per cent hydrogen peroxide, and 0.25 part 5N sodium hydroxide.

It also has been observed that etching can be obtained by using electrolytic polishing solutions^{2,3} if they are operated at lower current densities.

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SECRET

Chapter 5

BINARY ALLOYS OF URANIUM

By Robert W. Buzzard and Harold E. Cleaves

1. URANIUM AND ALUMINUM

The uranium-aluminum system has been fairly well established (Fig. 5.1) with the exception of the liquidus. The system contains three compounds: UAl_2 , UAl_3 , and UAl_5 . There are two eutectics in the system, one between gamma uranium and UAl_2 at 1105°C and one between UAl_5 and aluminum at 640°C .

The gamma-uranium eutectoid has been assumed from phase-rule considerations. The metallographic evidence for the solubility of aluminum in gamma uranium is conclusive, and alloys in the gamma-solubility range can be precipitation-hardened. The beta-gamma transformation temperature is lowered somewhat, but the alpha-beta transformation temperature does not appear to be affected by the presence of aluminum.

The compound UAl_2 is stable up to the melting point (approximately 1590°C), and it has a diamond cubic structure ($a_0 = 7.72 \text{ \AA}$). Both UAl_3 and UAl_5 decompose by a peritectic reaction, UAl_3 at 1350°C and UAl_5 at 730°C . The compound UAl_3 has a simple cubic structure ($a_0 = 4.26 \text{ \AA}$). The structure of UAl_5 has not been determined.¹⁻¹⁵

2. URANIUM AND ANTIMONY^{16,17}

Alloys containing 0.5, 2.6, and 5.0 wt. % antimony were made from biscuit metal in beryllia-lined alundum crucibles at 1200, 1205, and 1325°C , respectively. The structure of the 0.5 wt. % alloy consisted of a eutectic at the grain boundaries, precipitation of fine particles within the grains, and an angular phase. The 2.6 wt. % alloy showed segregation. The 5 wt. % alloy had the same phase as the 0.5 wt. % alloy plus a needlelike phase.

Fig. 5.1—Uranium-aluminum system. (UNCLASSIFIED)

The 5 wt. % alloy disintegrated in less than 24 hr in boiling distilled water. The approximate loss in weight of the 0.5 wt. % alloy was 5.5 mg/cm²/hr. Further data are not available.

3. URANIUM AND ARSENIC¹⁸⁻²⁴

Two compounds of arsenic and uranium have been identified, UAs and U₂As. UAs has a cubic structure ($a_0 = 5.76$ Å) with an x-ray density of 10.77 g/cm³. Further data are not available.

4. URANIUM AND BERYLLIUM

The major portion of the uranium-beryllium system (Fig. 5.2) has been fairly well established; only the metal-rich ends and the liquidus line remain doubtful. The system contains one compound having an ap-

SECRET

proximate composition of UBe_3 . The composition of the compound is uncertain by ± 3 per cent. There are two eutectics in the system, one at 1060°C between gamma uranium and the compound and one at 1200 to 1250°C between the compound and beryllium.

Fig. 5.2—Uranium-beryllium system. (UNCLASSIFIED)

The solubility of beryllium in gamma uranium has been assumed because of the lowering of the solidus and the appearance of the eutectic in the microstructure. The fact that the beta-gamma and alpha-beta transformation temperatures are lowered indicates some solubility of beryllium in both alpha and beta uranium. Although these solubilities have not been determined, they are known to be less than 10 at. %. The liquidus between 16 and 90 at. % beryllium has not been determined. A 90 at. % beryllium alloy gives a diffraction pattern corresponding to a simple cubic structure ($a_0 = 5.09 \text{ \AA}$). Precision

SECRET

parameter measurements indicate that the solid solubility of uranium in beryllium is exceedingly small, even at 1150°C. References: 1, 11, 14, 15, and 25 to 31.

5. URANIUM AND BISMUTH

The uranium-bismuth system (Fig. 5.3) has been partially established. The system contains two compounds: UBi and UBi_2 . X-ray

Fig. 5.3—Uranium-bismuth system. (UNCLASSIFIED)

studies have shown the presence of two phases other than uranium and bismuth in the system, but the complicated structures have not been worked out.

The melting point and transitions of uranium were unchanged by bismuth. The solid-solubility limits of the phases in each other have

SECRET

not been determined. Little evidence of solid solubility was found in the uranium-bismuth system. There appears to be an immiscibility gap between 4.9 and 43 per cent bismuth. At 4.9 per cent bismuth the two liquid phases appear immiscible, the heavier phase consisting of uranium containing 4 per cent bismuth and the upper layer containing 43 to 50 per cent bismuth. Bismuth distills from a 50 per cent alloy at 1650°C.

UBi melts above 1400°C, and UBi_2 decomposes peritectically at about 980°C. The pyrophoric nature of the alloys and the peritectic decomposition of UBi_2 make it quite difficult to obtain satisfactory microphotographs in the range between UBi and UBi_2 ; thus this region is somewhat uncertain. References: 1, 5, 8, 15, 27, and 32 to 39.

6. URANIUM AND CADMIUM

The high vapor pressure of cadmium prevents its addition to molten uranium. Two heats were made in which a slug of biscuit metal was soaked in molten cadmium at 650°C for 1- and 2-hr periods. In neither case was any evidence of alloying noted.⁴⁰

7. URANIUM AND CALCIUM

Very little work has been done in the uranium-calcium system. Compacts of uranium-calcium powders annealed at 800°C for 24 hr did not alloy. The samples were not pyrophoric and appeared to be a simple mixture of calcium and uranium with no reaction layers identified.⁴¹

8. URANIUM AND CARBON

The uranium-carbon system (Fig. 5.4) has been fairly well established up to 67 at. % carbon. Two compounds, UC and UC_2 , have been identified. The possibility of the existence of a higher carbide than UC_2 has been investigated, but no evidence that a higher carbide exists has been obtained.

It is reported that the solid-transformation temperatures of uranium are not affected by the presence of carbon, but other evidence is presented to show that carbon is soluble in gamma uranium to about 2 at. %, in beta uranium to about 0.6 at. %, and in alpha uranium to less than 0.2 at. %. Carbide inclusions in cast metal go into solution on prolonged annealing, indicating that the solubility of carbon in liquid uranium is less than it is in gamma uranium, which implies a peritectic reaction between UC and the liquid to form the solution of carbon in gamma uranium.

SECRET

Fig. 5.4—Uranium-carbon system. (UNCLASSIFIED)

The crystal structure of UC has been found to be cubic (NaCl type) ($a_0 = 4.948 \pm 0.001$ Å). The calculated density is 13.66 g/cm^3 , and the melting point is 2250°C . The variability of the UC x-ray spacing with oxygen and nitrogen impurities indicates that the carbon may be replaced by either nitrogen or oxygen in the UC structure. The com-

SECRET

Fig. 5.5—Uranium-chromium system. (UNCLASSIFIED)

pound UC_2 is tetragonal, with two molecules per unit cell ($a = 3.449 \pm 0.001 \text{ \AA}$, $c = 5.987 \pm 0.001 \text{ \AA}$, $c/a = 1.735$), the calculated density is 11.68 g/cm^3 , and the melting point is from 2350 to 2400°C . References: 1, 2, 5, 8, 23, 24, 25, 27, 32, 36, 37, and 42 to 58.

9. URANIUM AND CERIUM

Melts containing nominal amounts of 5.5 and 28.2 wt. % cerium were made. Investigators agree that the system consists in almost complete insolubility in both the liquid and the solid states. References: 16, 17, and 59 to 64.

10. URANIUM AND CHROMIUM

Only the uranium-rich end of the uranium-chromium system has been established (Fig. 5.5). The liquidus has not been established

SECRET

above 20 at. % chromium. Since the main interest of study was the uranium-rich alloys, the melting point and possible solid modifications of chromium were not investigated. There appears to be a compound in the region 40 to 45 at. % chromium and a second compound around 70 at. %. The second compound decomposes peritectically, and there are some indications of a compound between these two which also decomposes peritectically. Two eutectics exist, one in the neighborhood of 20 at. % chromium and the other around 50 at. %.

The maximum solubility of chromium in gamma uranium is estimated to be 4 at. % chromium. The gamma-beta transformation of uranium is lowered by chromium. The solubility of chromium in beta uranium is less than 2.5 at. %. There is little or no solubility of chromium in alpha uranium. Likewise there is little or no solid solubility of uranium in chromium.^{1, 65-70}

11. URANIUM AND COBALT

The uranium-cobalt system (Fig. 5.6) has been fairly well established. The system contains three compounds: U_6Co , UCo , and UCo_2 . There are two eutectics in the system, one at $734^\circ C$ and the other at $1063^\circ C$. Cobalt alloys are very hard and brittle and show no unusual resistance to corrosion.

There is no detectable solid solubility of either cobalt in uranium or uranium in cobalt.

The uranium liquidus is lowered rapidly by the addition of cobalt to a minimum of $734^\circ C$ at the eutectic composition, 34 at. % cobalt. The compound U_6Co (14.3 at. % cobalt) separates as a primary phase in alloys with less than 34 at. % cobalt. The compound U_6Co is a highly crystalline material formed by a peritectic reaction at $822^\circ C$. The compound is a body-centered tetragonal structure ($a = 10.34 \pm 0.02$ A, $c = 5.20 \pm 0.02$ A) with a calculated density of 17.7 g/cm³ and a unit-cell volume of 550 A³. The compound UCo decomposes peritectically at $805^\circ C$. It has a body-centered cubic structure ($a = 6.343 \pm 0.001$ A) with a unit-cell volume of 255.4 A³ and a calculated density of 15.4 g/cm³. UCo_2 has a face-centered cubic structure^{40, 65, 71} ($a = 6.9783 \pm 0.004$ A) with a calculated density of 13.83 g/cm³.

12. URANIUM AND COLUMBIUM

Information on the uranium-columbium system (Fig. 5.7) is incomplete. The principal emphasis has been concentrated on the uranium-rich alloys.

The maximum solubility of columbium in alpha uranium and beta uranium is less than 0.3 per cent above $600^\circ C$. The beta phase prob-

SECRET

ably decomposes eutectoidally at a temperature close to the gamma-eutectoid temperature. Columbium raises the freezing point of uranium and lowers the gamma-beta transformation. X-ray data on quenched alloys show the existence of alpha uranium in low-colum-

Fig. 5.6—Uranium-cobalt system. (UNCLASSIFIED)

bium alloys, and the gamma phase is shown in those containing 9 per cent or more columbium. Columbium additions in excess of 2 per cent increase the room-temperature stability of the gamma phase. With increasing columbium, the transformation of the gamma to the alpha plus columbium-rich phases becomes increasingly sluggish. Retention of gamma uranium by quenching is extremely difficult in alloys of less than 1 wt. % columbium. References: 16, 61 to 63, and 72 to 77.

SECRET

Fig. 5.7—Uranium-columbium system. (UNCLASSIFIED)

13. URANIUM AND COPPER

The uranium-copper system (Fig. 5.8) is relatively complete, although the portion of diagram between 7.76 and 83.3 at. % copper is somewhat tentative. The system contains one compound, UCu_5 . This compound forms a eutectic containing 92 at. % copper, melting at 1950°C. A monotectic is formed at 5.4 at. % copper and 1080°C. At this temperature, uranium separating from liquid containing 5.4 at.

SECRET

% copper shifts the composition of the liquid to 77.6 at. % copper. The boundary of the miscibility gap has not been determined, but a 61.6 at. % alloy quenched from 1700°C indicated miscibility at this temperature.

Fig. 5.8—Uranium-copper system. (UNCLASSIFIED)

The alpha-beta and the beta-gamma uranium-transformation temperatures are not affected by the presence of copper, indicating negligible solubility of copper in solid uranium. There is no evidence of the solubility of uranium in solid copper.

The compound UCu_5 has been established from x-ray data. Some microscopic evidence of the solubility of uranium in UCu_5 down to 80.9 at. % copper has not been substantiated. The compound UCu_5 has a face-centered cubic structure, with four molecules per unit cell

SECRET

($a_0 = 7.019 \pm 0.002$ A), an x-ray density of 10.61 g/cm^3 , and a measured density of 10.7 g/cm^3 . The compound oxidizes slowly in air and is extremely brittle. References: 1, 5, 8, 11, 22, 23, 36, 37, 52 to 54, and 78 to 81.

14. URANIUM AND GALLIUM

The microstructures and hardness values of alloys containing 2 at. % gallium have been investigated.^{66,82}

15. URANIUM AND GERMANIUM

Alloys containing 5 and 10 wt. % germanium were prepared using a procedure similar to that used in preparing manganese alloys. When the germanium was added, it appeared that a higher melting compound was formed. This caused the melt to freeze momentarily each time germanium was added.^{40,72}

16. URANIUM AND GOLD

Alloys with 2 at. % and 5 to 10 wt. % gold were prepared and studied. Some solubility of gold in alpha uranium was indicated. The 5 wt. % gold alloy, as-cast, showed a definite two-phase structure; the 10 wt. % alloy, as-cast, showed light particles of a gold-rich phase in a eutectic matrix. The gold alloys do not appear to have any unusual corrosion properties.^{40,66,82}

17. URANIUM AND HYDROGEN

Hydrogen reacts with uranium according to the reaction

the reaction being most rapid at 225°C . See Fig. 5.9 for the solubility of hydrogen in uranium.

Uranium hydride is a brownish-black or brownish-gray fine pyrophoric powder. The crystalline structure is cubic with eight uranium atoms per unit cell ($a_0 = 6.634 \pm 0.002$ A). It has an x-ray density of $10.91 \pm 0.05 \text{ g/cm}^3$.

Above the decomposition temperature of the hydride, 435°C , the capacity of uranium to dissolve hydrogen increases with temperature. The increase is particularly marked in the liquid state.

The equilibrium content of hydrogen in alpha uranium is about 2 ppm and changes little with temperature. The alpha-beta transformation at 660°C causes the solubility to increase from 2 to 8 ppm.

SECRET

The beta-gamma transformation increases the solubility to 15 ppm. Melting, which occurs at 1132°C, increases the solubility to 17 to 18 ppm, and the hydrogen content of the liquid metal continues to increase rapidly with temperature, reaching 30 ppm at 1250°C. In cast-

Fig. 5.9—Solubility of hydrogen in uranium in equilibrium with hydrogen at atmospheric pressure.

ing, a hydrogen content of 2 ppm is considered the danger limit. References: 1, 29, 49, 51, 54, 55, 57, and 83 to 96.

18. URANIUM AND INDIUM

Uranium-indium alloys with 5 to 10 wt. % indium were prepared.⁷² No results were reported.

19. URANIUM AND IRIIDIUM

Microstructures of an alloy containing 2 at. % iridium were studied.^{66,82}

20. URANIUM AND IRON

Data on the uranium-iron system are relatively complete (Fig. 5.10). The system contains two compounds, U_6Fe and UFe_2 . There

SECRET

are two eutectics in the system, one between U_6Fe and UFe_2 at 36 at. % iron and 725°C and the other between UFe_2 and iron at 1080°C. Phase-rule considerations and microscopic examination confirm the existence of a eutectoid somewhat below 1 at. % iron, the transformation occurring at 770°C.

Fig. 5.10—Uranium-iron system. (UNCLASSIFIED)

Iron is soluble in gamma uranium up to a maximum of 1.5 to 2.0 at. % at 815°C. The solubility of iron in both alpha and beta uranium appears negligible, as does the solubility of uranium in alpha iron. The solubility of uranium in gamma iron is limited; its actual extent has not been determined, but it is known to be less than $\frac{1}{2}$ at. % uranium. The details in the delta-iron region are doubtful, but the solubility of uranium in delta iron appears negligible. The alpha-

SECRET

gamma transformation appears at 910°C, but the data are somewhat inconsistent.

The compound UFe_2 has been identified from x-ray and microscopic analyses and is stable up to the melting point, 1235°C. The compound has a diamond cubic structure ($a = 7.004 \text{ \AA}$) and an x-ray density of 13.2 g/cm^3 . The compound U_6Fe has been identified from microscopic analysis. It decomposes peritectically at 815°C, and its crystal structure has not been established. References: 1, 9, 46, 65, 92, 97, and 98.

21. URANIUM AND LEAD

There is little information on the uranium-lead system. The alloys, in general, appear to be pyrophoric, and the microstructures are reported as being complex. There is some evidence of liquid immiscibility, a normal 50 at. % alloy having been reported to have formed two liquid layers. References: 1, 11, 27, 42, and 45.

22. URANIUM AND MAGNESIUM

Although some work has been done on the uranium-magnesium system, no definite conclusions are available. Attempts to prepare uranium-magnesium alloys have met with little success. There is some evidence that the solubility of magnesium in uranium is extremely low and that at least one and possibly two compounds are formed. References: 1, 8, 41, 49, 56, 78, and 79.

23. URANIUM AND MANGANESE

The uranium-manganese system is relatively complete (Fig. 5.11). The system contains two compounds, U_6Mn and UMn_2 . There are two eutectics in the system. One eutectic is formed between U_6Mn and UMn_2 at 21 at. % manganese and 716°C. A second eutectic exists at 85 at. % manganese between UMn_2 and manganese at 1035°C. The liquidus between 21 to 85 at. % manganese has been plotted above many of the actually determined points. The liquidus between 85 to 100 at. % manganese is fixed from two points, one of which lies considerably under the curve as plotted and, if considered, would reverse the curvature of the liquidus.

It has been established from microstructures that the maximum solubility of manganese in gamma uranium approximates 4 per cent. The solubility is less in the beta region, and there is a eutectoid at 1 at. % manganese at 626°C. There is no appreciable solubility of manganese in alpha uranium. The solubility of uranium in manganese has been investigated, but the results are inconclusive.

SECRET

The compound U_6Mn is formed by a peritectic reaction between beta uranium and liquid at 726°C . The compound is a body-centered tetragonal structure ($a = 10.265 \pm 0.01 \text{ \AA}$, $c = 5.23 \pm 0.02 \text{ \AA}$) with a unit volume of $550 \text{ \AA}^3$ and a calculated density of 17.8 g/cm^3 . The com-

Fig. 5.11—Uranium-manganese system. (UNCLASSIFIED)

pound UMn_2 is stable, with a melting point of 1120°C . The compound is a face-centered cubic structure^{99,100} ($a = 7.1484 \pm 0.001 \text{ \AA}$) with a calculated density of 12.57 g/cm^3 .

24. URANIUM AND MERCURY

The uranium-mercury system is apparently complete (Fig. 5.12). The system contains three compounds, UHg_2 , UHg_3 , and UHg_4 . The compound UHg_4 decomposes at 360°C to UHg_3 and mercury (vapor);

SECRET

UHg_3 decomposes at 390°C to UHg_2 and mercury (vapor); UHg_2 breaks down at 460°C into uranium plus mercury (vapor). Because of the pyrophoric nature of the amalgams, all work was done in vacuum or in an inert atmosphere.

Fig. 5.12 — Uranium-mercury system. (UNCLASSIFIED)

There appears to be some solubility of uranium in mercury and mercury in uranium but no mutual solubility between the compounds.

The compound UHg_2 has a hexagonal structure ($a = 4.77 \text{ \AA}$, $c = 3.21 \text{ \AA}$) with a calculated density of 15.29 g/cm^3 ; UHg_3 has a hexagonal close-packed structure ($a = 3.30 \text{ \AA}$, $c = 4.86 \text{ \AA}$) with a calculated density of 14.88 g/cm^3 ; and UHg_4 has a pseudo body-centered cubic structure ($a = 3.62 \text{ \AA}$) with a calculated density of 14.59 g/cm^3 . References: 1, 22, 37, 52 to 54, and 101.

SECRET

Fig. 5.13—Uranium-molybdenum system. (UNCLASSIFIED)

25. URANIUM AND MOLYBDENUM

The uranium-molybdenum system has been partially established (Fig. 5.13). The system is characterized by a peritectic type of liquidus and solidus, extensive solubility of molybdenum in uranium, the

SECRET

lowering of the transformation temperatures of uranium, and the absence of any intermetallic compound. In view of the very sluggish solid transformations, the phase boundaries in some regions have been drawn somewhat arbitrarily. The liquidus temperatures have not been located with certainty above 1285°C. Little work has been done on the molybdenum-rich side of the system because of the difficulty in preparing these alloys. The existing data indicate that the region is as postulated in the tentative diagram.

Solubility of molybdenum in gamma uranium, as determined by x-ray studies, varies from 35.8 at. % molybdenum at 1000°C to 29.9 at. % at 700°C. There appears to be no appreciable solubility of molybdenum in alpha uranium. The lattice parameter of the molybdenum phase corresponds to a solubility of approximately 1 at. % of uranium in molybdenum at 900°C. Microscopic studies indicate one- and two-phase alloys in the general ranges so indicated, but the exact limits of these transformations have not been determined. The peritectic reaction occurs at 1285°C and extends from 25 to approximately 95 at. % molybdenum.

X-ray diffraction patterns of the alloys in the gamma field show a body-centered cubic structure, the lattice parameter varying with the molybdenum content. The beta phase has been retained by quenching low-molybdenum alloys, its diffraction pattern indicating a complicated and, as yet, undetermined structure. References: 1, 16, 23, 38, 40, 65, 66, 72, 73, 78 to 80, and 102 to 111.

26. URANIUM AND NICKEL

The uranium-nickel system has been fairly well established. Figure 5.14 is based chiefly on thermal and microscopic studies since extensive segregation made interpretation of x-ray data somewhat uncertain. The system contains four compounds, U_6Ni , UNi , UNi_2 , and UNi_5 . There are two eutectics in the system, one between U_6Ni and UNi at 37 at. % nickel and 738°C and the other between UNi_5 and nickel at about 88 at. % nickel and 1104°C.

The lowering of the gamma-beta transformation in uranium and the microstructure of the low-nickel alloys indicates a eutectoid somewhere below 2 at. % nickel. The solubility of uranium in nickel and the mutual solubility of the compounds have not been determined.

The compound U_6Ni decomposes peritectically at 780°C. X-ray diffraction patterns of a 15 at. % nickel alloy indicate the existence of a face-centered cubic structure ($a_0 = 4.876 \text{ \AA}$).

The compound UNi decomposes peritectically at 810°C, and UNi_2 decomposes peritectically at 985°C. The peritectic reactions are clearly shown in the thermal data, but the composition has been as-

SECRET

signed on rather poor microscopic evidence. No definite evidence of the compound has been found in the x-ray patterns.

The melting point of UNi_5 is about 1295°C . The composition has

Fig. 5.14—Uranium-nickel system. (UNCLASSIFIED)

been determined with some certainty from microscopic examination. The x-ray diffraction pattern of a 90 at. % nickel alloy indicates a hexagonal structure ($a = 4.70 \text{ \AA}$, $c = 7.24 \text{ \AA}$), presumably UNi_5 . References: 1, 65, and 112 to 115.

27. URANIUM AND NITROGEN

No data are available on the solubility of nitrogen in uranium, and the melting point and dissociation temperature of the compounds have not been determined. The reaction between massive uranium and

SECRET

nitrogen becomes rapid above 450°C. Nitrides formed below 750°C and above 800°C, although of apparently the same composition, differ in visual appearance. At the lower temperatures a dark adherent scale with a metallic luster is formed; above 800°C the reaction is much more active, producing a dull nonadherent powder. The sharp break in the reaction rate is presumably associated with the beta-gamma transformation in the metal.

The uranium nitride phase with the lowest nitrogen content is the mononitride, UN. This mononitride sinters at 2300°C and melts in an atomic-hydrogen arc at about $2630 \pm 50^\circ\text{C}$. This compound is a face-centered cubic structure ($a = 4.881 \pm 0.001 \text{ \AA}$) with a calculated density of 14.31 g/cm^3 . Between uranium and UN there are no intermediate stoichiometric compounds, nor is there extensive solid solubility. Between UN and U_2N_3 the system is inhomogeneous, consisting of two separate phases, UN and U_2N_3 . The suggested structure of U_2N_3 is a body-centered cubic structure ($10.678 \text{ \AA}$) with a calculated

Table 5.1 — Lattice Constants of U_2N_3 to UN_2

Composition	Body-centered cubic, A	Face-centered cubic (pseudocell), A	Density, g/cm^3
UN 1.435	10.678 ± 0.005	5.339 ± 0.003	
UN 1.52	10.658 ± 0.005	5.329 ± 0.003	11.24
UN 1.75	10.580 ± 0.005	5.290 ± 0.003	
UN 2		5.31 ± 0.001	11.73

density of 11.24 g/cm^3 . From U_2N_3 up to the dinitride UN_2 , which is the third stoichiometric nitride of uranium, the system is homogeneous. With increasing nitrogen content the U_2N_3 crystal structure is transformed gradually, without discontinuity, into the UN_2 structure (see Table 5.1). References: 1, 2, 8, 10, 24, 29, 49, 57, 85, 89, 93, and 116 to 119.

28. URANIUM AND OSMIUM

Microstructures and hardness of a uranium-osmium alloy containing 2 at. % osmium were determined.^{66,82}

29. URANIUM AND OXYGEN

The uranium-oxygen system (Fig. 5.15) is incomplete. The common oxides of uranium appear to be UO_2 and U_3O_8 . Three other oxides or suboxides have been reported: UO , U_2O_5 , and UO_4 (see Table 5.2).

SECRET

Fig. 5.15—Uranium-oxygen system.

Table 5.2—Oxide Structures

Composition	Type	Lattice constants, A	Calculated density, g/cm ³
UO	Face-centered cubic	$a = 4.91$	14.2
UO ₂	Face-centered cubic (CaF ₂)	$a = 5.4568 \pm 0.0005$	10.97
U ₂ O ₅	Orthorhombic	$a = 6.72 \pm 0.01$ $b = 11.88 \pm 0.03$ $c = 8.26 \pm 0.02$	8.35
U ₃ O ₈	Orthorhombic	$a = 6.70 \pm 0.01$ $b = 3.98 \pm 0.01$ $c = 4.14 \pm 0.01$	8.41
UO ₃	Hexagonal	$a = 3.963 \pm 0.004$ $c = 4.160 \pm 0.008$	8.34
UO ₄ .3(?)H ₂ O	Orthorhombic	$a = 8.74 \pm 0.05$ $b = 6.50 \pm 0.03$ $c = 4.21 \pm 0.02$	4.90

SECRET

The solubility of O_2 in uranium is very small, even at temperatures above $2000^\circ C$. A solubility of 0.05 at. % at the melting point of uranium, rising to 0.1 per cent at $1400^\circ C$ and 0.4 per cent at $2000^\circ C$, is reported.

The solubility of oxygen in solid uranium is less, the microstructures indicating a solubility of less than 0.003 wt. % (0.04 at. %).

X-ray evidence indicates that UO exists and that the next higher oxide stable at room temperature is UO_2 . Further increase in the oxygen content decreases the UO_2 spacing, as though uranium were removed from the UO_2 structure. At compositions above UO_2 two phases appear, UO_2 with decreased spacing and U_2O_5 . The U_2O_5 structure is related to the U_3O_8 structure, and there is a gradual change in structure until the composition U_3O_8 is reached; only one phase is present between the compositions UO_2 and U_3O_8 . References: 6, 7, 9, 10, 48, 54, 98, 113, 117, 118, and 120 to 131.

30. URANIUM AND PALLADIUM

Information on the uranium-palladium system is rather meager. Apparently there is a eutectic at about 40 to 45 at. % palladium and $1000^\circ C$ and a new phase at about 75 at. %.

The temperatures of the alpha-beta and beta-gamma transformations of uranium appear unaffected by palladium. The solid solubility of palladium in uranium appears to be about 4 at. % palladium at $980^\circ C$. References: 40, 50, 60, 66, 82, and 132.

31. URANIUM AND PLATINUM

Hardness and microstructures of alloys with 5 wt. % and 2 at. % platinum were determined.⁶⁵⁻⁶⁷

32. URANIUM AND RHENIUM

The hardness and the microstructure of an alloy with 2 at. % rhenium were determined.^{66,82}

33. URANIUM AND RHODIUM

Little information is available on this system because the tests were terminated as a result of poor corrosion resistance.^{17,66,82}

34. URANIUM AND RUTHENIUM

Investigation was terminated because of poor corrosion properties. References: 17, 63, 66, and 82.

SECRET

35. URANIUM AND SILICON

The uranium-silicon alloy system (Fig. 5.16) has been fairly well established. The system contains four terminal solid solutions: alpha, beta, and gamma uranium and delta silicon; six intermediate phases: epsilon, which contains about 23 at. % silicon (the structure

Fig. 5.16—Uranium-silicon system. (UNCLASSIFIED)

and composition of this phase have not been satisfactorily ascertained), and U₅Si₃, U₂Si, USi₂, and USi₃. There are three eutectics in the system, one between gamma uranium and U₅Si₃ at 985°C, the second between U₅Si₃ and USi at 1570°C, and the third between delta silicon and USi₃ at 1315°C.

SECRET

The solubility ranges are very limited. Alpha uranium is stable up to 665°C and contains a negligible amount of silicon in solid solution. Beta uranium is stable between 665°C and 770 to 795°C, depending on the composition. Solubility of silicon in beta uranium is less than 1 at. %. Gamma uranium is stable between 770 to 795°C and 1125°C. It contains a maximum of 1.75 at. % silicon in solution at 980°C. Delta silicon melts at 1420°C and contains very little uranium in solid solution.

Epsilon, which forms by a peritectoid reaction between gamma uranium and U_5Si at 930°C, contains 23 at. % silicon. This phase has excellent resistance to corrosion. U_5Si_3 melts at 1665°C and forms a eutectic with USi at 1570°C. USi forms by a peritectic reaction between liquid and U_2Si_3 at 1575°C. The compound U_2Si_3 forms by a peritectic reaction between liquid and USi_2 at 1610°C. The melting point of USi is approximately 1700°C. Its crystal structure is body-centered tetragonal, containing four molecules per unit cell ($a = 3.97 \pm 0.03$ Å, $c = 13.76 \pm 0.08$ Å), with a calculated density of 8.98 g/cm³. The compound USi_3 is formed by a peritectic reaction between USi_2 and liquid at 1510°C. Its crystal structure is simple cubic ($a = 403$ Å). References: 1, 9 to 11, 117, 125, 128, 129, 133, and 134.

36. URANIUM AND SILVER

Silver alloys separate into two distinct phases: silver and uranium. No alloying effect was observed. Silver was lost by vaporization.^{38,65}

37. URANIUM AND STRONTIUM

Little information is available on this system.^{25,34,135}

38. URANIUM AND TANTALUM

The system (Fig. 5.17) consists of four solid phases: tantalum and alpha, beta, and gamma uranium; with a liquid solution of two metals above the liquidus line. No intermediate phases are present, and the solubility at both ends of the phase diagram is quite limited, less than 2 at. %. The solidus transformation is of the peritectic type and occurs at a temperature of 1175°C, about 50°C above the melting point of uranium. The alpha-beta and beta-gamma uranium-solid transformations appear at 680 and 760°C, respectively. The solubility of tantalum in liquid uranium is 19.5 at. % at 2000°C. No reliable results for the liquidus line were obtained above 2000°C. There is a complete absence of intermediate phases, and no evidence of intermetallic compounds was found.^{15,136}

SECRET

Fig. 5.17—Uranium-tantalum system. (UNCLASSIFIED)

UNCLASSIFIED

39. URANIUM AND THORIUM

The uranium-thorium system (Fig. 5.18) has been partially established, although the liquidus beyond 5 at. % thorium has not been located. One eutectic has been established at approximately 5 at. %

Fig. 5.18—Uranium-thorium system.

thorium and 1085°C. No evidence was found of the formation of intermetallic compounds or of the solid solubility of one component in the other. Very little is known definitely as to the degree of solubility of uranium in thorium; if any exists, it is estimated to be less than 2 at. %. References: 61, 62, 137, and 138.

40. URANIUM AND TIN

The uranium-tin system (Fig. 5.19) has been partially established. The extremely pyrophoric nature of most of the alloys has handicapped the investigation, but it is believed that three compounds exist in the system: U_5Sn_4 , U_3Sn_5 , and USn_3 .

Fig. 5.19—Uranium-tin system.

The evidence indicates no solubility of tin in uranium. The transformation temperatures of uranium do not appear to be affected by tin. There appear to be no material solubilities of the intermediate phases. The solubility of uranium in tin is less than 0.005 at. % at 600°C.

SECRET

The compounds U_5Sn_4 and U_3Sn_5 are proposed on the basis of thermal and microscopic data, but they oxidize so readily that positive identification has been impossible. Thermal data indicate that U_5Sn_4 is stable up to the melting point (approximately $1500^\circ C$) and that U_3Sn_5 decomposes peritectically at $1380^\circ C$.

The alloy USn_3 is not as pyrophoric as the lower tin alloys. It decomposes peritectically at $1350^\circ C$. It has a simple cubic structure ($a = 4.62 \text{ \AA}$) and a calculated density of 10 g/cm^3 . References: 1, 5, 8, 11, 22, 23, 27, 36, 37, 52 to 54, 78 to 80, 106, 116, and 139.

41. URANIUM AND TITANIUM

Few data are available for this system. The beta-gamma uranium transformation is lowered somewhat by small amounts of titanium, whereas the alpha-beta transformation is not appreciably altered. References: 40, 72 to 74, and 110.

42. URANIUM AND TUNGSTEN

The uranium-tungsten system (Fig. 5.20) consists of four phases: alpha, beta, and gamma uranium and tungsten. No evidence was found of the formation of intermetallic compounds. The liquidus line was established up to $2400^\circ C$.

The temperatures of the alpha-beta and beta-gamma uranium transformations are not altered by tungsten. The nature of the solidus transformation is in some doubt. The solidus line at $1135^\circ C$ is about $10^\circ C$ higher than the melting point found for uranium. The solidus transformation is tentatively described as a peritectic transformation.

The solid solubilities of tungsten in uranium and uranium in tungsten were found to be very limited. The solubility limits in both cases are probably less than 1 at. %. References: 10, 17, 126, 128, and 140.

43. URANIUM AND VANADIUM

Investigation of these alloys was terminated because of their poor resistance to corrosion. References: 16, 63, 82, and 141.

44. URANIUM AND ZINC

Preliminary work in the uranium-zinc system indicated that alloys containing more than 20 at. % uranium are difficult to prepare because of the distillation of zinc as the liquidus rises. Microscopic examination of high-zinc alloys shows a eutectic, apparently between

SECRET

Fig. 5.20—Uranium-tungsten system. (UNCLASSIFIED)

UNCLASSIFIED

90 and 100 at. % zinc. No information is available concerning the second phase involved in this eutectic or the number of compounds existing in the system. References: 1, 4, 8, and 22.

45. URANIUM AND ZIRCONIUM

Exploration of this system is incomplete. Results indicate that zirconium displaces the solid-state transformation considerably and that, above 5 at. % zirconium, two horizontals exist, one at approximately 675°C and the other at 605°C. These horizontals indicate the presence of two eutectoids. There appears to be no considerable solubility of zirconium in gamma uranium. References: 16, 17, 59, 60, 63, 132, and 142 to 145.

46. SUMMARY

The phase diagrams of the binary systems of uranium with aluminum, beryllium, carbon, chromium, cobalt, copper, iron, manganese, mercury, nickel, silicon, tantalum, tin, and tungsten have been established for the greater part, although information on some areas is incomplete.

Partial data exist for a number of other systems. Some alloys were studied principally in the hope of obtaining alloys with corrosion resistance superior to that of uranium itself. Only the alloys of uranium with columbium, molybdenum, silicon, and zirconium appear to have enhanced resistance to corrosion.

No substantial solubility of any metal in alpha uranium has been reported. However, a number of metals are soluble with the gamma phase. Solubility of uranium in most solid metals appears to be negligible.

A considerable number of intermetallic compounds of uranium have been identified. Compounds of the type U_6M are usually exceedingly brittle although not hard enough to scratch glass.

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Chapter 6

MECHANICAL PROPERTIES OF ALLOYS OF URANIUM

By A. B. Greninger

1. ALUMINUM-URANIUM ALLOYS

1.1 **Hardness.** The addition of aluminum to uranium increases the hardness in the as-cast condition as shown below:¹

Wt. % Al	Rockwell A hardness
0.0	55
3.7	62
6.3	67
8.0	68

A study of the effect of heat-treatment on the hardness of these low-aluminum alloys was made,² extensive data being obtained on an alloy containing 0.3 wt. % aluminum. This alloy was successfully forged from a 1-in.-diameter ingot to a 0.5-in.-square bar; the forging was done at a temperature in the upper alpha-uranium region. Cube specimens were then cut from this bar. After checking the as-cast hardness of the metal, all specimens were soaked at 1040°C in helium and were brine-quenched. There were indications that the metal was quite soft at the soaking temperature. Hardness measurements were made on the quenched, homogenized metal and after aging at various temperatures and times. The results of these tests are plotted in Figs. 6.1 and 6.2. Hardness measurements made on the as-quenched specimens were erratic and may have been subject to some error. However, the readings taken on the reheated specimens were considered to be accurate to ± 1 Rockwell C unit. The increase in hardness on further heating the alloy after quenching from 1040°C has been

Fig. 6.1—Precipitation hardening in an aluminum-uranium alloy (0.3 wt. % aluminum) quenched from 1040°C and reheated to indicated temperatures for $\frac{1}{2}$ hr.

Fig. 6.2—Precipitation hardening in an aluminum-uranium alloy (0.3 wt. % aluminum) quenched from 1040°C and reheated to 500°C.

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shown to be caused by the precipitation of UAl_2 . This compound begins to precipitate from solid solution at 700°C in the 0.3 per cent alloy.

A 1-in.-diameter ingot of an alloy containing 0.6 wt. % aluminum was made with the idea of subjecting it to a series of tests similar to those used for the 0.3 per cent alloy. This new alloy was too brittle to forge, and therefore samples were cut from the as-cast ingot. The as-cast hardness of this alloy was 24 to 26 Rockwell C. After quenching from 1040°C , the hardness increased to 35 to 36 Rockwell C. This quenched specimen contained quenching cracks, and further cracking and spalling occurred under the pressure of the Rockwell indenter. This brittleness was attributed to unremoved grain-boundary compound. The amount of undissolved compound was materially reduced by quenching from 1090°C , but traces of the grain-boundary compound still remained, and hence the alloy remained brittle. Reheating the two quenched specimens at 500°C for 0.75 and 2.5 hr produced hardnesses of 41 and 43 Rockwell C, respectively.

Additions of uranium to aluminum cause the resulting alloy to increase in hardness with increasing uranium contents. The hardness readings, given in Table 6.1, taken along three chill-cast aluminum-uranium ingots demonstrate this point.³

Several high-aluminum alloys were tested for possible age-hardening effects.⁴ The alloys were given a solution heat-treatment for 3 hr at 535°C . Samples were water-quenched and furnace-cooled from this temperature. The quenched alloys were aged for periods of 2, 4, 6, and 8 hr at 170°C to determine the effect of time at temperature on hardness. The Rockwell H values are given in Table 6.2.

1.2 Tensile Strength. Tensile-strength determinations were made on rolled aluminum-uranium alloys containing 4.95, 11.3, and 16.8 wt. % uranium.⁴ The alloys were tested as-rolled and after various heat-treatments. The results of these tests are recorded in Table 6.3.

1.3 Fabrication. Several rolling ingots of aluminum with additions of uranium were cast. The size of these ingots was 6 by 10 by 1.5 in. The melting and rolling data for these ingots are shown⁵ in Table 6.4. A comparison of the rolling of aluminum-uranium alloys with the rolling of common commercial alloys may be made by stating that the alloys with up to 18 wt. % uranium can be rolled on a commercial mill if rolling losses somewhat in excess of those usually encountered in industrial rolling can be tolerated.

Aluminum alloys containing 18.1, 20.3, and 21.2 wt. % uranium have been successfully alclad with 2S aluminum.⁴ This was done by rolling cast ingots of the alloys to $\frac{5}{8}$ in., followed by scalping and thorough grease removal. A sheet of 2S aluminum, which had been scratch-

SECRET

Table 6.1—Hardness Readings for Aluminum-Uranium Ingots

Wt. % U	Rockwell H hardness													
	Top of ingot			→										Bottom of ingot
2.8	47	53	55	54	58	59	40	43	47	40	41	52	52	
4.2	49	65	63	63	58.5	54	60	62	54.5	60	60	58	58	
15.2	79	73	77.5	75	79	77	79	76	78	79.5	78	76.5	76	

Table 6.2—Rockwell H Hardness of Heat-treated Aluminum-Uranium Alloys

Wt. % U	As-quenched from 535°C	Aged at 170° C			
		2 hr	4 hr	6 hr	8 hr
2.43	25	27-29	29-31	35-36	24
3.10	29-31	32-33	36-38	37-38	33
4.95	41-42	43-44	45-47	43	
7.00	53-54	57	55-56	56	
9.00	57-59	60-61	61	59-61	
11.3	61-62	64-65	64-65	65	
14.3	56	58	58-59	58-59	
16.3	58-59	60-62	61-62	62-63	
16.8	58-59	60-62	61-62	60	

Table 6.3—Tensile Properties of Heat-treated Aluminum-Uranium Alloys

Treatment	Property	4.95 wt. % U	11.3 wt. % U	16.8 wt. % U
As-rolled	Tensile strength, psi	22,900	25,600	24,400
	Yield strength, psi	17,400	19,250	19,500
	Elongation, %	7.4	4.5	4.0
	Rockwell H	81	87	84
Furnace-cooled after 3 hr at 535°C	Tensile strength, psi	14,000	18,100	18,000
	Yield strength, psi	5770	9700	8810
	Elongation, %	35	25	22
	Rockwell H	41	60	60
Water-quenched after 3 hr at 535°C	Tensile strength, psi	14,100	18,470	17,850
	Yield strength, psi	5580	9000	8300
	Elongation, %	36	25	25.3
	Rockwell H	40	63	61
Aged 6 hr at 170°C after quenching from 535°C	Tensile strength, psi	14,300	18,470	17,850
	Yield strength, psi	6590	9000	8500
	Elongation, %	32.5	25	21
	Rockwell H	41	60	58
Annealed 1 hr at 370°C	Rockwell H	54	70	61

SECRET

Table 6.4—Melting and Rolling Data for Aluminum-Uranium Alloys

Composition, wt % U		Pouring temp., °C	Hot rolling at 425°C	Cold rolling
Nominal	Analysis			
2.25	1.88	705	1.5 to 0.340 in., 12 passes, two reheats	0.340 to 0.80 in., four passes, one anneal
2.5	2.43	705	1.5 to 0.340 in., 12 passes, two reheats	0.340 to 0.80 in., four passes, one anneal
3.0	3.10	705	1.5 to 0.340 in., 12 passes, two reheats	0.340 to 0.80 in., four passes, one anneal
5.0	4.95	705	1.5 to 0.340 in., 12 passes, two reheats	0.340 to 0.80 in., four passes, one anneal
8.0	7.0	705	1.5 to 0.340 in., 18 passes, two reheats	0.340 to 0.080 in., seven passes, one anneal
10.0	9.0	705	1.5 to 0.340 in., 18 passes, two reheats	0.340 to 0.080 in., seven passes, one anneal
12.0	11.3	715	1.5 to 0.147 in., 36 passes, six reheats	0.147 to 0.080 in., five passes
14.0	14.3	715	1.5 to 0.147 in., 36 passes, six reheats	0.147 to 0.080 in., five passes
16.0	16.3	715	1.5 to 0.147 in., 36 passes, six reheats	0.147 to 0.080 in., five passes
18.0*	16.8	720	1.5 to 0.147 in., 36 passes, six reheats	0.147 to 0.080 in., five passes
18.0 A†	17.3	750	1.3/1.5 to 0.200 in., 39 passes, six reheats	0.200 to 0.080 in., five passes
B	18.1	750		
21.0 A	20.3	790	1.3/1.5 to 0.200 in., 39 passes, six reheats	0.200 to 0.080 in., five passes
B	20.4	790		
24.0 A	21.2	800	1.3/1.5 to 0.200 in., 39 passes, six reheats	0.200 to 0.080 in., five passes
B	23.8	800		
27.0 A	26.6	940	1.3/1.5 to 0.200 in., 39 passes, six reheats	0.200 to 0.080 in., five passes
B	27.7	940		
30.0 A	27.3	980	1.3/1.5 to 0.200 in., 39 passes, six reheats	0.200 to 0.080 in., five passes
B	29.1	980		

*Scalped at 0.63 in. to remove defects.

†All alloys, except 18 per cent, showed cross checking and edge cracking during hot rolling and were scalped again at about $\frac{5}{8}$ in. thickness, then were rolled without further cross checking, but edge cracks formed up to $\frac{1}{2}$ in. deep. The 0.200- to 0.080-in. reduction was started hot but can probably be regarded as cold rolling since the stock would not hold heat long at this thickness.

brushed and solvent-cleaned, was wrapped around the leading edge of the slab, and this assembly was heated to 425°C. The first pass was light, about 0.005 in., to seat the 2S aluminum to the slab. The second pass was also light, but it was somewhat heavier than the first, about 0.007 in. From this point, reductions of 0.025 to 0.030 in. per pass were made, using two reheats, to finish at 0.080 in. No blistering of the alclad coating was observed on subsequent annealing, and micro-examination showed satisfactory bonding.

2. ANTIMONY-URANIUM ALLOYS

Hardness values of 88 Rockwell B have been reported for cast antimony-uranium alloys containing 0.5, 2.6, and 5.0 wt. % antimony.³

SECRET

3. BERYLLIUM-URANIUM ALLOYS

Vickers Diamond Pyramid hardness values have been reported for beryllium-uranium alloys of a number of compositions.⁶ The specimens were studied both in the as-cast condition and after being an-

Fig. 6.3—Effect of beryllium additions on the hardness of uranium (Vickers Diamond Pyramid numbers). (a) As-cast alloys. (b) Alloys annealed at 900°C for 3 hr.

nealed at 900°C for 3 hr. These measurements are plotted in Fig. 6.3. The marked increase in hardness at about 90 at. % beryllium is believed to be due to an intermetallic compound at about that composition.

4. CARBON-URANIUM ALLOYS

Studies on carbon-uranium alloys, initiated because of the wide adoption of graphite crucibles for melting uranium, have shown that carbon has a definite hardening effect on the metal.

The usual way of preparing various carbon-uranium alloys has been by melting the metal in a graphite crucible, the desired final carbon content being regulated by means of the maximum temperature which the melt was allowed to reach.

The data in Table 6.5, which are plotted in Fig. 6.4, were obtained on one series of carbon alloys.⁷ The results of tests on another series

SECRET

Table 6.5—Data on the Preparation of Carbon-Uranium Alloys

Alloy No.	Casting temp., °C	Time at max. temp., min	Wt. % C	Rockwell B hardness
V	1400	60	0.06	88
IV	1400	20	0.08	88
VIII*	1400	15	0.168	91
VI	1750	20	0.25	95
VII†	1600	20	0.30	95
II	1650	20	0.70	99
IX	1800	20	1.99	111‡
I	1850	20	7.6	
X	1500	60		

*Closed crucible used.

†Powdered graphite charged with the metal.

‡Converted from Rockwell A.

Fig. 6.4—Effect of carbon content on the hardness of uranium (Rockwell B).

SECRET

Table 6.6—Data on Carbon-Uranium Alloys

Specimen No.	Temp. range, °C	Wt. % C	Rockwell A hardness
1	1350–1400	0.10	
2	1450–1500	0.17	52.8
3	1550–1600	0.36	54.8
4	1650–1700	0.42	56.0
5	1750–1800	0.51	58.7
6	1850–1900	1.60	63.1
7	1950–2000	1.74	63.8
8	2080–2130	2.92	63.6

Fig. 6.5—Effect of carbon content on the hardness of uranium (Rockwell A).

of carbon-uranium alloys⁸ are given in Table 6.6. The hardness values given in Table 6.6 are averages of wide variations; these variations were reported to have been caused by carbide segregation in the samples. These data are presented graphically in Fig. 6.5.

5. CERIUM-URANIUM ALLOYS

The hardness data have been reported for as-cast cerium-uranium alloys³ and are presented in Table 6.7.

SECRET

Table 6.7—Hardness Data for Cerium-Uranium Alloys

Nominal composition, wt. % Ce	Analysis	Rockwell B hardness
0.5	Ce, 0.52–0.53%; C, 100–230 ppm; Be, 8–15 ppm	82
3.0		96

6. CHROMIUM-URANIUM ALLOYS

A nominal 5 wt. % chromium-uranium alloy was tested for hardness both in the as-cast condition and after various heat-treatments.⁹ This alloy analyzed 3.2 wt. % chromium at the top and 1.5 wt. % at the bottom. The hardness data obtained on this alloy are as follows:

Heat-treatment	Rockwell A hardness
As-cast	64
2 hr at 800°C, water-quenched	72
Reheated 2 hr at 300°C, water-quenched	72
Reheated 2 hr at 400°C, water-quenched	71
Reheated 2 hr at 500°C, water-quenched	68
Reheated 2 hr at 600°C, water-quenched	60
Reheated 2 hr at 700°C, water-quenched	67

A $\frac{9}{16}$ -in.-diameter 2-in.-long rod of nominal 0.45 wt. % alloy was cast. This alloy analyzed 0.41 to 0.55 wt. % chromium, averaging 0.45 wt. %. The hardness measurements made on this alloy gave the results listed in Table 6.8.

7. COBALT-URANIUM ALLOYS

Cobalt-uranium alloys are extremely brittle. Alloys between 1.3 and 2.7 wt. % were prepared for hardness measurements, but they have always been so brittle that hardness tests could not successfully be made on them. Furthermore, these alloys have not shown any appreciable response to heat-treatment.^{9,12}

8. COLUMBIUM-URANIUM ALLOYS

8.1 Hardness. The effects of composition and heat-treatment on the hardness of columbium-uranium alloys have been studied in con-

SECRET

Table 6.8—Hardness Data for Chromium-Uranium Alloys

Heat-treatment	Rockwell A hardness	Eberbach hardness, D.P.N.	Rockwell A from Eberbach
As-cast alloy ¹⁰	69	425*	73
Air-cooled from 900°C, followed by 24 hr at 600°C, water-quenched ¹¹	59	308	64
Water-quenched from 900°C	66		
Reheated at 300°C, water-quenched	66		
Reheated at 400°C, water-quenched	68		
Reheated at 500°C, water-quenched	64		
Reheated at 600°C, water-quenched	57		

*Matrix.

siderable detail.¹³ This information is presented graphically in Figs. 6.6 to 6.9.

Columbium is extensively soluble in gamma uranium and tends to stabilize the gamma phases; the ease with which the gamma phase can be retained increases with increasing columbium content. Small additions of columbium increase the hardness of gamma-quenched alloys as shown in Fig. 6.6. In this range martensitic microstructures have been observed. In the range 2 to 9 wt. % columbium there is a progressive increase in the ease with which the soft-gamma phase can be retained by quenching. In the range 9 to 14 wt. % columbium there is a marked increase in hardness, presumably as a result of solid-solution hardening. Beyond 14 wt. % columbium the hardness is independent of composition.

Heat-treatments for two weeks at 500 and 600°C, after homogenizing for 2 hr at 900°C and water quenching, revealed a drop in hardness in the 4 wt. % alloy and an increase in hardness with increasing columbium contents (Fig. 6.7). Tempering at 600°C produced softer alloys in all compositions up to 20 wt. % columbium than did tempering at 500°C. The suggestion that tempering at 500°C produces maximum hardness in columbium alloys is further substantiated by Fig. 6.8, in which the hardness of a 5 wt. % columbium alloy is plotted as a function of the tempering temperature.

The effect of the columbium content on the hardness of hot-rolled and heat-treated columbium-uranium alloys is plotted for three heat-treatments in Fig. 6.9.

8.2 Tensile Properties. Billets of nominal 2, 4, and 6 wt. % columbium-uranium alloys were hot-rolled into rods.¹³ The billets

SECRET

Fig. 6.6—Columbium-uranium alloys homogenized as shown. Curve A, the result of heating the alloy 1 hr at 1000°C and then water-quenching it. Curve B, the result of heating 69 hr at 850°C and then water-quenching.

Fig. 6.7—Columbium-uranium alloys homogenized 2 hr at 900°C, water-quenched, and aged as shown. Curve A, aged two weeks at 500°C. Curve B, aged two weeks at 600°C.

SECRET

Fig. 6.8—Columbium-uranium alloy (5 wt. % columbium) homogenized for 2 hr at 900°C, water-quenched, and tempered for 2 hr at indicated temperatures.

Fig. 6.9—Effect of heat-treatment on hot-rolled columbium-uranium alloys. The conditions were as follows: Curve A, heated 2 hr at 850°C and water-quenched. Curve B, heated 2 hr at 850°C and water-quenched, tempered for 24 hr at 350°C, and water-quenched again. Curve C, heated 2 hr at 850°C and furnace-cooled.

SECRET

were 2 in. in diameter, weighing between 25 and 35 lb, and were hot-rolled at 1450°F (790°C). The 2 and 4 wt. % alloys were rolled to 0.5-in. rounds, but the 6 wt. % alloy stuck in the $\frac{5}{8}$ -in. oval pass and could be rolled no further.

Tensile specimens were machined from sections of the rods after they had been subjected to the following heat-treatments: (1) 2 hr at 850°C, water-quenched; (2) 2 hr at 850°C, furnace-cooled; and (3) 2 hr at 850°C, water-quenched, then tempered for 24 hr at 350°C, water-quenched.

The results of the tensile tests which were run on the finished specimens have been reported in detail elsewhere.¹³ A summary of the results of the tests is presented graphically in Fig. 6.10, and from these curves and experimental observations the following conclusions may be drawn:

1. The ductility of columbium-uranium alloys, as indicated by the elongation and reduction in area of the tested specimens, increased with increasing columbium content when the alloys were quenched from the gamma region. This observation, together with the concurrent decrease in tensile strength, indicates that the stability of the gamma-uranium phase was enhanced by increased columbium content.

2. Furnace cooling from the gamma region caused a decrease in ductility and an increase in strength with higher columbium content. This indicates that, during this heat-treatment, decomposition of the gamma phase occurred in the low-columbium alloys. In the higher columbium alloys the transformation is more sluggish, and even furnace cooling produces martensitic or pearlitic structures of considerable strength.

3. Tempering for 24 hr at 350°C after water quenching from 850°C caused a marked increase in tensile strength and a decrease in ductility of the alloys containing over 2 wt. % columbium. The properties of the 2 wt. % columbium alloy were about the same before and after tempering, indicating again that this alloy was transformed from the gamma to the alpha condition during quenching from the gamma region. The higher columbium alloys show a notable increase in strength as the result of tempering.

8.3 Compression Properties. Hot-compression tests were conducted on 6.5 and 10.5 wt. % columbium-uranium alloys up to a temperature of 1050°C.¹³ Tests were made on these alloys both as-cast and after a homogenizing anneal of one week at 1000°C. The results of the compression tests on columbium alloys are shown graphically in Figs. 6.11 to 6.14. The homogenizing anneal had no effect upon the hot-compression strength. With increasing temperatures the yield-point stress of the 6.5 wt. % columbium alloy was found to drop rapidly

SECRET

Fig. 6.10—Effect of columbium content on the tensile properties of a hot-rolled uranium rod annealed at 850°C for 2 hr. ---, reheated to 350°C. ----, furnace-cooled. —, water-quenched.

to a value of about 6000 psi in the temperature range 600 to 700°C. From 700 to 1000°C, in the gamma-uranium region, the yield point remained fairly constant at about 5000 psi. In the case of the 10.5 wt. % columbium alloy, the decrease in stress with increase in temperature was gradual and did not drop below 10,000 psi until a tempera-

SECRET

Fig. 6.11—Compressive strength of a cast columbium-uranium alloy (6.5 wt. % columbium) at elevated temperatures.

ture of approximately 1000°C was reached. It was suggested that the absence of a sudden change in stress of the specimen after it had passed out of the alpha-temperature region may have been due to a sluggish phase transformation in this higher columbium alloy. Microscopic examination of specimens which had been heat-treated for two days also gave evidence of slow transformation.

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Fig. 6.12—Compressive strength of a cast and annealed columbium-uranium alloy (6.5 wt. % columbium) at elevated temperatures. The alloy was annealed for one week at 1000°C and then was water-quenched.

Although the results of the hot-compression tests are relative, the indicated changes in plasticity with temperature do give useful information. Figure 6.11, in which stress vs. temperature for the as-cast 6.5 wt. % columbium alloy is plotted, shows that a temperature above 700°C would be desirable for the hot working of this alloy. Figure 6.13

SECRET

Fig. 6.13—Compressive strength of a cast columbium-uranium alloy (10.5 wt. % columbium) at elevated temperatures.

Fig. 6.14—Compressive strength of a cast and annealed columbium-uranium alloy (10.5 wt. % columbium) at elevated temperatures. The alloy was annealed for one week at 1000°C and then was water-quenched.

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reveals that hot working of the 10.5 wt. % columbium alloy would be difficult at temperatures below 1000°C.

9. COLUMBIUM-ZIRCONIUM-URANIUM ALLOYS

In an effort to obtain information concerning the transformations which occur on heat-treating columbium-zirconium-uranium ternary

Table 6.9—Hardness Data for Columbium-Zirconium-Uranium Alloys

Nominal composition		Rockwell C hardness number*			
Wt. % Cb	Wt. % Zr	I	II	III	IV
2	2	35	53	30	35
2	4	31	55	30	31
2	6	32	52	31	33
4	2	20	30	30	30
4	4	20	46	30	31
6	2	90†	21	38	31

*The conditions for these tests were as follows:

I. Heat-treated: homogenized one week at 1000°C and water-quenched.

II. Heat-treated (after homogenizing treatment I) at 850°C for 256 hr and air-cooled.

III. Heat-treated (after homogenizing treatment I) at 600°C for 64 hr and air-cooled.

IV. Heat-treated (after homogenizing treatment I) at 600°C for 256 hr and air-cooled.

†Rockwell B.

alloys, hardness data were compiled.¹⁴ These data are given in Table 6.9.

10. COPPER-URANIUM ALLOYS

Additions of uranium to copper have been found to have a definite hardening effect on the copper.¹ Hardness values for a series of as-cast copper-uranium alloys in the miscible liquid area of the phase diagram are as follows:¹⁵

SECRET

Wt. % Cu	Rockwell A hardness
91	29.3
75.5	49.8
68.5	49.5
63.8	63.4
58.6	74.5
56.8	72.1
55.9	74.6
50.9	74.2
48.4	75.0
0.7	66.2

The hardness of a nominal 5 wt. % copper alloy has been measured after various heat-treatments. This alloy analyzed 1.4 wt. % copper at the top and 1.5 wt. % at the bottom. Measurements yielded:⁹

Heat-treatment	Rockwell A hardness
As-cast	56
2 hr at 900°C, water-quenched	59
Reheated to 300°C for 2 hr, water-quenched	60
Reheated to 400°C for 2 hr, water-quenched	60
Reheated to 500°C for 2 hr, water-quenched	58
Reheated to 600°C for 2 hr, water-quenched	58
Reheated to 700°C for 2 hr, water-quenched	58

11. GALLIUM-URANIUM ALLOYS

A nominal 0.6 wt. % gallium-uranium alloy was tested for hardness after various heat-treatments.¹⁶ This alloy analyzed 0.30 wt. % gallium at the top and 0.74 wt. % at the bottom. Hardness data are as follows:

Heat-treatment	Rockwell A hardness
As-cast	63
2 hr at 900°C, water-quenched	62
Reheated to 800°C, water-quenched	64
Reheated to 700°C, water-quenched	61
Reheated to 600°C, water-quenched	56
Reheated to 500°C, water-quenched	61
Reheated to 400°C, water-quenched	63

This alloy showed cracking when it was quenched from 900°C.

SECRET

12. GOLD-URANIUM ALLOYS

A nominal 1.0 wt. % gold-uranium alloy was prepared for a hardness and heat-treatment study.¹⁶ This alloy contained, by analysis, 1.40 wt. % gold at the top and 1.18 wt. % gold at the bottom of the ingot. The information obtained by this work is as follows:

Heat-treatment	Rockwell A hardness
As-cast	63
2 hr at 900°C, water-quenched	65
Reheated to 800°C, water-quenched	64
Reheated to 700°C, water-quenched	56
Reheated to 600°C, water-quenched	60
Reheated to 500°C, water-quenched	69
Reheated to 400°C, water-quenched	70

The alloy was observed to crack when quenched from 900°C.

13. IRIIDIUM-URANIUM ALLOYS

A 1-g melt of a nominal 1.6 wt. % iridium-uranium alloy was found to have an Eberbach microhardness (D.P.N.) of 440. This sample was quenched from 900°C before the hardness measurements were made.¹⁰

A larger melt of the same alloy was made for further hardness study.¹⁶ This alloy had an actual analysis of 1.40 wt. % iridium at the top and 1.24 wt. % at the bottom. The hardness data obtained on these alloys are as follows:

Heat-treatment	Rockwell A hardness
As-cast	72
2 hr at 900°C, water-quenched	65
Reheated to 800°C, water-quenched	63
Reheated to 700°C, water-quenched	63
Reheated to 600°C, water-quenched	59
Reheated to 500°C, water-quenched	60
Reheated to 400°C, water-quenched	69

The alloy was cracked in the as-cast condition and also after quenching from 900°C.

SECRET

14. IRON-URANIUM ALLOYS

An attempt was made to prepare a 5 wt. % iron-uranium alloy for hardness measurement.⁹ However, this alloy was too brittle to be prepared for a hardness specimen, and hence no hardness values were reported.

15. LEAD-URANIUM ALLOYS

Tests on 0.5 wt. % as-cast lead-uranium billets have shown that this alloy can be easily forged at 1000°C and that it is somewhat easier to machine than pure commercial uranium.¹⁷

16. MANGANESE-URANIUM ALLOYS

A sample of a nominal 5 wt. % manganese-uranium alloy was made for hardness study.⁹ The top of this ingot analyzed 8.8 wt. % manganese, and no analysis was reported for the bottom of the alloy. The alloy was found to have a hardness of 65 Rockwell A in the as-cast condition. Since melting occurred when attempts to heat the alloy at 800 and 900°C were made, no hardness values after heat-treatment were reported.

17. MOLYBDENUM-URANIUM ALLOYS

17.1 Hardness. The effect of quenching from 900°C and reheating for 2-hr periods at temperatures from 300 to 800°C on the hardness of a series of molybdenum-uranium alloys is shown in Table 6.10.

The highest hardness values were obtained by reheating the 900°C quenched alloys at 400°C. The comparatively high hardness of the 1.09 and 2.26 wt. % molybdenum alloys as-quenched from 900°C was probably due to the partial transformation of the gamma-uranium phase to the alpha phase. The alloys of higher molybdenum contents were observed to be softer when given this same quenching treatment, thus indicating the greater stability of the gamma phase in these alloys. A sample of the 2.26 wt. % molybdenum alloy gave¹⁸ a Rockwell A reading of 77 when quenched from 700°C and aged for 1 hr at 400°C. This demonstrated that, in this case at least, the 700°C quench was just as effective as the 900°C quench when both were followed by a 400°C temper.

The data listed in Table 6.11 were obtained in order to gain information concerning the effect of the time of tempering on some of the molybdenum-uranium alloys.

SECRET

Table 6.10—Hardness Data for Quenched and Reheated Molybdenum-Uranium Alloys

Analysis, wt. % Mo	As- cast	Rockwell A Hardness						
		2 hr at 900°C, water- quenched	Reheated 2 hr at indicated temperatures, °C					
			300	400	500	600	700	800
0.71*	65	60		65	65	53	66	63
1.09*	64	71		74	69	63	71	70
2.26*	69	70		78	70	66	71	70
2.01–3.83†	68	65						
2.6–2.8†	67	63	75	77	73	57	67	
3.12*	71	65		77	70	67	65	65
3.89–6.88†	69	60						

*See reference 18.

†See reference 9.

Table 6.11—Effect of Time of Tempering on Hardness of Molybdenum-Uranium Alloys

Analysis, wt. % Mo	Temperature water- quenched from, °C	Tempering temperature, °C	Rockwell A hardness							
			As- quenched	Tempering time, hr						
				1	2	4	8	16	32	
2.00–2.02*	700	200		75	75	75	76			
2.00–2.02*	700	300		77	77	77	78			
2.00–2.02*	700	400		76	76	76	74			
2.6–2.8†	900	400	68	77	78	78	77	76	75	

*See reference 18.

†See reference 9.

It will be observed that, at any of the temperature levels for either of the alloys listed in the table, the tempering time seems to be unimportant with respect to the hardness of the alloys. Also, the final hardness of the quenched and tempered 2.00 to 2.02 wt. % molybdenum alloy was found to be practically the same for any of the tempering temperatures used.

Annealing treatments after cold rolling were performed on samples of 5 wt. % molybdenum alloy to determine the effect of the annealing temperature upon the hardness and structure.¹⁹ The material used

SECRET

was quench-annealed at 700°C and was then cold-rolled to a 60 per cent reduction. The cold-rolled material was then annealed 0.5 hr at temperatures up to 750°C before hardness readings were made and the structures were examined. The samples were heated in argon and water-quenched.

Fig. 6.15—Effect of annealing $\frac{1}{2}$ hr at temperature on the hardness of molybdenum-uranium alloy (5 wt. % molybdenum).

Although this scale was not quite adequate, Rockwell G readings were used to facilitate comparison with previous work. The results of the hardness tests are shown in Fig. 6.15.

There was a marked increase in hardness up to 400°C. At 500°C a gradual softening began which continued to 600°C. At 650°C there was a great loss of hardness, but annealing at still higher temperatures, up to 750°C, produced no further softening.

Hot hardness of the 5 wt. % molybdenum alloy was determined by the mutual-indentation method.¹⁹ These results are given in Table 6.12, including the room-temperature value on the as-rolled 0.5-in. rod as determined by the standard Brinell test. The specimens used in the high-temperature tests were machined from the hot-rolled 0.5-in. bar and were tested without further heat-treatment.

The values vary considerably from those reported for pure metal.²⁰ The hardness is substantially higher in the alpha-temperature ranges;

SECRET

Table 6.12—Elevated-temperature Hardness Data

Testing temp., °C	Time at temp., min	Load, kg	Brinell hardness		Remarks
			Pure U*	U + 5 wt. % Mo†	
Room	30	3000	185‡	195	
400	30	2000		253	
500	30	2000		133	
520	45	2000		141	
600	30	500		69	
604	20	500	19		
650	30	500		23	Flattened under 2000 kg
654	20	500	25		
702	20	500	44		
750	30	500		14	
753	20	500	16		
772	20	500			Sample flattened
800	45	500		12.6	
900	30	500			Sample flattened

*See reference 20.

†See reference 19.

‡Converted from Rockwell B.

Table 6.13—Tensile Properties of Molybdenum-Uranium Alloys

Intended composition, wt. % Mo	2	5	5
Heat-treating temp.,* °C	700	700	700
Yield strength (0.2 offset), psi	148,000		
Ultimate tensile strength, psi	165,750	93,750	89,850
Elongation in 2 in., %	0.25	3.0	9.4
Reduction in area, %	0	4.4	11.1
Young's modulus, $E \times 10^6$, psi	24		

*All specimens were quenched.

no indication of the hard-beta phase is noted, but the hardness of gamma metal compares favorably. These tests are in agreement with the elevated-temperature tensile tests.

17.2 Tensile Properties. The tensile results on cast and heat-treated uranium-molybdenum alloys are given in Table 6.13.²¹

The stress-strain curve for the 2 wt. % molybdenum alloy is given in Fig. 6.16. No stress-strain curves were obtained for the 5 wt. % molybdenum alloys.

Fig. 6.16—Tensile test of molybdenum-uranium alloy (2 wt. % molybdenum) as-quenched from 700°C. This was a standard 0.505-in.-diameter specimen.

Rolled slugs, 1.360 in. in diameter by 8 in. long, of a 5 wt. % molybdenum alloy were rolled at 1400°F (760°C) into $\frac{7}{8}$ - and $\frac{1}{2}$ -in. rods.¹⁹ Specimens were heat-treated as follows: Two blanks were quenched (specimens 2 and 3 in Table 6.14) after heating at 700°C for 1 hr and one (specimen 1) after heating at 500°C for 1 hr. These were then machined to standard 0.505-in. test specimens.

Loading and unloading tests, first to 10,000 psi and then to 25,000 psi, were made on the 700°C-treated (gamma) specimens. Examples

SECRET

of these loading and unloading curves for both lateral and longitudinal strains are shown in Fig. 6.17. The 500°C (alpha) specimen was similarly loaded. Following the loading and unloading tests, the specimens were broken, giving the curves shown in Fig. 6.18. These curves show a substantial straight portion as compared to pure-metal curves.

The results of these tests are summarized in Table 6.14. It will be noted that the properties of the gamma specimens are not greatly

Table 6.14—Mechanical Properties of 5 Wt. % Molybdenum Alloy

Properties	Specimens		
	1	2	3
Heat-treating temp., °C	500	700	700
Ultimate strength, psi	183,000	80,000	86,500
Yield strength, psi			
1% offset	153,500	51,250	52,050
2% offset	166,500	58,750	59,050
Elongation in 2 in., %	6	1 (?)	1 (?)
Reduction in area, %	8	0	0
Brinell hardness in grip	363	160	163
Type of fracture	Fine, flat	Fine, rough edge	Fine, rough edge
Character of curves	Smooth	Smooth	Smooth

different from those reported for gamma-extruded material. The elongation values for this heat-treatment are indeterminate because of small fractures throughout the gauge length. There appears to be a considerable hardening effect in heating for 1 hr at 500°C. The strength and yield properties in this condition are much higher than any reported for pure metal.

High-temperature tensile tests were made on standard 0.25-in. test specimens in an argon atmosphere at 520 and 600°C on stock from the 0.5-in.-diameter hot-rolled bar.¹⁹ Both specimens were held at temperature about 2.5 hr before testing. The 600°C specimen showed almost 100 per cent reduction in area. The strength values at 600°C are slightly higher than those obtained on rolled pure metal at the same temperature, but the ductility is very much greater.

The elevated-temperature tensile results for the 5 wt. % molybdenum-uranium alloy are given in Table 6.15.

Tensile tests were also made on heat-treated hot-rolled 5 wt. % molybdenum alloys.¹⁴ One billet was cast from beryllia into a 2-in.-diameter silica-lined mold and was then hot-rolled at about 1450°F

SECRET

Fig. 6.17—Elastic deformation of specimen 700-1, a molybdenum-uranium alloy (5 wt. % molybdenum). The curves were taken from the data supplied by a Tuckerman extensometer and a lateral-strain gauge. A, longitudinal strain, first loading. B, longitudinal strain, second loading. C, lateral strain, second loading.

(790°C) to a 0.5-in. rod. After rolling, the rod was water-quenched, straightened, and ground to a 0.45-in. diameter. The other billet, $4\frac{1}{8}$ in. square by 20 in. long was cast from a graphite crucible into a graphite mold and was rolled at 1400°F (760°C) to a 1.525-in.-diameter rod. The rod was finished to 1.360 in. in diameter by machining and grinding.

SECRET

Fig. 6.18—Room-temperature stress-strain curves of a molybdenum-uranium alloy (5 wt. % molybdenum) taken from data from a Peters extensometer.

Table 6.15—Results of Elevated-temperature Tensile Tests on 5 Wt. % Molybdenum-Uranium Alloy

Test temp., °C	600	520
Ultimate tensile strength, psi	14,200	64,000
Yield strength, psi		
1% offset	8,400	24,200
2% offset	8,870	26,800
Elongation, %	273	48
Reduction in area, %	99.1	73.1

The two alloy rods were cut into 6-in. lengths, and specimens were heat-treated as follows: (1) 2 hr at 850°C and water-quenched; (2) 2 hr at 850°C and furnace-cooled; and (3) 2 hr at 850°C and water-quenched, followed by a 24-hr tempering treatment at 350°C and water-quenching.

In general, the results obtained from the tests on molybdenum alloys corresponded to the results of similar tests on uranium-columbium alloys. The addition of 5 wt. % molybdenum to uranium has been shown to be sufficient to retain the gamma phase in alloy

SECRET

samples that are quenched from 850°C. Also, hardening and extreme brittleness were manifest in the alloys upon tempering at 350°C for 24 hr after previously homogenizing at 850°C. The hardening effect is probably due to a partial transformation of the gamma phase.

The carbon content of the two billets was approximately the same, even though one was melted and cast out of contact with carbon; therefore little can be said concerning the effect of carbon on the behavior of uranium-molybdenum alloys.

The results of the tensile tests are shown in Table 6.16, and they correspond with the hardness data. Stress-strain curves of the two alloys are shown in Figs. 6.19 and 6.20.

The elastic constants of a 5 wt. % molybdenum alloy have been determined.¹⁹ The alloy was rolled at 1400°F (760°C), heated 1 hr at 700°C, and water-quenched. The modulus of elasticity and Poisson's ratio values for the gamma state compare reasonably well with those reported for pure metal. The results are tabulated in Table 6.17.

17.3 Compression Properties. Compression tests of uranium-molybdenum alloys were made on small cylinders, 0.371 to 0.382 in. in diameter and 0.151 to 0.238 in. long, for determination of plastic flow characteristics.²¹ The tests were run by compressing the disks at known loads and measuring the thickness and area of the samples after removing the load. The actual or true stress at each compression stage was plotted against the percentage reduction in height. The plastic or permanent deformation was measured, and the curves show the course of plastic deformation as a function of the true stress at each point. These curves are shown in Figs. 6.21 to 6.23.

The alloys were tested as-quenched from 700°C and as-aged at 300 or 400°C for various periods of time. The aging treatments cause an increase in strength, and the 400°C treatment is more effective than the 300°C treatment. This increase in strength is reflected in the increased resistance to deformation in the flow properties.

In the 2 wt. % molybdenum alloys quenched from 700°C, the stress required for plastic deformation rises rapidly with increased strain until, at 30 per cent reduction in height, a stress of about 300,000 psi is required. In the 2 wt. % molybdenum alloys aged at 300 and 400°C, the stress rises even more rapidly with the strain. Two samples aged 2 hr at 300°C required about a 280,000-psi stress for a 2 per cent reduction in height. Aging for 1 hr at 300°C did not confer as much resistance to deformation as the 2-hr aging treatment. Aging for 50 hr at 400°C was not very effective in causing high resistance to flow.

SECRET

SECRET

Table 6.16—Results of Tensile Tests on Rolled and Heat-treated Molybdenum-Uranium Alloys

Specimen*	Chemical analysis			Heat-treatment		Tensile strength, psi					Rockwell hardness before test		Size of standard specimen, in.
	Wt. % Mo		Carbon, ppm			Ultimate	P.L.,† psi	Yield point	Elongation, %	Reduction in area, %	B	A	
	Min.	Max.		in vacuo, Hr	°C								
1.4	4.61	4.62	840, 670	2	850‡	95,200	23,000	95,200	9.3	10.2	81	48	1/4
1.4	4.61	4.62	840, 670	2	850‡	97,800	30,000	97,800	9.9	11.2	81	48	1/4
2.4	4.61	4.62	840, 670	2	850§	167,000	158,000	167,000	8.3	30.3	108	66	1/4
2.4	4.61	4.62	840, 670	2	850§	169,500	157,000	169,500	8.0	29.0	108	66	1/4
3.4	4.61	4.62	840, 670	2	850‡	219,000			0	0	117	73	1/4
				24	350‡								
3.4	4.61	4.62	840, 670	2	850‡	202,000			0	0	117	73	1/4
				24	350‡								
1.5	4.39	4.40	850, 805	2	850‡	100,000	45,000	100,000	10.7	10.8	80	47	5/16
1.5	4.39	4.40	850, 805	2	850‡	96,600	30,000	96,600	14.8	13.6	80	47	5/16
2.5	4.39	4.40	850, 805	2	850§	176,500	165,000	176,500	9.3	33.8	108	68	5/16
2.5	4.39	4.40	850, 805	2	850§	180,000	168,000	180,000	9.2	32.4	108	68	5/16
3.5	4.39	4.40	850, 805	2	850‡	170,000			0	0	116	75	5/16
				24	350‡								
3.5	4.39	4.40	850, 805	2	850‡	191,500			0	0	116	75	5/16
				24	350‡								

*Specimens 1.4 to 3.4 were melted in beryllia and cast into silica. Elongation measurements were measured in percentage per 1 1/4 in. Specimens 1.5 to 3.5 were melted in graphite and cast into graphite. Elongation measurements for these specimens were measured in percentage per 1 1/2 in.

†P.L. = proportional limit.

‡Water-quenched.

§Furnace-cooled.

SECRET

Fig. 6.19—Stress-strain curves of molybdenum-uranium alloy melted in a BeO crucible (5 wt. % molybdenum). A, specimens 1.4-A and 1.4-B were heated 2 hr at 850°C and were then water-quenched. B, specimens 2.4-A and 2.4-B were heated 2 hr at 850°C and then were furnace-cooled. C, Specimens 3.4-A and 3.4-B were heated 2 hr at 850°C, water-quenched, tempered 24 hr at 350°C, and then water-quenched again.

SECRET

Fig. 6.20—Stress-strain curves of a molybdenum-uranium alloy melted in a graphite crucible (5 wt. % molybdenum). A, specimens 1.5-A and 1.5-B were heated 2 hr at 850°C and then water-quenched. B, specimens 2.5-A and 2.5-B were heated 2 hr at 850°C and furnace-cooled. C, specimens 3.5-A and 3.5-B were heated 2 hr at 850°C, water-quenched, tempered 24 hr at 350°C, and then water-quenched again.

SECRET

Table 6.17—Elastic Constants of Uranium Plus 5 Wt. % Molybdenum¹⁹

Load, psi	Increasing load				Decreasing load					
	Specimen 1		Specimen 2		Specimen 1		Specimen 2		Average	
	Mod. of elast.*	Poisson's ratio	Mod. of elast.*	Poisson's ratio	Mod. of elast.*	Poisson's ratio	Mod. of elast.*	Poisson's ratio	Mod. of elast.*	Poisson's ratio
5,000	16.3	0.40	17.4	0.38	16.5	0.40	18.0	0.36	17.0	0.39
10,000	16.3	0.40	17.5	0.39	16.5	0.40	16.9	0.37	16.8	0.39
15,000	16.8	0.38	15.9	0.41	18.1	0.37	17.7	0.36	16.8	0.38
20,000	16.9	0.38	15.9	0.41	18.1	0.37	17.7	0.36	16.8	0.38
All loads									16.9	0.38

*In modulus values, $\times 10^6$ has been omitted. Values are average of all runs in which Tuckerman gauges were used.

Fig. 6.21—Compression stress-strain curves for a molybdenum-uranium alloy (2 wt. % molybdenum) after two heat-treatments. Curve A, the result of quenching at 700°C. Curve B, the result of quenching from 700°C and then aging 2 hr at 400°C.

A summary of the compression flow results may be stated as follows:

1. The addition of about 2 wt. % molybdenum to uranium causes a tremendous increase in resistance to compressive flow.
2. The 700°C-quenched alloys flow much more readily than the aged alloys.
3. Specimens aged for 2 hr at 400°C show more resistance to flow than specimens aged for 1 hr at 300°C.

SECRET

Fig. 6.22—Compression flow curves for a molybdenum-uranium alloy (2 wt. % molybdenum). Curve A, as-quenched from 100°C. Curve B, quenched from 100°C and aged 1 hr at 300°C. Curve C, quenched from 100°C and aged 2 hr at 300°C.

4. Whereas unalloyed uranium can be reduced by at least 50 per cent in disks of this size, the molybdenum alloy can only be deformed about 20 to 30 per cent.

5. Overaging at 400°C does not appear to cause brittleness.

18. NICKEL-URANIUM ALLOYS

The as-cast hardness of a 5 wt. % nickel-uranium alloy was found⁹ to be 67 Rockwell A. The actual analysis of the alloy ingot was 5.7 wt. % nickel at the top and 2.1 wt. % at the bottom. Attempts at heat-treating this alloy at 800 to 900°C were unsuccessful because melting occurred.

Further hardness measurements were made on a 0.5 wt. % nickel-uranium alloy. This ingot analyzed 0.42 wt. % nickel at the top and 0.48 wt. % at the bottom. The hardness data for this alloy are given in Table 6.18.

SECRET

Fig. 6.23—Compression flow curves for a molybdenum-uranium alloy (2.5 wt. % molybdenum) quenched from 700°C and aged. Curve A, aged 1 hr at 300°C. Curve B, aged 2 hr at 300°C.

Table 6.18—Hardness Data for Nickel-Uranium Alloy

Heat-treatment*	Hardness		
	Rockwell A	Eberbach microhardness (D.P.N.)	Rockwell A from Eberbach
2 hr at 900°C, water-quenched ¹⁰	67	345	66
2 hr at 900°C, cooled in argon, reheated to 600°C for 24 hr, water-quenched ¹¹	58	256	59
2 hr at 900°C, water-quenched (new sample)	60		
Reheated to 300°C	63		
Reheated to 400°C	62		
Reheated to 500°C	67		
Reheated to 600°C	63		

*The samples that were heated at 900°C showed quenching cracks, even when cooled in argon.

SECRET

19. OSMIUM-URANIUM ALLOYS

The Eberbach microhardness of an as-cast nominal 1.6 wt. % osmium-uranium alloy was reported¹⁰ to be 470 (D.P.N.). A series of heat-treatment and hardness tests was then run on a new sample of a 1.6 wt. % alloy.¹⁶ The results of these tests are as follows:

Heat-treatment	Rockwell A hardness
As-cast	61
Quenched from 900°C	66
Quenched from 900°C, reheated to 800°C	65
Quenched from 900°C, reheated to 700°C	63
Quenched from 900°C, reheated to 600°C	63
Quenched from 900°C, reheated to 500°C	61
Quenched from 900°C, reheated to 400°C	67

20. PALLADIUM-URANIUM ALLOYS

The as-cast Eberbach hardness number (D.P.N.) of a nominal 0.9 wt. % palladium-uranium alloy was found¹⁰ to be 404. Results of further work on a new alloy of this same nominal composition, which actually averaged 1.02 wt. % palladium by analysis, are as follows:

Heat-treatment	Rockwell A hardness
As-cast	66
Quenched from 900°C	70
Quenched from 900°C, reheated to 800°C	70
Quenched from 900°C, reheated to 700°C	61
Quenched from 900°C, reheated to 600°C	57
Quenched from 900°C, reheated to 500°C	69
Quenched from 900°C, reheated to 400°C	70

21. PLATINUM-URANIUM ALLOYS

A nominal 5 wt. % platinum-uranium alloy was prepared for hardness and heat-treatment experiments.⁹ This alloy analyzed 5.20 wt. % platinum at the top and 5.32 wt. % at the bottom. The results obtained by the work on this alloy are as follows:

SECRET

Heat-treatment	Rockwell A hardness
As-cast	65
2 hr at 900°C, water-quenched	72
Reheated to 300°C	74
Reheated to 400°C	73
Reheated to 500°C	70
Reheated to 600°C	67
Reheated to 700°C	71

Further studies on the effect of heat-treatment on platinum-uranium alloys were carried on, using samples cut from a nominal 1.7 wt. % platinum-alloy casting. The results of these tests are given in Table 6.19.

Table 6.19—Hardness Data for Platinum-Uranium Alloys

Heat-treatment*	Hardness		
	Rockwell A	Eberbach microhardness (D.P.N.)	Rockwell A from Eberbach
Water-quenched ¹⁰ from 900°C	66	372	69
2 hr at 900°C, cooled in argon, reheated to 600°C for 24 hr, water-quenched ¹¹	54	358	67
2 hr at 900°C, water-quenched (new sample)	65		
Reheated 2 hr at 300°C	66		
Reheated 2 hr at 400°C	64		
Reheated 2 hr at 500°C	62		
Reheated 2 hr at 600°C	56		

*Both the water-quenched and argon-cooled samples which had been heated to 900°C showed cracking.

22. RHENIUM-URANIUM ALLOYS

A 160-g melt of a nominal 1.6 wt. % rhenium-uranium alloy was prepared for hardness studies.¹⁶ The results of hardness measurements on as-cast and heat-treated samples of this alloy were as follows:

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Heat-treatment	Rockwell A hardness
As-cast	73
Quenched from 900°C	65
Quenched from 900°C, reheated to 800°C	65
Quenched from 900°C, reheated to 700°C	66
Quenched from 900°C, reheated to 600°C	65
Quenched from 900°C, reheated to 500°C	65
Quenched from 900°C, reheated to 400°C	68

This alloy was cracked in the as-cast condition, and it cracked further on quenching from 900°C.

23. RHODIUM-URANIUM ALLOYS

The as-cast hardness of rhodium-uranium alloys of three different compositions have been reported as follows:²²

Wt. % rhodium	Rockwell B hardness
0.4	100
2.0	107
4.0	107

Work on the effect of heat-treatment of a nominal 0.9 wt. % rhodium-uranium alloy has yielded the following results:¹⁶

Heat-treatment	Rockwell A hardness
As-cast	63
Water-quenched from 900°C	73
Water-quenched from 900°C, reheated to 800°C	72
Water-quenched from 900°C, reheated to 700°C	70
Water-quenched from 900°C, reheated to 600°C	63
Water-quenched from 900°C, reheated to 500°C	69
Water-quenched from 900°C, reheated to 400°C	72

The effect of heat-treatment on the hardness of a 4.6 wt. % rhodium-uranium alloy has been reported as follows:²³

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Heat-treatment	Rockwell B hardness
As-cast	107
Water-quenched from 1000°C	115
Water-quenched from 845°C	116
Water-quenched from 710°C	104
Slow-cooled from 710°C	109

24. RUTHENIUM-URANIUM ALLOYS

A 160-g melt of a nominal 0.9 wt. % ruthenium-uranium alloy was prepared for hardness study.¹⁶ This alloy analyzed 1.40 wt. % ruthenium at the top and 1.24 wt. % at the bottom. The results of this study are as follows:

Heat-treatment	Rockwell A hardness
As-cast	66
2 hr at 900°C, water-quenched	72
Reheated to 800°C	70
Reheated to 700°C	69
Reheated to 600°C	64
Reheated to 500°C	69
Reheated to 400°C	71

25. SILICON-URANIUM ALLOYS

25.1 Hardness. The study of silicon-uranium alloys has been largely limited to those alloys in the region of 25 at. % silicon (3.78 wt. % silicon). These alloys have been considered important because their corrosion resistance has been shown to be 500 to 1000 times greater than that of pure uranium, and the addition of about 3.75 wt. % silicon does not cause excessive neutron absorption. The good corrosion resistance of the silicon alloy has been shown to be due to the formation of the epsilon phase. The epsilon phase is not present in the as-cast silicon alloys but is formed by subsequently annealing the metal. Since the formation of epsilon was accompanied by softening, hardness measurements can be used to follow the formation of the epsilon phase as a function of the time and temperature of annealing.²⁴ The results of such tests on 3.76 and 2.95 wt. % silicon-uranium alloys are shown graphically in Fig. 6.24.

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Fig. 6.24—Effects of annealing time and temperatures on the hardness of silicon-uranium alloys. (a) 3.76 wt. % silicon. (b) 2.95 wt. % silicon.

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25.2 Tensile Strength. Tensile tests have been run²⁵ on a set of silicon-uranium alloy samples of unspecified analysis at room temperature and at 150 and 600°C. The metal was in the as-cast condition and was machined to standard tensile test bars with 0.25-in.-diameter reduced sections. The tensile data for these silicon alloys, together with similar data obtained on specimens of pure uranium (Metal Hydrides scrap metal), are given in Table 6.20.

Table 6.20—Comparison of Tensile Data for Silicon-Uranium Alloys and Pure Uranium

Material	Temp., °C	Tensile strength, psi	Yield strength (0.2% offset), psi	Elongation, %	Reduction in area, %
Si alloy	Room	73,500	67,350	0	Nil
Si alloy	150	74,800	66,800	0	0.81
Si alloy	600	25,300	19,800	9.1	6.9
MH cast metal	Room	52,500	43,000	1.8	0.8
MH cast metal	150	27,130	Brittle	1.7	None
MH cast metal	600	11,720	8900	17.9	31.3

The silicon alloy showed some superiority in strength at all three testing temperatures, being less affected at 150°C than the MH scrap metal. The ductility of the silicon alloy at all temperatures was lower than that of the MH metal.

25.3 Compressive Strength. Results of room-temperature compression tests on epsilon silicon-uranium alloys have demonstrated that the metal is ductile to such an extent that 15 per cent plastic deformation is possible.²⁴ The results of a number of such tests made on 0.5-in.-diameter 0.5-in.-long specimens of cast epsilon alloys are plotted in Figs. 6.25 to 6.27.

Elevated-temperature compression tests have been made¹⁴ on 0.500-in.-diameter 1.000-in.-long specimens of epsilon alloy containing 3.4 wt. % silicon. These specimens were tested at six different temperatures, ranging from 610 to 850°C.

The shapes of the samples after testing indicated that a fair amount of ductility existed in the metal at the testing temperatures. The data obtained by these high-temperature compression tests are shown in Table 6.21 and are presented graphically in Fig. 6.28.

25.4 Fabrication. It has been shown that the epsilon silicon-uranium alloy, with a Rockwell C hardness of 25 to 30, can be machined in a lathe.²⁵

SECRET

Fig. 6.25—Compression stress-strain curve for a silicon-uranium alloy (2.44 wt. % silicon) annealed at 750°C for 150 hr.

Fig. 6.26—Compression stress-strain curves for a silicon-uranium epsilon alloy (3.53 wt. % silicon). Curve A, as-cast. Curve B, annealed at 750°C for 150 hr.

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Fig. 6.27—Compression stress-strain curves for a silicon-uranium epsilon alloy (3.76 wt. % silicon). Curve A, as-cast. Curve B, annealed at 750°C for 150 hr.

Table 6.21—Data Derived from High-temperature Compression Tests

Temp., °C	Yield point, psi	Load for 10% reduction, psi	Load for 20% reduction, psi	Rockwell B hardness before compression	Time in test furnace, hr
610	54,300	51,000*	49,500	102	2
700	17,750	15,500	14,500	102	1.5
750	9,550	9,550†	8,800	104	1
780	10,200	10,200†	8,900	103	3
840	4,650	4,650†	4,500	102	1.5
850	4,300	3,500	1,500	100	1.25

*Load before yield point was reached.

†Same point as yield-point load.

An attempt has been made to extrude epsilon alloy. Unfortunately the specimens were heated above the temperature at which epsilon is stable—the exact temperature is unknown. In spite of this, one alloy containing 3.6 wt. % silicon did come through the die, but it was not

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Fig. 6.28—Relation of temperature to compression strength of a silicon-uranium epsilon alloy (3.4 wt. % silicon). (a) Yield-point curve. (b) 20 per cent reduction curve.

in very good shape. In this trial a 2-in.-diameter billet was extruded to a 0.5-in.-diameter rod. This result indicated that the successful extrusion of the epsilon alloy may be possible after further experimentation to find the best conditions.²⁵

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26. SILVER-URANIUM ALLOYS

The hardness of a nominal 5 wt. % silver-uranium alloy has been studied after various heat-treatments.⁹ The alloy actually contained only a trace of silver at the top and 0.09 wt. % silver at the bottom. The effect of heat-treatment on this alloy is summarized as follows:

Heat-treatment	Rockwell A hardness
As-cast	54
2 hr at 900°C, water-quenched	54
Reheated to 300°C	58
Reheated to 400°C	57
Reheated to 500°C	56
Reheated to 600°C	56
Reheated to 700°C	57

27. TANTALUM-URANIUM ALLOYS

27.1 Hardness. The earliest information reported on the hardness of tantalum-uranium alloys was the data obtained on a nominal 5 wt. % tantalum alloy.¹ This alloy analyzed 7.7 wt. % tantalum at the top and 1.5 wt. % at the bottom. The results of this work are as follows:

Heat-treatment	Rockwell A hardness
As-cast	63
2 hr at 900°C, water-quenched	70
Reheated to 300°C, water-quenched	70
Reheated to 400°C, water-quenched	70
Reheated to 500°C, water-quenched	70
Reheated to 600°C, water-quenched	67
Reheated to 700°C, water-quenched	68

Later metallographic work on tantalum-uranium alloys established the fact that as-cast 91.57 and 98.68 wt. % tantalum alloys, made by heating compressed mixtures of the powdered metals with an atomic hydrogen torch, consisted of two phases. The Rockwell A hardness of these alloys averaged²⁸ 70.

Other tests on tantalum-uranium alloys led to the expression of the opinion that tantalum additions up to 11.8 wt. % probably have little effect on the hardness of uranium.²⁷

SECRET

27.2 Fabrication. Alloys containing 7.8 and 11.8 wt. % tantalum in uranium were found to be too brittle to be successfully machined in the as-cast condition.²⁸

28. THORIUM-URANIUM ALLOYS

An attempt to prepare a 5.1 wt. % thorium-uranium alloy was made using powdered thorium. It appeared as though most of the thorium powder oxidized during the preparation of the alloy, and therefore the thorium content was probably lower than the nominal specification. This alloy had a hardness⁹ of 53 Rockwell A in the as-cast condition and 59 when water-quenched from 900°C.

Hardness measurements on a series of as-cast thorium-uranium alloys varying from 1 to 95 wt. % thorium demonstrated that these alloys become softer with increasing thorium contents²² above 20 wt. %. The data obtained by these tests are as follows:

Wt. % thorium	Rockwell G hardness
1.0	91
3.4	93
6.8	95
20	95
30	90
40	86
50	90
60	77
70	74
90	75
95	70
100	27

The 50, 90, and 95 wt. % thorium alloys in the above series were made using Ames extruded thorium. The other alloys contained Westinghouse sintered thorium.

29. TITANIUM-URANIUM ALLOYS

The first hardness data reported for titanium-uranium alloys were the results of work done on a nominal 5 wt. % alloy.⁹ This alloy was made by adding the titanium in the form of titanium hydride, and, because of the poor yield obtained from the hydride, the alloy analyzed only 1.4 wt. % titanium at the top and bottom. The following hardness information was reported for this alloy:

SECRET

Heat-treatment	Rockwell A hardness
As-cast	60
2 hr at 900°C, water-quenched	63
Reheated to 300°C	64

Further work was done on a titanium-uranium alloy which analyzed 0.40 wt. % titanium at the top and 0.23 wt. % at the bottom. The data obtained on this alloy are given in Table 6.22.

Table 6.22—Hardness Data for Titanium-Uranium Alloy

Heat-treatment	Rockwell A	Hardness	
		Eberbach microhardness (D.P.N.)	Rockwell A from Eberbach
Water-quenched ¹⁰ from 900°C	56	240	58
2 hr at 900°C, slow-cooled to 600°C, held 24 hr, water-quenched ¹¹	54	188	49
2 hr at 900°C, water-quenched (new sample)	59		
Reheated to 300°C	58		
Reheated to 400°C	57		
Reheated to 500°C	57		
Reheated to 600°C	56		

30. TUNGSTEN-URANIUM ALLOYS

A series of heat-treatment and hardness measurements have been made on a nominal 5 wt. % tungsten-uranium alloy.⁹ This alloy actually contained 1.2 wt. % tungsten at the top and 1.5 wt. % at the bottom. The data obtained on this alloy are as follows:

Heat-treatment	Rockwell A hardness
As-cast	61
2 hr at 900°C, water-quenched	59
Reheated to 300°C	60
Reheated to 400°C	60
Reheated to 500°C	62
Reheated to 600°C	64
Reheated to 700°C	59

SECRET

31. VANADIUM-URANIUM ALLOYS

A preliminary study of the hardness of vanadium-uranium alloys led to the preparation of a nominal 0.45 wt. % vanadium alloy.¹⁰ After being heated at 900°C for 2 hr and water-quenched, this alloy had an average Eberbach microhardness number (D.P.N.) of 410.

Further studies on a new uranium alloy were made.¹⁶ This alloy analyzed 0.47 wt. % vanadium at the top of the ingot and 0.38 wt. % at the bottom. The results obtained from this work are as follows:

Heat-treatment	Rockwell A hardness
As-cast	59
2 hr at 900°C, water-quenched	71
Reheated for 2 hr at 800°C	71
Reheated for 2 hr at 700°C	60
Reheated for 2 hr at 600°C	60
Reheated for 2 hr at 500°C	69
Reheated for 2 hr at 400°C	69

This alloy was observed to contain cracks after being quenched from 900°C.

Another series of three vanadium-uranium alloys was prepared for metallographic and hardness examination.³ These alloys were made

Table 6.23—Analyses and As-cast Hardnesses of Vanadium-Uranium Alloys

Vanadium, wt. %	Analyses		Rockwell C hardness
	Beryllium, ppm	Carbon, ppm	
0.2–0.23	10–25	100–300	30
0.93–0.99	5–7	100–300	22
2.14–2.2	5–7	100–300	31

in beryllia crucibles, and hence it was considered important to consider the beryllium contamination of the metal. The analyses and as-cast hardnesses of the alloy are given in Table 6.23.

SECRET

32. ZIRCONIUM-URANIUM ALLOYS

The reported work on the hardness of zirconium-uranium alloys was initiated with measurements on a nominal 5 wt. % zirconium alloy.⁹ This alloy analyzed 1.16 wt. % zirconium at the top and 1.05 wt. % at the bottom. The results of this work are as follows:

Heat-treatment	Rockwell A hardness
As-cast	60
2 hr at 900°C, water-quenched	63
Reheated for 2 hr at 300°C	64

A new study of the hardness and heat-treatment of zirconium-uranium alloys involved the examination of a small casting of a nominal 0.8 wt. % alloy. This alloy is highly segregated, analyzing 2.7 wt. % zirconium at the top and less than 0.1 wt. % zirconium at the bottom. The data given in Table 6.24 were reported for this alloy.

Table 6.24—Hardness Data for Zirconium-Uranium Alloys

Heat-treatment	Hardness		
	Rockwell A	Eberbach microhardness (D.P.N.)	Rockwell A from Eberbach
2 hr at 900°C, water-quenched ¹⁰	55	219	55
2 hr at 900°C, cooled in argon, reheated to 600°C for 24 hr, water-quenched ¹¹	49	22	56
2 hr at 900°C, water-quenched (new sample)	57		
Reheated for 2 hr at 300°C	57		
Reheated for 2 hr at 400°C	58		
Reheated for 2 hr at 500°C	57		
Reheated for 2 hr at 600°C	55		

It has been determined that zirconium additions to uranium, up to 8.1 wt. %, increase the hardness of the uranium. Furthermore, the hardness of the 8.1 wt. % alloy was found²⁷ to increase to 51 Rockwell C after being quenched from 100°C.

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~~**SECRET**~~**UNCLASSIFIED**

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INDEX

A

Allotropy, 73-76

Alloys, 148

etching of, 116

grinding and polishing of, 114-115

rolling of, 59

uranium and aluminum, 118-119

fabrication of, 115-157

hardness of, 153-155

tensile strength of, 155-156

uranium and antimony, 118-119

hardness of, 157

uranium and arsenic, 119

uranium and beryllium, 119-121

hardness of, 158

uranium and bismuth, 121-122

uranium and cadmium, 122

uranium and calcium, 122

uranium and carbon, 122-124

hardness of, 158-160

preparation of, 158-159

uranium and cerium, 124

hardness of, 160-161

uranium and chromium, 124-125

hardness of, 161

uranium and cobalt, 125-126

hardness of, 161

uranium and columbium, 125-127

compression properties of,

165-170

hardness of, 161-164

tensile properties of, 162,

165-166

uranium-columbium-zirconium,

hardness data of, 170

Alloys, uranium and copper, 127-129

hardness of, 170-171

uranium and gallium, 129

hardness of, 171

uranium and germanium, 129

uranium and gold, 129

hardness of, 172

uranium and hydrogen, 129-130

uranium and indium, 130

uranium and iridium, 130

hardness of, 172

uranium and iron, 130-132

hardness of, 173

uranium and lead, 132

hardness of, 173

uranium and magnesium, 132

uranium and manganese, 132-133

hardness of, 173

uranium and mercury, 133-134

uranium and molybdenum, 135-136

compression properties of,

181, 186-188

hardness of, 173-176

tensile properties of, 176-185

uranium and nickel, 136-137

hardness of, 187-188

uranium and nitrogen, 137

uranium and osmium, 138

hardness of, 189

uranium and oxygen, 138-140

uranium and palladium, 140

hardness of, 189

uranium and platinum, 140

hardness of, 189-190

uranium and rhenium, 140

hardness of, 190-191

~~SECRET~~

UNCLASSIFIED

Alloys, uranium and rhodium, 140
 hardness of, 191-192
uranium and ruthenium, 140
 hardness of, 192
uranium and silicon, 141-142
 compression properties of,
 194-197
 fabrication of, 194, 196-197
 hardness of, 192-193
 tensile properties of, 194
uranium and silver, 142
 hardness of, 198
uranium and strontium, 142
uranium and tantalum, 142-143
 fabrication of, 199
 hardness of, 198
uranium and thorium, 144
 hardness of, 199
uranium and tin, 145-146
uranium and titanium, 146
 hardness of, 199-200
uranium and tungsten, 146-147
 hardness of, 200
uranium and vanadium, 146
 hardness of, 201
uranium and zinc, 146, 148
uranium and zirconium, 148
 hardness of, 202
Atomic dimensions, 73-76

B

Boiling point, 85

C

Casting, 37-40
Cold drawing, 59-61
 commercial, 61-62
Cold rolling, 56
Crystal structure, 73-76

D

Defects, 65
 in alpha-extruded rod, 66

Defects, in alpha-rolled rod, 66
 in gamma-extruded and cold-
 swaged rod, 66
 in gamma-extruded rod, 65-66
 inspection methods for, 67-68
 types of, 66
Density, 73-76
Drawing, 59-62

E

Elastic deformation, 94, 97-104
 (See also Alloys)
Electrical conductivity, 85-88
Electrolysis of fused salts for
 uranium production, 36-37
Emissivity, 90
Entropy, 83-84
 of fusion, 85
Etching, 116
Extrusion defect, 42, 49
Extrusion process, 42-45
 commercial, 45-49
 dies for, 44

G

Graphite centrifuge for bomb re-
 duction, 27-30
Graphite molds for casting, 37-38
Grinding, 64-65, 114-115
 safety precautions in, 65

H

Hardness, 90-94
 of alloys (see Alloys)
 in high-temperature tests, 96
 macrohardness values, 91-93
 microhardness determinations,
 95
 standard state of, 90
Heat content, 83-84
Heat of fusion, 85
Heat treatment (see Hardness)

UNCLASSIFIED

Heats of transition, 83-84
 Hot drawing, 61
 Hot rolling, in alpha range, 56-57
 commercial, 57-58
 in beta range, 58-59
 in gamma range, 59
 of uranium alloys, 59
 Hot-wire method for uranium
 production, 35-36

I

Isotopes of uranium, abundance
 of, 73
 radioactive properties of, 73

L

Lost-wax technique, 39

M

Machinability, 62-64
 Machining, 62
 safety precautions in, 65
 Magnesia liners, 32-33
 Melting point, 84
 Metallographic examination, 114

O

Open-crucible method for uranium
 production, 34-35
 Outgassing treatment, 49-52
 commercial, 52-56

P

Plastic deformation, 94, 96, 99
 105-106
 (See also Alloys)
 Poisson's ratio, 94, 104, 185
 Polishing, 114-115
 Precision casting, 39-40

Production, small scale, 25
 by electrolysis of fused salts,
 36-37
 by hot-wire method, 35-36
 by uranium halide reduction,
 25-26
 in graphite centrifuge, 27-30
 in open crucibles, inert
 atmospheres, 34-35
 in stationary bombs, 31-34
 by uranium oxide reduction, 34-35

Q

Quality of metal, 65

R

Reductions, 25-35
 in graphite centrifuge, 27-30
 in open crucible, 34-35
 in pyrex apparatus, 25-26
 in refractory crucible, 26
 in stationary bombs, 31-34
 Refractories for remelting and
 casting, 37-38
 Refractory crucible, 26
 Refractory molds for precision
 casting, 39-40
 Remelting and casting, 37-40
 Rolling, 56-59

S

Specific heat, 83-84
 Stationary bomb reduction, 1- to
 250-g scale, 31-34

T

Tensile properties, 99-102, 107-109
 (See also Alloys)
 Thermal conductivity, 88-89
 Thermal expansion, 76-83
 Thermoelectric potential, 88

~~**SECRET**~~**UNCLASSIFIED**

UNCLASSIFIED

SECRET

U

Y

Ultimate tensile strength, 99-102, 107-109

Young's modulus, 94, 103

V

Z

Vapor-pressure measurements, 85-87

Zirconia liner, 38

UNCLASSIFIED

SECRET