URANIUM ALLOY METALLURGY
AN ANNOTATED BIBLIOGRAPHY

ARMY MATERIALS AND MECHANICS RESEARCH CENTER

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URANIUM ALLOY METALLURGY -
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January 1974

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ARMY MATERIALS AND MECHANICS RESEARCH CENTER
Watertown, Massachusetts 02172
An annotated bibliography is presented of the work reported in the literature in the English language (1957-1973) on fabrication, mechanical and physical properties, physical metallurgy, and corrosion behavior of depleted uranium-base alloys. Also included are testing and preparation techniques and other studies for unalloyed uranium which are applicable to uranium alloys. (Authors)
PURPOSE - Nonnuclear applications of high-density, high-strength uranium-base alloys, exclusive of nuclear applications, have evolved in recent years. Bibliographies covering specific binary uranium-base alloy systems have been published. The purpose of this bibliography is to summarize the literature available on all uranium-base alloys covering the unique metallurgical characteristics, including corrosion behavior, complex phase transformations, and fabrication which must be considered by metallurgists and engineers.

SCOPE - The scope of this bibliography includes the fabrication, mechanical and physical properties, physical metallurgy, and corrosion behavior of depleted uranium-base alloys. Testing and preparation techniques and other studies for unalloyed uranium which are applicable to uranium alloys are also included.

Standard bibliographic reference tools including:

- CHEMICAL ABSTRACTS
- CORROSION ABSTRACTS
- DEFENSE DOCUMENTATION CENTER, TECHNICAL ABSTRACTS BULLETIN (TAB)
- METALS ABSTRACTS
- NASA SCIENTIFIC AND TECHNICAL AEROSPACE REPORTS (STAR)
- U.S. ATOMIC ENERGY COMMISSION, NUCLEAR SCIENCE ABSTRACTS
- U.S. GOVERNMENT REPORTS ANNOUNCEMENTS (GRA)

were the basic sources of information. Further references were found in texts, especially those publications covering proceedings of conferences in the field.

ARRANGEMENT - The bibliography is arranged in subject areas. References that could have been included in more than one subject area are entered only in the main subject area. Entries are in alphabetical order by author followed by U.S. and U.K. patents.

LIMITATIONS - Because of the vast body of literature on uranium alloy metallurgy in many languages only material published between 1957 and 1973 in the English language has been included.

In instances where the results of a specific research study have been published in the open literature and in report form, only the open literature reference is given.

This bibliography is an attempt to present a comprehensive but not exhaustive study of the literature in the field.

ACKNOWLEDGMENTS - The able assistance and patience of Mrs. A. V. Gallagher and the typing of the manuscript provided by Mrs. M. Gould, Miss J. Romano, Miss J. Caliguri, Miss J. Mc Farlane, Mrs. E. Dugan, and Miss C. Josey are gratefully acknowledged. The assistance of Mr. J. Greenspan in the original planning and scope of this bibliography and the persevering efforts of Miss R. Markus to provide many of the references and her diligent proofreading of the manuscript are also greatly appreciated.
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References to published articles and reports obtained from Nuclear Science Abstracts Vol. 12 through Vol. 16, Review of Metal Literature 1956 to 1960, and AWRE Library Indexes, including 130 references on U-Wo alloys are listed.


Specific use of U in shielding materials in relation to cost and/or weight savings realized in various designs are discussed. Information concerning commercial grades and forms of U and fabrication methods are included.


The dimensional stability and corrosion resistance of U and U-alloys including U-Zr, U-Nb, and U-Zr-Nb were evaluated for use as fuel element materials. Emphasized were: (1) "a-phase" alloys in which relatively low concentrations of alloying addition modify the kinetics of 8-or y-phase decomposition to yield a structure combining random orientation and fine grain size, (2) "y-phase" alloys in which sufficient concentrations of alloying additions stabilize partially or completely the cubic y-phase, circumventing the instability of the orthorhombic a-structure. Properties are discussed in relation to constitution, heat treatability, transformation kinetics, and micrographic features.


Physical and chemical properties of U-metal which may have a bearing on the industrial production of U-alloys are summarized. The legal aspects of natural U use are reviewed and some experimental data on airborne U dust obtained during pilot plant tests are presented.


Collection of 18 papers presented at the conference on the research and development of high-density structural U-alloys. Pertinent papers are annotated in this bibliography with each entry under the name of the specific author.
History, occurrence, preparation, crystallography, physical, chemical, and mechanical properties, and deformation of U are discussed at length. Transformation kinetics of U and U-alloys and diffusion in binary U-alloys are presented.


An annotated bibliography for binary U-Ti alloys complete as of 1 January 1973. The references obtained primarily from Nuclear Science Abstracts have been categorized according to subject within subject groupings.


Study of the explosive behavior of two phase U-Nb alloys after immersion in HNO₃ showed it to be related to finely divided microstructure and to a high C content.


The more common U-alloys and the reagents used to dissolve them are summarized in tabular form. The behavior of U and U-alloys in the common acids, ethyl acetate solutions of Br₂, HCl, NaOH, Na₂O is described.


Density, tensile strength, and impact strength of both U and U-Mo alloys are presented. Because of high density, good castability, and mechanical properties of U-alloys, they are considered for applications as aircraft counterweights and radiation shields for high energy sources and nuclear reactors.


Mechanical, physical, chemical, and metallurgical properties of U and U binary alloys are given and the effects of irradiation, thermal cycling, fabrication techniques, and alloy additions are discussed.

Wide range of potential industrial, nonnuclear applications of U, U compounds, and U-alloys are described.


High density W, Ta, and U-alloys were fabricated into cubes. Mechanical tests - tensile, compression, hardness, and impact - as well as chemical and metallographic examinations were conducted.


Short resume of the results of investigations of the physical-mechanical properties of U and U-alloys is given. The effect of irradiation and, in particular, the phenomenon of gas swelling as well as the effect of elevated temperature on dimensional stability of various α-phase and γ-phase U-alloys are examined. Techniques for metallographic examination of U are given.


Selected reviews of the production methods and properties of U, U-alloys, and U-compounds.


Brief report of work sponsored by DREV on various aspects of the physical metallurgy of U and U-alloys. The work consisted of general studies on the properties of alloys, effects of hot working, vacuum annealing, grain size refinement, and thermomechanical treatments. Some general remarks on cost, availability, health and safety problems, pyrophoricity, and oxidation of the alloys are included. The possibility of making composite alloys and the problems of static and dynamic properties are also included.


Report of the Ad Hoc Panel on Depleted Uranium of the Committee on the Technical Aspects of Critical and Strategic Materials. Supply situation, nonenergy consumption, potential uses, metallurgy, chemistry, and effectiveness as a catalyst and alloying element of depleted U are discussed. High density and ability to be alloyed make U a promising material for the development of new nonnuclear applications. Appendices on metallurgy of U and its alloys are included.

Proceedings of the Specification Coordination Meeting on Uranium Materials for Davy Crockett and Other Ordnance Programs held at Watertown Arsenal, 7 June 1960.


Short history of the development of U as an engineering material and current practices of Watertown Arsenal regarding materials processing, accountability, and health and safety are presented. Several U-alloys having high strength and high density are reviewed.

Phase Diagrams


U-alloy constitutional data are presented in the form of diagrams and crystal structure data for U phases and compounds are tabulated.


Reports of investigations of the constitution of U-Mo alloys are summarized and differences between the proposed equilibrium diagrams are discussed. It appears that the differences are due partly to the difficulty of establishing equilibrium in the solid state transformations. A compiled equilibrium diagram is presented.


The equilibrium diagram for U up to 19 wt% Mo alloy at < 900°C was determined. The eutectoid transformation, $\delta \rightarrow \gamma$, occurred at 639±5.0°C at 10.5 wt% Mo - earlier investigations used a maximum of 6 wt% Mo content. Tetragonality of the $\delta$-phase was confirmed and it was probably formed congruently.


Studies of the U-Zr-Si alloys by metallographic, x-ray diffraction, and thermal analysis techniques were used to construct tentative isothermal ternary sections at temperatures from 550°-950°C. Solubility of Si in U-Zr alloy or of Zr in U3Si2 was <2a/o but solubility of Zr in U3Si2 was fairly extensive and a phase (U,Zr)3Si2 was formed.

After being continuously cooled at different rates from the α-phase some U up to 5a/oNb alloys were aged at 550°C and resulting structural change is observed. Step annealing in the phase fields (α+γ), (α+γ), and (α+γ) is also described.


Bibliography containing 470 references on U-alloys prepared for metallurgists and fuel element designers at Sylvania-Corning Nuclear Corporation. Supplements include a collection of phase diagrams of binary U-alloys and tables showing behavior of U-alloys with respect to corrosion and irradiation.


The U-Ti-Zr alloy system containing up to 40a/oTi and 70a/oZr is reported in nine isothermal sections between 575°C-1000°C. Lattice parameters of U₁₂Ti which vary with composition confirm evidence for the high solubility of Zr in the U₃Ti phase.


U-Ti and U-Zr and U-Ti-Zr alloy systems are described. Alloys were prepared by arc-melting in purified Ar from high-purity materials with <0.5% weight loss. Phase diagrams were determined using dilatometric, metallographic, and x-ray analyses. Isothermal sections of the U-Ti-Zr system are given for temperatures of 575°C-850°C.


Brief summary of the results of investigations into alloy structures and phase diagrams of certain ternary systems, including U, Th and their refractory compounds is presented. U-Zr, Nb, Mo, and Zr alloys, first in binary systems then in ternary systems were studied. Emphasis was placed on that section of the phase diagrams which relates to solid state. X-ray and microstructural data were used to plot the phase diagrams.


The phase diagram of the U-Re alloy is presented. It is of the double eutectic type having the composition U₉Re₂ which exhibits allotropy at 180°, melts congruently at 2200°, and reacts sluggishly with U solid solutions at <750° to form the peritectoid compound U₂Re. Eutectoid reactions occur at 643°, 681°, 1105°, and 2105° at compositions of 1.4, 6.0, 10.5, and 65.5w/oRe respectively. The maximum stability of Re in α-U is ~ 0.4w/o at 643°, in β-U ~ 1.9w/o at 681°, and in γ-U ~ 7.0w/o at 975°C. Investigation of transformation kinetics of U solid solutions indicated the existence of transition states; one involving γ-phase decomposition and the other the β→α transformation. Sequence of phase changes is discussed.
Debye-Scherrer photographs of ternary alloys of \( \gamma \)-U-\( \gamma \)-Mo (up to 30\% Mo) were obtained and lattice parameters studied. Parameter measurements and compositions of the alloys are presented in tabular form.


The U-Th phase diagram is reported: a simple eutectic system was formed with a region of liquid immiscibility extending from 0-49\% Th. Solubility of Th in U is 0.3\% at 900° and 0.05\% at 700°C.


Thermal analysis, x-ray diffraction, and metallographic techniques were used to construct U-Ra and U-Rh phase diagrams. Heats of solution for the intermetallic compounds were calculated from activity of U in solution in the liquid phase. Correlation between solid solubility of various elements in \( \gamma \)-U is demonstrated using a model based on atomic radius ratio at the temperature of reaction.


The addition of 4.5\% Hf to U increased the temperature of \( \gamma \)-\( \gamma \) transformation on heating from 668° to 676° and lowered the \( \beta \)-\( \gamma \) transformation to a eutectoid at 733°. At elevated temperatures, the bcc U and Hf were soluble in all proportions. U-20-65\% Hf alloys quenched from 1100° and above contained a hexagonal \( \gamma \)-phase, \( a=4.97\AA \) and \( c=3.04\AA \) with x-ray powder pattern almost identical to that of \( \gamma \)-phase of U-Zr system. It is believed to be a metastable phase which decomposes to U and Hf at equilibrium. U lowered the \( \alpha \)-\( \beta \) transformation in Hf to a monotectoid at 1150° and 55\% Hf. At 1425°C the solubility of Hf in U is 2.0-2.3\%.


Metallography, x-ray diffraction, and thermal analysis were used to determine equilibrium diagrams and apparent phase boundaries in U-Nb alloys.


The U-Cb phase diagram was determined using x-ray, metallographic, dilatometric, resistivity and melting point measurements.


Compilation written in cooperation with representatives of the U.K. of U and Th constitution diagrams (Supersedes BMI-1000). Both major sections - U alloys and Th alloy - are preceded by a discussion of transformation and melting temperatures of the base metal followed by discussion of the various binary and ternary alloy systems. Crystallographic data, phase diagrams whenever possible, and references to the literature are included for each alloy.

A compilation of U. S. and U. K. constitutional diagrams and data on crystallography of intermetallic compounds including both binary and ternary systems is presented. Melting points and allotropic modification of the base metals are included.


A constitutional diagram of a ternary section, U-74\%/Zr to U-35\%/Ti, based upon the results of thermal, metallographic, and x-ray analyses of a series of U-alloys in ranging in composition between the 6-phases of the U-Zr and U-Ti systems is presented. At all temperatures investigated no evidence for a three phase region or a new phase, not found in the binary system, was indicated.


U-Mo-Nb alloys were examined after H\(_2\)O quenching from 675\(^\circ\), 700\(^\circ\), and 720\(^\circ\) to determine the composition and quantity of \(\gamma\) - and \(\beta\) -phases. The shape of the \((\alpha+\beta)/\gamma\) phase boundaries agree with the finding that an increased \(\gamma/\beta\) ratio can be obtained by step-annealing at 675\(^\circ\)-700\(^\circ\)C the U-3.6\%/Mo-6.9\%/Nb alloys.


U-rich end of the Zr alloy system was investigated. Solubilities of Zr in both \(\alpha\)- and \(\beta\)-U were found to be 6.2\%/ at 662\(^\circ\) and 0.4\%/ at 693\(^\circ\). The monotectoid decomposition of \(\gamma\) at 693\(^\circ\) and the eutectoid decomposition of \(\beta\) at 662\(^\circ\) were confirmed. In alloys containing <150 ppm O the \(\gamma\) and \(\gamma\) phase region boundaries were located at 4.5 and 22.0\%/O/Zr at the monotectoid temperature. Results indicate that O content of 160-355 ppm has a marked effect on phase relations in the \(\gamma\) and \(\gamma\) phase regions.

Testing Techniques


Principles involved in the eddy current method of measuring electrical conductivity of a solid are discussed and the meter used is described. The relationships between electrical conductivity and alloy structure, composition, and metallurgical conditions involved in the conductivity analysis of U-2\%/Mo alloy are described and results agreed with chemical analysis within ±0.128\%/Mo.

The techniques and limitations of residual stress analysis of U using an x-ray diffractometer are discussed. A review of the theory and the type of equipment and x-ray geometry are described. Preparation of the standard U plate and the procedure used to standardize x-ray results are presented in detail. Data showing a linear plot of increasing deflections of a U plate vs change in the "d" spacing are given.


Data obtained by x-ray diffraction techniques derived from the Berg-Barrett method were used to describe the structure of U-alloys. Exact numerical data concerning the grain and sub-grain size and the distribution of the sizes were obtained. Any disorientation >30 sec of arc can be detected. Observations of internal disorientations were carried out on one- or two-phase U-alloys and compared successfully to high-purity U. Although limited in certain respects the use of x-ray topographies allows a quantitative characterization of the structure.


The transition from orthorhombic to monoclinic structure in quenched U-Nb alloys was investigated by following the changes of ratio between the breadths of a number of selected diffraction lines. The α'-phase represents only the period between the outset of monoclinization and the moment when line-splitting becomes visible on x-ray photographs and it is not justified to regard it as a separate phase.


Stress relaxation properties of U-7.5Nb-2.5Zr alloy were determined by two separate series of tests at both room temperature and at elevated temperatures (2500°-3000°F). Only the testing procedure and initial treatment of data are discussed.


Techniques and equipment used at NLCO for γ radiation of U and procedures for handling specimens of various thicknesses are described. The γ-ray source was 15 Ci of Co-60. In addition to γ-radiography, autoradiography, radiography in color, and neutron radiography are discussed.
A standard method for making U-alloy specimens to study the effect of varying quenching rates by Debye-Scherrer x-ray analysis was developed. The method uses a spark-discharge machine to taper small, square metal rods which are subsequently electropolished to remove the disturbed surface layer of metal remaining after the machining process. Advantages of the method are discussed.

A method for the determination of diffraction peak positions (to a precision of ±0.01°) and their associated precision in U and Zr was developed using x-ray diffraction. It was found that the second-order polynomial provided the best least squares fit within the known precision of the observed data. The stress constant for U was determined to be 1308±110psi/0.01° shift in 620.

Ultrasonic testing for grain size, grain orientation, and defects during the various stages of manufacturing in an automated production line are described. Data from the resonant-frequency method and x-ray diffraction are compared and hopefully will lead to a NDT grain-orientation test for fuel specifications.

Metallographic Techniques

An attack-polish reagent consisting of a slurry of H₂O₂ (30w/o) and 5% NH₃ for the metallographic preparation of U-alloys was developed. Polishing was carried out on a polythene lap covered with terylene velvet. A new electrolytic etching technique using bright field illumination for the study of the grain structure of U and U-alloys is also described. Results obtained using these techniques with specific metallographic structures as examples are described.

Microstructural changes of U that occurred during heating and cooling through the allotropic transformation were recorded by time-lapse photography. Electropolished U specimens were heated in a hot stage, mounted on a metallograph and evacuated to at least 3x10⁻⁶mm. The original grain structure became visible during heating due to relief caused by the differences in thermal expansion between adjacent grains. The α-β transformations occurred at a faster rate than the β-γ. Grain boundaries of the β and γ-phases were visible after the respective transformations.

Foils of U suitable for direct observation in the electron microscope were prepared by electropolishing in a solution containing phosphoric acid, ethylene glycol, and ethyl alcohol followed by a solution containing sulphuric acid, glycerine, and H$_2$O. Twins, dislocations, and dislocation loops were observed in typical specimens.


Development of techniques for thinning U specimens by electropolishing and removal of the oxide film formed during the electropolishing to enable direct observation of the configurations and movements of dislocations and other crystallographic defects are described. Specimens were heat treated by two methods: (1) annealed for 1 hr at 600°C to recrystallize the structure, (2) α-transformed for 1 hr at 720°C. Observations concerning the nature of dislocations are discussed.


Optical and electron metallography and electron diffraction were used to characterize the response of U-2 wt. % Zr to heat treatment and the effects of irradiation on the alloy. A new technique for isolating the second-phase particles for identification by electron diffraction was developed. Matrix grain size and second-phase distribution were dependent on heat treatment before irradiation. Retention of the γ, phase on quenching from temperature in the (α+γ) region and the formation of a δ-phase instead of γ$_2$ upon annealing at 650°C were important observations.


Studies showed that the use of SEM for fractographic study instead of conventional replica techniques in the TEM not only shortened the time necessary to obtain data, but also provided information concerning deep cracks that might have been lost using the replica technique.


Conventional metallography, precharacterized surface metallography, and fractography were used to study the nature and extent of reactor-induced microstructural changes in uranium.

Polished metallographic surfaces bombarded by rare gas ions can be etched due to ejection of surface atoms. Two of several possible artifacts which may result from improper etching conditions were studied by statistical metallography: (1) atoms rejected from a U surface collide with gas atoms and redeposit at referred sites on the specimen with redeposition increasing with increased gas pressure, (2) small pores, 0.08-1.0 umd, intersected by the specimen surface become artificially enlarged as a result of selective etching of the pore interior. Techniques used and results obtained are described.


Microscopic inspection using a method similar to grain size determinations was used to obtain relatively accurate estimates of impurity contents in U and U-alloys, UC, UO₂, UN, and UN₃ being the most prominent inclusions.


Fracture surfaces of U-2Mo and U-1Mo-1Nb-1Zr alloys during mechanical properties investigation are reported. Examination revealed two predominant modes of fracture: dimpled rupture at the highest hardness levels in the quaternary alloy and quasi-cleavage rupture in the binary alloy at room temperature. The binary alloy tested above room temperature displayed a fracture mode transition. Results suggest that the high hardness of the complex alloy at room temperature is related to a lower "ductile-brittle" fracture mode transition temperature.


Techniques for the preparation of U-specimens for both macro- and microstructure studies are presented. Procedures for sample preparation, etching, and photography are included. Also included are metallographic standards for the evaluation of grain size, recrystallisation, and inclusion content of the specimens.


Visual standards are presented for rating the degree of recrystallisation of formed U metal, the inclusion content of as-cast U metal, and the inclusion and stringer content of formed U metal.

Single-stage and two-stage methods of replica preparation are described. Shadowing of replicas with Au and Pd alloys improved contrast. Extraction replicas were prepared for study of metals containing intermetallic compounds and inclusions. Thin films were prepared by deposition, deformation, ion bombardment, dissolution, electrolytic polishing, and chemical polishing. Recommended chemical polishing agents for U and other metals and alloys are given.


Paper attempts to summarize the state of the art of U metallography. Conventional preparatory techniques as mechanical and electrolytic polishing methods, etching techniques, and the use of polarized light for the delineation of structure are discussed in detail. Nonconventional techniques such as vacuum-cathodic etching, anodic oxidation, hot-stage microscopy, and strain etching are described and evaluated. The five so-called basic metallographic structures of U: (1) α-annealed, (2) α-transformed - β quenched, (3) α-transformed - β slow cooled, (4) α-transformed - γ quenched, (5) α-transformed - γ slow cooled are illustrated and described. Techniques for determining grain size of U in different conditions of heat treatment and a discussion of nonmetallic inclusions in U including micrographs are briefly described.


A TEM study investigated the effects of cold work on crystallization of α-U. An electropolishing bath, composed of chromic acid, glacial acetic acid, and H₂O was used to prepare thin U films. The standard window technique was used and the thinned specimens were rinsed in anhydrous methanol and dried in a stream of warm air.


Cathodic vacuum etching technique for isothermally treated U-alloys resulted in a very clean etched surface free from an interfering oxide film. The degree of etching was easy to control and the etching parameters - current, gas, pressure, and time - were easy to reproduce. Sharp fine detail in the microstructure and good definition of grain boundaries with somewhat reduced grain contrast were revealed.


A scanning electron microprobe method for identifying small particles in fractured surfaces is described. U-7.5w/o Nb-2.5w/o Zr alloy was analyzed by this method.

Paper presented at 16th Conference on Analytical Chemistry in Nuclear Technology, 1972. The use of TEM, SEM, and electron microprobe analysis of small specimens is discussed. TEM examination of thin parts of U-Ti alloy showed an increase in strength due to precipitation of U$_3$Ti on aging at moderate temperatures. Crystallographic data and microstructure observed in electron micrographs of the U-Ti alloy were correlated. Identification, size, and distribution of carburized U-loaded ion exchange resins were determined by TEM. Results of fractographic study of U-7.5w/oNb-2.5w/oZr alloy using SEM are given.


Foils of U-7.5Nb-2.5Zr alloy suitable for TEM were produced by spark cutting and spark planing followed by a final thinning in a dual jet thinner using a solution of 6%HClO$_4$ in CH$_3$OH. A chemical etch-polishing solution consisting of 35ml HCl, 25ml H$_2$O, 5ml HNO$_3$, and 1ml HF was also developed for intermediate thinning to replace spark planing for faster preparation of $\gamma$-phase material.


U-based and W-based penetrators which had impacted a semi-infinite soft steel target were metallographically studied. The U alloys tend to shear adiabatically at the surface due to: (1) low work-hardening capability, (2) severe stress concentration at the change in penetrator cross section produced during deformation, (3) bending as a secondary influence. Within the bands of adiabatic shear ductile cracks propagate. Martensitic U-2w/oMo alloy is mechanically unstable but the U-1w/oMo-0.7w/oNb-0.7w/oZr alloy shows potential as an alternative to W-Ni-Cu penetrator material when used at >70.76mm/us impact.


Explosive behavior of U-Nb alloys after immersion in HNO$_3$ was found to be related to a finely divided, two-phase microstructure and to a high C content. The HNO$_3$ selectively attacked the $\alpha$-U leaving behind a finely divided U-rich $\gamma$-phase which can oxidize with explosive violence and NbC inclusions accumulated at the surface and were likewise subject to rapid oxidation. The most violent explosions occurred with slow-cooled, slab-cast, high-C alloys having a scale of UO and UO which the HNO$_3$ dissolved and a reactive surface layer of finely divided $\gamma$-U, Nb$_2$C, NbC, and UO$_2$(NO$_3$)$_2$.6H$_2$O remained. When this wet or dry layer was sparked or struck a violent decomposition occurred and a residue of UO$_2$ remained on the surface of the metal.


Two techniques, one standard (normal) polishing and the other an electrolyte-abrasive slurry on a rotating cathode were developed and were suitable for U-alloys, Be, and stainless steel. It is an economic operation in which mounting and polishing steps are eliminated.
U-base alloys were macroetched by two procedures; one consisted of electropolishing followed by immersion etching in HNO₃ and is ideal for large nonflat specimens, the other of mechanical grinding followed by a vibratory polish-attack macroetch which is ideal for macropatterns that are difficult to develop. The limitations of the procedure and photomacrographs of U-alloy castings and forgings prepared are presented.

Metallographic and dilatometric data on dilute U-alloys containing one or more of: Al, Cr, Fe, Mo, Nb, Si, Ti, V, and Zr are summarized. Grain refinement results as a whole are considered and general trends in structural effects produced by alloying U are established.

Method for the metallographic identification of common inclusions in U metal - UN, U(C,N), UC, UO, UN₁₂, and UF₆ is described. Identity was established by removing the inclusion by ultrasonic "jack hammer" and subjecting debris created to x-ray diffraction analysis. Metallographic identification was established on the basis of time required to electroplate the inclusions with Cu and on the character of the Cu deposit.

Thin foil U-specimens suitable for high magnification study with TEM were prepared and metallographic techniques used are described. Studies of three β-quenched, α-annealed U-Si alloys revealed no U₃Si occurred within reasonable annealing times at 500°C but at 600°C precipitation readily occurred at dislocation sites with the number and size of particles increasing with annealing time. Other studies indicated that the deformation of U-Si alloys at room temperature was by twinning and at 500°C by slip.

Note describing the application of extraction replica technique as used in the preparation of electron microscope replicas for the identification of inclusions and precipitates in metals. Stainless steel and U-replicas are presented.

Particle size and density distribution in dilute U alloys were studied by TEM and results agreed with those derived from replica electron microscopy. High resolution in TEM permitted inclusion of particles usually too small to be resolved by replica methods.


Results of preliminary fractography on U-7.5w/oNb-2.5w/oZr alloy and comparison of the topographies by SEM and replica microscopy are presented. From various laboratory experiments six types of failure were identified: (1) fatigue from a fatigue pre-crack Charpy specimen, (2) dimple rupture from tensile specimens, (3) intergranular, (4) quasi-cleavage, (5) intergranular and dimple rupture mixed mode, (6) intergranular and quasi-cleavage mixed mode. All tests were conducted in various corrosive environments and proved that the alloy was sensitive to environment and susceptible to both stress corrosion and H embrittlement types of failure.


Methods of polishing and etching U-1.8-4.0w/oSi alloys are described and the results obtained with the most satisfactory techniques are described. α-U, U₃Si₂, and U₃Si can be distinguished by normal etches but a line etch is required to show the microstructure of the U₃Si.


Thin foils (<2000Å) of U and U-No and U-7.5w/oNb-2.5w/oZr alloys produced by electropolishing using both the "window" and "jet polishing" techniques were studied by TEM. Larger quantities of material were produced by the "window" technique but it is slower, involves more preparation, and examination of areas from the fractured end of a tensile test piece is more difficult than with "jet polishing" technique.


The times required for preparing satisfactory metallographic specimens were determined by studying the behavior of mechanically polished U during electropolishing. Specimens suitable for preferred orientation and microstructural examination were obtained after 10mins in an orthophosphoric acid-ethyl alcohol-ethylene glycol solution. Evaluations were based on x-ray diffraction and microscopic observations.

Techniques for the optical identification of various phases and inclusions in U-metal by controlled oxidation or heat tinting were developed. In this method the various phases assume characteristic colors by which they can be identified. At present UC, U₆Fe, U₃Si, and UAl₂ particles can be distinguished. Sample preparation, heat tinting procedures, and identification are discussed.


Ion-etching technique and SEM were used to study U-7.5w/oNb-2.5w/oZr alloy. Inclusions exposed in relief by etching were studied by energy-dispersive x-ray analysis and samples were examined for microstructural changes within grains after various aging treatments and after mechanical deformations.


Electropolished U samples of varying thermal, chemical, and fabrication histories were studied in a vacuum heating stage. Microstructural changes resulting from the allotropic transformations were recorded. Dilute U-base alloys were prepared and their inclusions examined in situ with an electronprobe x-ray microanalyzer. The effect of annealing on the precipitation of U₆Fe from a dilute solution of Fe in U is shown.
PROCESS METALLURGY


Unique characteristics which dictate the processing methods for U-3.7w/oNb and Zr (\(\alpha'\)), U-10-20w/oNb or Mo (\(\gamma\)), and U-3.8w/oSi and U-40-80w/oZr (\(\epsilon\)) alloys for use as fuel elements are discussed. Induction and arc melting for alloy preparation and brazing, roll banding and pressure bonding for cladding are discussed but for U-Zr alloys discussion is limited to cladding with metallurgical bonding. Detailed metallurgical characteristics of the materials which affect the fabrication operations are provided in the Appendix.


Mechanical and physical properties of Pu and U and their principal alloys are summarized. Fabrication and joining techniques including forging, plating, machining, and welding are presented. The peculiar difficulties and hazards of packaging and handling assemblies containing these materials are discussed.


The types of surface oxidation on U-alloys, the chemical pickling procedures for the removal of the surface oxides, and a proven electrochemical procedure for the removal of the scale surface are presented and discussed.


Fabrication of high density components is evaluated, using the example of 0.20 in d. balls weighing 20 gms. The problem of fabricating a smooth wire, free from kinks and surface imperfections was undertaken. Processing phases - casting, heat treating, forging, rolling, annealing, swaging, and testing - are described. A cold head technique followed by grinding was used to study the manufacture of production quantities of U-balls. The methods of pressing the material, roll filling, and tumbling are described.


Fabrication techniques for reactor materials reported are: (1) powder metallurgy methods, (2) melting, casting, and fabrication of cast structures including U-\(\alpha'\) alloy, (3) welding and brazing, (4) nonelectrolytic chemical plating including electroless Ni plating of U to form U-Ni alloy at the interface, (5) electroplating, (6) explosive metalworking, (7) NDT with emphasis on ultrasonic techniques. For each technique reported the location of where the work is or has been done is included.

Metallurgical processing of U-ores and metals and safe practice in U metallurgy are presented. Some aspects of processing U-alloys are included.


Method of preparing dimensionally stable U-Cr alloys by adding 0.1-10a/oCr to U and heating to a temperature above the $\gamma$ - $\alpha$ transformation temperature, quenching to $\alpha$-phase, and holding at this temperature until transformation to $\alpha$-phase crystals is complete.


Method of preparing U-1-3w/oMo fuel alloys by vacuum melting the two metals in a graphite crucible, coated with a reducing cement, casting and cooling the ingots at a rate of $10^\circ$-80$^\circ$C/min. The microstructure is characterized by grain size of 20-80um, with no preferred orientation, making them superior to unalloyed U with respect to creep, traction, and cycling resistance.


Method of preparing U-3-6w/oMo fuel alloys by vacuum melting the two metals in a graphite crucible, coated with a reducing cement, casting, heating the ingots to 675$^\circ$-800$^\circ$, quenching to 550$^\circ$ in a fused salt bath, and finally cooling at a rate of 20$^\circ$-60$^\circ$C/min. Alloys have microstructure with no preferred orientation and grain size of 60-80um making them superior to unalloyed U with respect to creep, traction, and thermal cycling resistance.


Method of making U-0.05-0.3w/oCr-0.5-3.0Nb alloys for use as nuclear fuels present in which U is present in the form of a fine-grained dispersion of $\gamma$-phase U in fine-grained (<40um) $\alpha$-phase, which is stable and free from preferred orientation. Alloy is cooled from the $\gamma$-phase 300$^\circ$-50$^\circ$C/min, then reheated to the $\gamma$-phase, maintained for a period <3 hrs, cooled to room temperature at a rate of 400$^\circ$-4$^\circ$C/min and subsequently reheated in the $\alpha$-region.
Melting and Solidification


Process for obtaining sound, segregation-free ingots of U-2.2w/oNb and U-0.75w/oTi by induction melting is described and casting parameters are discussed.


Development work and production experience of induction melting and casting of U-Sw/oZr-l.5w/oNb alloy for coil materials in nuclear reactors are described. Furnace design, crucible materials Zr dissolution, and ingot soundness are discussed.


U-Mo shapes of varying sizes and thicknesses were cast from a molten charge using specifically designed graphite distributors and molds. Solid cylinders, hollow cylinders, and flat plate shapes were cast in gang molds. All castings had smooth surfaces and the solid shapes were cast to 0.006in tolerance on all dimensions except length.


Consumable and non-consumable arc-melting techniques have been successfully used in production melting of U-12w/oNb and U-12w/oMo alloys. Control of macro-segregation and removal of contaminants are the major problems that have been encountered. Arc-melting techniques have been limited but possible future developments are considered.


The techniques and equipment for induction melting of binary U alloys containing Nb, Zr, Mo, Si, Cr, and Al with melting points <1600°C are described. The use of coated graphite and other materials as crucibles and molds are discussed.


Processing techniques including melting, casting, and heat treatment for making structural U-base alloys having high strength and good corrosion resistance are described.
Foundry practice suitable for casting U parts of exceptional soundness and uniformly high mechanical properties is described. Vacuum melting and pouring are required, and the casting produced must be kept under high vacuum until cooled to ~1200°F.

Mass production application of U-8%Mo alloy for "spotting rounds" for atomic warhead firing of weapons using a coaxial melting-casting process with a refractory mold is reported.

Studies to determine the method and parameters of heat treatment for homogenization of a U-10%Mo alloy were conducted. The specimens were tensile tested and metallographically examined. The as-cast material indicated evidence of coring, heat treatment homogenized the material, and the wrought material indicated evidence of chemical inhomogeneity.

Practical methods for producing tonnage quantities of U-Mo, Nb, and Zr alloys and dilute alloys of Si, Fe, and C are reviewed. Comparative data on grain size and grain refinement by alloying additions of static and centrifugally cast, rolled, and heat treated sections are presented.

A method of casting blind holes, having a L/d ratio of 5:1, into a small U-8.5%Mo alloy shape is described. A rammed bentonite-bonded graphite mix was used for both the core and the mold. Castings were sound and possessed satisfactory properties.

An oscillating crucible viscometer capable of precise viscosity determinations at >1000°C was constructed. The operating details were developed and the viscosities of U and two U-Cr alloys were measured.

Large cylindrical U ingots cast in graphite molds were studied using a mathematical model and a digital computer. Heat flow during solidification is described.


U and U-5%Zr-1.5%Nb were cast with Zr and Zircaloy-2 molds of various sizes and shapes. Provided the molds were out-gassed at elevated temperature before use and proper preheating precautions were observed, the castings made in cans of circular cross section were well bonded and sound.


Equipment and procedure for vacuum melting and casting of low-alloyed U into tubes weighing <40kg each. The method is recommended for metals with a high gas content or high vapor pressure which are unsuitable for electron beam melting. Test results are presented and important design parameters are derived.


U, U-3.6%Zr, and U-1.0%Nb alloys were melted and cast in a skull furnace by using bottom pouring crucibles incorporated with a centrifugal casting device. Eight pieces of 1/2 in o.d. x 6 in L rods were cast in vacuum (<1 torr) using U-alloy consumable electrodes and there was no contamination or segregation in the cast shape but density was slightly less than theoretical value which may be improved by heating up the mold.


Vacuum induction furnaces of 180, 350, 3000lbs. working capacity with ceramic crucibles were used for the melting and casting of U-Mo alloys, with specifications on Mo and C content. Mo is easily held to within ±5% of requested target analysis but C is very difficult to control. The types of furnace and crucible used depended on C level that must be maintained.

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Crucible materials for melting U and U-alloys were evaluated. Depending on the application ZrO$_2$ fused 97% MgO, and graphite have been found to be the most suitable. Ceramic-type molds are superior to graphite for small sections (<4sq in) and those shaped castings which prohibit the economic use of machined graphite molds. Heating cycles, addition mechanisms, and furnace conditions for melting are discussed.


Three types of Nb segregation occur in U-Nb alloys: (1) dendritic coring, (2) macrobanding, (3) gross-ingot segregation. The origin and nature of these inhomogeneities and the optical and x-ray metallographic techniques for their identification are discussed. Half-height peak widths, peak intensity ratios, hardness, and optical microstructure measurements are related to segregation. Procedures for minimizing segregation are described.


U and U alloys were cast into slugs ranging in weight from 1 to 1800 kg in various size vacuum furnaces with either micarta tube or steel tank construction. Graphite was used for crucibles and molds which were coated with a ceramic to prevent metal-graphite contact. The use of uncooled Cu induction coils, induction stirring, and castable refractories are described. Neither Cu nor glass provided any advantage when compared with graphite.


Vacuum-induction casting of U-Mo alloys into both thin (0.01-0.10in) and long (56in) plates is described. Melting and casting cycles and casting techniques are explained. The plates were evaluated by radiographic and metallographic examination and by chemical analysis.


U-base alloys containing Ti and Fe were prepared using consumable-electrode vacuum-arc melting. Electrode and ingot production are described.

U-8.5w/oMo alloy plates were investment cast in a vacuum induction furnace using a bottom pour technique. Three specimens (5in, 4in, 3in) were chill cast and one (5in) remained unchilled. Tensile properties, ductility, and grain size of the cast plates were determined.


Coreduction method of producing U-3, 6, 10, 14w/oMo and U-2w/oZr alloys at the Paducah Metals Plant is described. Various ratios of Mo powder and MoO₃ were blended with UF₄ and Mg and charged into refractory lined (MgF₂) bomb shells for reduction. Surface impurities were removed from the resultant alloy debris by roasting at 1200°F for ~2 hrs, followed by a H₂O quench, melted under vacuum in an induction furnace, and cast into ingots (3in d.). The U-2z alloy debris were also produced by the coreduction of UF₄, ZrF₄ and Mg.


Coreless induction furnaces may be effectively standardized by tuning the high-frequency by adjusting the variable capacitor banks to obtain a standard impedance of 3.3-3.60. Correct time-power control also controls the C pickup.


Problems related to U and Th and their alloys, because of their high reactivity, are presented. Data on thermal analysis, the use of the arc furnace for casting various shapes, and determination of melting points are included.


This study provided preliminary qualitative information on two important points regarding oxidation reactions occurring in molten U-alloys: (1) relative ability of various elements to deoxidize U, (2) relative stability of the oxides formed as a result of deoxidation. Ideas have been proposed regarding the extent to which U can be purified by conventional melting techniques.

Melting, refining, and casting procedures of depleted U-alloys using vacuum induction melting techniques, a hand-rammed commercially pure MgO crucible, and a phosphate-bonded ZrO₂-SiO₂ mold are discussed. Casting with ductile qualities in the as-cast condition and a reduction of residual gas concentration in the alloy resulted.


Design, development, and experimental evaluation of multiple mold centrifugal casting machine with precision Cu molds used in conjunction with high-vacuum induction heated melting and pouring to produce U-2w/oZr alloy fuel rods or slugs is described.


U-10Mo alloys were cast into slugs 0.16in/d. and 12in/l using a multicavity mold. The alloys were vacuum induction melted in MgZrO₃ coated graphite crucibles and cast into coated graphite molds. The graphite used is an important variable in the process. The homogeneity, surfaces, and dimensional tolerances of the 3850 slugs of as-cast U-10w/oMo met all the requirements for nuclear fuel materials.


Various pure oxide refractories used for melting and casting of U, U-alloys and other metals are discussed. The purity and methods of fabrication of the refractories, coating materials, and methods of applying them to molds and crucibles are considered. A typical vacuum furnace for melting U and U-alloys is described.


A continuous carbide layer was formed when molten U contacted graphite. The layer clads the graphite and probably protects the graphite against rapid corrosion. The layer itself is not stable toward U.
Specifications are given for methods of preparing and heat treatment of U-Mo-Nb and U-Mo-Nb-Zr alloys for reactor fuels that are stable, fine grained, and free from preferred orientation or distortion as-cast.

Process for preparing a U-0.3-0.6%Mo alloy by melting under vacuum in a graphite crucible covered with a reducing cement as Al₂O₃ or CaZrO₃, maintaining the product in a molten state and pouring it into molds in the form of bars or tubes, heat treating at 675°-800°, followed by a stepped tempering with the main tempering 550° for 15 min and cooling to the ambient temperature at a rate of 20⁰-60°C/min to produce a microstructure with grains of 60-80μm.

Method of preparing U-0.4-0.55%Mo and U-0.15-0.25%Mo 0.1-0.15%Al alloys having fine as-cast grain structures after being subjected to accelerated cooling in the range of 75⁰-300°C/min.

Process for casting U-alloys in which specially designed mold and metal are first brought to a temperature higher than the solidification point of the U-alloy and then cooled by bringing into contact with the mold a cooling material which is initially at a temperature below the solidification point of the casting metal.

Dispersion strengthened U was prepared by the mechanical mixing of UH₂ and Al₂O₃, compacting in vacuum, dehydriding in vacuum at 260°, and extruding at 620°C. The resulting dispersions were very fine and the creep-rupture properties were superior to hot rolled U.

Powder production, compacting, and sintering techniques for shapes consisting of U, U-alloys, and U-compounds are described. U samples prepared by hot pressing are high in hardness and tensile strength and low in ductility. Special heat treatments of the sintered product are necessary to increase ductility and lower hardness.


U-base binary alloys containing 0.5-5.0w/o Mo, Nb, Si, Cr, Zr, or V (fine powder <250 mesh) may be fabricated by powder metallurgy: cold pressing and sintering, hot pressing, or powder rolling. Cold pressing, being the most straightforward approach, was used. Mo was the most successful alloying addition, increasing ductility and controlling grain size. The smaller the Mo addition, the larger the grain growth, and grain growth can be eliminated at >3.0w/o Mo. In thermal cycling, additions of >1.0w/o Mo showed excellent stability.


Preparation by powder metallurgy, melting and coreduction, fabrication, heat treatment, physical and mechanical properties, corrosion, and irradiation effects of U-alloys are included in this chapter. Also included are 198 references and a Selected Reading List.


Infiltration processes for the manufacture of U-alloys, U-pseudo-alloys and combinations of U-compounds with metals are described. A metal powder or liquid was infiltrated into a porous body by means of capillary action at 750°-1250°C. Strength of infiltrated materials can be increased by incorporating metallic reinforcements (rods, nets, coils, etc.) into the skeleton prior to infiltration.


The feasibility of producing U-Ni, Cr, Mo, Nb alloy powders by direct reduction of compound oxides and mixed oxides by Ca was studied. Reduction usually begins at >630°C after which bomb temperature is rapidly raised to above the liquidus of the alloy. Compound oxide reduction resulted in high heats of reaction, low yields, irregular particle size, and inhomogeneous composition. The mixed oxide process was superior, in all respects, except particle-to-particle composition variation.

U-10-15w/oMo alloy rods were fabricated by hot, rotary swaging both U-Mo powder mixtures and prealloyed powders contained in Inconel tubing with a Mo barrier. Densities >95% of theoretical were obtained with reductions in area of 55% at 1050°-1100°C. Alloy homogeneity was good for prealloyed powders, but traces of Mo-rich, γ-phase persisted in rods made from elemental mixtures.


Powder metallurgy processes in reactor metallurgy and atomic energy work are emphasized. Experimental applications included the use of powder metallurgy in determining phase diagrams for various alloy systems and also studying homogenization or diffusion specimens.


Powders of Zr, Zr-alloys, and both α-U-4%Mo and γ-U-12%Mo alloys were produced by the hydride method or by a direct comminution of selected turnings. A fluidized bed process was developed for the reduction of the hydride. Results of a study on hydriding kinetics are presented.


The electron probe was used in the quantitative analysis of individual microparticles of U-Mo and U-Nb-Zr alloys. The characteristic x-ray intensities of the alloyed metals excited by the electron beam for absorption were corrected by a computer program. A simplified specimen preparation and handling procedure was devised. The accuracy of the method is comparable to that of the conventional chemical analysis of bulk samples.


Processing of uniformly porous U and U-alloy components by hot pressing of powders is described. Closed porosity is encountered which decreases with increasing porosity U-Mo, Si, Nb, Cr, Zr, V densification and grain sizes are tabulated.
Thermomechanical Processing


γ-forming and isofoming treatments were investigated as a means for producing fine grained α structure in U-2.5w/oMo, U-4.5w/oMo - 1w/oV, and U-2w/oNb-2w/oZr alloys. γ-forming had little or no effect on the shear transformation to α1 or α2 but fine grain α structures with some compressive ductility were produced. Additions of V to U-Mo raised the eutectoid temperature so that isofoming could be performed at higher temperatures. Hardening was caused by high isofoming deformation, usually at 525°C and by reheating prior to final deformation.


A literature survey including unclassified reports appearing in Nuclear Science Abstracts and also journals, patents, books, and other published material on the subject of extrusion of U, U-alloys, and U-compacts covering the period 1951-1961. A subject index is included.


Method of producing a blind hole in U-8.5%Mo alloy core of a 15mm spotter round by hot reverse extrusion is described. Parts had smooth surfaces and were within specified concentricity requirements. It is believed other U-alloys could be used.


U and high-purity binary U-alloys were developed by thermit bomb reduction of massive charges. Remelting step to convert to ingot forms is not required. Press forging and extrusion procedures are discussed. Examples of structure and the influence of alloy additions on both forming characteristics and product quality are illustrated.


Levels and distribution of residual stresses encountered in specimens from the manufacture of hollow U fuel elements were determined. To obtain data, an electrochemical procedure was developed and used for removal of metal from the bores of the specimens. Residual stresses in H2O quenched specimens was <60,000 psi and in the oil-quenched specimens <12,000 psi. In the final production step earlier stresses are almost cancelled; the final stresses were 15,000-20,000 psi. After various mechanical and thermal treatments, measurements of internal friction and Young's modulus went through a maximum and minimum, respectively, with increasing severity of quenching from a β temperature.
147. Grigorovich, V. K. and Golovkina, N. E., "Deformation of Binary Alloys of Uranium Containing 0-20 at % Mo, 0-30 at % Nb and 0-100 at % Zr When Subjected to Cyclic Heating up to 600°-900° C." Translated from the Russian for University of California, Lawrence Radiation Laboratory, Livermore. Report No. UCRL-Trans-10453, 1970.

Shape stability and the tendency toward surface hummock formation on cyclic heating of U- up to 20a/o Mo, U- up to 30a/o Nb, and U- up to 100a/o Zr alloys were studied. Specimens were forged, quenched from 800°, and annealed at 500° C for 24 hrs. It was found that shape stability after cyclic heating increases as the alloying additive is increased to 20-30a/o. Residual deformations increase at the upper temperature limits of the heating cycles and the shapes that undergo a + B transformation are sharply changed.


U-8Mo and U-8Mo-1Ti alloys were investigated to determine the advantage of forging depleted U components directly from co-reduced ingot stock eliminating remelting and alloying operations. Upset forging and cogging hot-working methods were used and improved the mechanical properties, especially ductility and Charpy V-notch impact.


Practical problems experienced in extruding U-alloys in a laboratory are described and suggestions for handling these problems in a production situation are given. Mechanical aspects of the thermomechanical working of U alloys are discussed in the Appendix.


Effects of extrusion variables - prior texture, billet and linear temperature, reduction in area, and ram speed - on textures were examined by crystallographic pole figures for extruded U rods and tubes. In general, increase in extrusion temperature and increase in reduction ratio have similar effects. Final texture can be deeply effected by prior texture. Ram speed has no important effect between 13 and 101n/min, and temperature effects the development of a strong 110 axial texture at temperatures > 525°, a strong 010 axial texture at < 400°, and a mixture of both at intermediate temperatures. A 900° C, γ-phase extrusion is shown to have a mild 100, 010, 021, 001 texture.

The effect of thermomechanical processing on the hardness and grain morphology of U-3Mo alloy was studied. The hardness was increased when deformation occurred as an intermediate step below the γ→α transition temperature. Maximum hardness (Rc 62) was attained by heating at 760°C, followed by a H2O quench and further deformation at room temperature and also by aging at 380°C for 2 hrs. There was no significant variation in grain size or systematic variation of hardness with grain size.


A rolling study in which as-extruded or β-heat treated U and U-base alloys were fabricated in the form of tubes and rods was made. Roll separation force, mill power input, time of rolling, and specimen temperature for each specimen and pass values were recorded using the necessary instrumentation. Data for each specimen and pass were tabulated. As-extruded U in either tube or rod form was easiest to fabricate and U alloyed with Fe, and most difficult with Si and Al.


Microstructure and mechanical properties of technically pure and lightly alloyed U after fabrication or after fabrication followed by tempering or heat treatments were studied. The quality of U and characteristics of the alloys, and fabrication by casting and plastic deformation, are evaluated. Results obtained from each method are compared.


Preliminary studies of the mechanical properties of both hot-worked and as-cast U-alloys containing 2-3w/o Mo, Nb, V, and/or Zr are reported. Commercially produced alloys with differences in composition, processing variables, and heat-treatment temperatures showed no significant differences in properties between the cast and wrought alloys. Results of all tests are presented in tabular form. Further studies using split heats are suggested.
The results of work on the thermomechanical treatments of U-2-6w/oMo and other U-alloys (minimum hardness Rc 54) containing γ soluble elements are summarized. Results indicate that the significant changes expected in strength and ductility were not produced. Low temperature γ-phase forming and isoforming were investigated. Some grain refinement and increase in hardness was achieved by γ working and it is considered that controlled deformation of the stable γ-phase could produce improved properties.


A cam plastometer was used to determine the resistance to deformation of U-1, 2, and 5w/oMo alloys at several strain rates. In general, the deformation resistance decreased with increasing temperature. Results have been applied to the formation of hot rolled schedules for the U-1-2w/oMo alloys.

The use of pure Au as a catalyst appeared to have promising characteristics for catalyzing diffusion bonding reaction of joining U to itself. Phase diagrams for U alloyed with Au, Cr, Co, Cu, Fe, Mn, and Ni are given.

U-0.75w/oTi alloy plates, 360 mils thick, were welded using multipass electron beam methods. Partial penetration electron beam welds having a minimum penetration of 75-85% of the plate thickness were obtained without experiencing meltthrough ("spiking") or damage at the backside of the joint. Single pass methods were unsuccessful.
Techniques for producing sound welds with U sheet, plate, and shapes using inert-gas W-arc method is described. Microstructure and mechanical properties of the welds and other welding techniques are also included.

U welds free from porosity, cracks, and oxide inclusions were produced by an inert gas-shielded arc method. Tensile and yield strengths exceeded the strength of cast metal. Surface condition and purity of the inert gas used affected weld soundness. Straight polarity direct current was used to achieve maximum penetration and to provide arc stability.

A machining study in which tool force and surface finish were determined as functions of six machining and tool-geometry variables was made of a U-4.3w/oNb alloy. Nose radius, top rake angle, front clearance angle, feed rate, surface speed, and depth of cut were parameters investigated. Empirical equations for tool force and surface finish were derived as a function of the independent variables. Tool-force minima were observed in surface speed and feed rate and were used advantageously when optimizing parameters.

Adaptability of Ar arc, metal arc, and resistance spot welding processes to joining of U were assessed. Chemical and physical properties of U and their effect on welding are described.

Joining of U to U using a number of soft solders and fusible alloys is described. Ultrasonic soldering iron proved to be the best method for joining U to itself and to other metals such as stainless steel. Soldering characteristics of U may be best compared to those of Al.

Summary of experience in machining and handling depleted U acquired at Oak Ridge Y-12 Plant is presented. Parameters for turning, boring and facing, cutting, drilling, gun drilling, reaming, threading, tapping, milling, and grinding are given. The use of carbide tooling is recommended. Health and safety hazards are also discussed.


An electron beam welding study to determine the effects of the variations of welding parameters on the weld characteristics in partial penetration welds on 0.08-0.25in stock sheets of U-7.5\%Nb-2.5\%Zr alloy is reported. In the thin sheets (0.08in) welding defects were in the form of porosity and in the thicker sheets (0.25in) in the form of weld spiking and cold sheets at the weld root. Frequencies of 200-400 cps in a circle generator drastically reduced the number of weld defects.


The addition of a dibasic NaPO_{3} during machining operations of U increased the Cl tolerance ~10x. In the phosphate coolant sodium caprylate is required to retard corrosion of cast iron in machine tools and benzotriazole is required to eliminate corrosion of the Cu alloys in the coolant circulation system and bearings.
PHYSICAL AND MECHANICAL BEHAVIOR

Physical Properties


Quenched U-alloys containing Zr, Cb, and Mo, metals which have 4, 5, and 6 outer electrons respectively, were measured from 90° to 300°K. General features of the three systems are the same, with anomalies at - 0.5 and 2.0a/o of the solute.


The electrical resistivity and superconductivity of a series of α (orthorhombic) and quenched γ bcc binary U-alloys containing up to 15a/o Nb and Mo where investigated at temperatures down to 1°. Anomalous resistivity behavior occurred in both types of structures. The γ-alloys possessed a negative temperature coefficient from room temperature to the superconducting transition point, - 2°, and for the α-alloys - 1°K. A hypothetical band structure for U was determined. Work was also conducted on some intermetallic U compounds.


Technique for making dynamic measurements of the Grüneisen coefficients of high atomic number materials and measuring the Grüneisen coefficients of U, U-1.5Nb/Mo, and heat treated as well as as-cast U-10Nb/Mo alloys is described. Ta was used as the reference material. Results showed γ-phase had slightly lower coefficients than the α-phase and that the U-10Nb/Mo alloy specimens, despite differences in grain size, had similar coefficients.


Thermal expansion, specific heat, electrical resistivity, thermal conductivity, Seebeck coefficient, Poisson's ratio, and the shear Young's and bulk moduli were measured as a function of temperature for two polynary U alloys, U-3/4Nb-3/4Zr-3/4Ti and U-1Nb-12r-1Mo-1/2Ti.


Physical properties including elastic and anelastic moduli, phase transformation temperatures, expansion coefficients, magnetic and paramagnetic susceptibilities, conductivities, and crystallographic orientation of U and Pu alloys are described. Phase diagrams for U-Mo alloys are presented.

Electrical resistivity and magnetic susceptibility of U-70a/oZr and U-25a/oMo alloys are described and interpreted in terms of a theoretical structure derived by x-ray work of P.C.L. Pfeil. Results showed fair agreement with the proposed structure based on a contribution of four electrons per atom by U in the γ and γ′ modifications.


Low-temperature internal friction of U-0.4w/oMo alloy subjected to various heat treatments was measured to explain the behavior of Mo atoms in the alloy and the swelling of U-alloy fuels. On the internal friction temperature curves three maxima were discovered from 77°-360°K: (1) at 120°K, in alloys β-quenched or tempered at < 350°C, (2) at 150°K, in alloys in the annealed or fully tempered condition, (3) at 23°K obtained by β-quenching and disappeared after tempering at 100°C for 1 hr. Young's modulus was decreased abruptly by β-quenching and recovered during the room temperature aging, the rate formula of recovery agreed with the martensitic and nucleation theories.


A three-phase equation of state for metals was developed, based on the assumptions: (1) entropy of melting is independent of pressure, (2) temperature dependence of the specific heat in the liquid is a universal curve, scaled on the melting temperature, (3) pressure dependence of the melting temperature is given by a modified Linnemann law, (4) liquid-vapor region may be described by a hard-sphere model perturbed by van der Walls attraction. The equation of state is laid out in a P(V,E) form for use in numerical hydrodynamics calculations. Parameters for Be, Mg, Al, Ti, stainless steel, Ni, Monel, Cu, Nb, Ta, W, Au, Pb, Th, Mulberry, and U are given. Experimental evidence for the validity of the equation of state is presented.


The effect of alloying additions on the ignition of U was studied and differences in the temperature-time curves for U alloys containing 0.5, 1.0, and 2.0% of 24 different elements were noted. Results were interpreted in terms of the effect of the alloying addition on the previously demonstrated transition of the oxide at 400°-500° from an autocatalytic to a protective form. Additions of Al, particularly, and certain other alloys containing, for example Mo, Nb, Ti and V, inhibited the oxide transition and thereby lowered the ignition temperature. Additions of Cu, in particular, increased the protectiveness of the oxide formed at > 500°C. Complex but reproducible temperature-time curves were obtained when both effects occurred within the same alloy.

Methods of measurements of ignition characteristics of Zr and U were developed and used to determine the effects of variables as surface preparation, metallurgical history, specific area, additives to the metal, U content and presence of moisture in the oxidizing gas. Ignition temperature was determined for binary U-base alloys containing up to 2% of Al, Nb, Si, C, Mo, Ru, Re, Zr, H, Fe, or Cu.


Thermodynamic properties of the U-6Mo system were investigated from 1048°-1173°K using the ENF method with a liquid electrolyte. Results indicated that the excess thermodynamic functions change their signs depending on the concentration. The concentration dependencies of the excess entropy, enthalpy, and free energy of mixing of the solid solution were calculated at 1100°K, based on new physical concepts advanced for the solid solution. An interpretation to explain the double change of sign of the excess entropy of mixing and the change of sign of enthalpy of mixing as a function of concentration is offered.

Mechanical Properties


Elastic constants for U-7.5w/oNb-2.5w/oZr alloy (Mulberry) were measured in the pressure range, 1-18 kbar. Initial C11 and C12 values were found to be 1.34 and 0.92 mbar. For dC11/dP and dC12/dP values of 5.8 and 4.1 were obtained. Initial bulk modulus, K, was 1.06 mbar and dK/dP was determined to be 4.6.


The stress-strain response of polycrystalline, γ-quenched U-7.5w/oNb2.5w/oZr alloy was studied as a function of strain rate and compared to equilibrium stress-strain tests which were also performed on prestressed samples. Stress-induced transformation first occurred at stresses > 40,000 psi, was complete at stresses < 120,000 psi, and involved a transformation from a γs(bcc) structure to a γo(BCT) structure which is postulated to be a thermelastic martensitic transformation. Tensile tests indicated strain rate sensitivity at strain rates > 3.0x10⁻²/min−¹. A semiempirical equation describing the stress-strain behavior in terms of stress induced phase transformation was developed.

Work on U-Nb-X ternary alloys included studies on $\delta$-quenched and tempered U-1%Nb-0.15-0.2%Cr alloys. Laboratory tests showed these alloys to have good structural stability at 550°C, improved mechanical resistance both when cold and at high temperatures (550°C), and improved thermal cycling resistance (0°C-550°C). Creep resistance was higher when the Cr content was low (0.15%).


Mechanical properties - hardness, creep, brittleness, and tensile strength - of U-alloys containing <10%Mo, Zr, Ti, and Al in the temperature range where they are believed to be useful as nuclear fuel materials are presented in tabular form.


Temperature dependence of strength and elastic modulus was measured for a series of U alloys in the $\alpha$ region (< 500°C) and in the $\gamma$ region (700°C-900°C). In the $\alpha$ region, U-20%Zr and U-70%Zr were stronger than U-20%Nb and U-20%Mo, but weaker than the ternary U-10%Zr-20%Nb. In the $\gamma$-phase the U-20%Nb and U-20%Mo alloys are strongest. Young's modulus of $\alpha$-phase decreased with increasing temperature, then fell sharply when transformation $\alpha \rightarrow \gamma$ began.


The deformation of compressed specimens of martensitic $\alpha$-phases and metastable $\gamma$-phases of quenched U-3-30/oMo alloys was studied by metallographic and x-ray techniques. The various twinning phenomena in $\gamma$-phase deformation are discussed in terms of the order thought to exist in metastable phases.


Principal factors which influence the plasticity of $\alpha$-U are summarized. Both the temperature dependence and strain-rate dependence of the flow of stresses in U appear to originate in the U crystal itself. Although $\alpha$-U can have reasonable ductility at room temperature and normal strain rates it appears to be basically unsuitable for high strain rate applications.

The influence of tempering heat treatments associated with extrusion on the ductility of γ-extruded U-5w/o Mo-3w/o Nb and U-3w/o Mo-3w/o Nb alloys was studied. Mechanical properties of both alloys were measured in the extruded, extruded and annealed, and quenched and tempered conditions. Maximum elongation of 13.7% with a low amount of work hardening, was obtained for U-3Mo-3Nb alloy after annealing 1 1/2 hrs at 850°C followed by a rapid cooling in H2O at 21°C and 17.3%, with a large amount of work hardening, for U-5Mo-3Nb alloy after vacuum annealing, normalizing, γ-phase solutionizing at 815°C, and quenching in H2O at 21°C.


Mechanical properties of U-1Mo-.8Nb-.8Zr alloy were measured after oil-quenching from the γ-phase and aging in the temperature range 450°-1200°F. Isothermal interrupted quench treatments were evaluated as well, and resulting microstructures were observed and correlated with mechanical test results. Hardnesses from 32 to 56Rc were developed by aging and they correspond to tensile yield strengths from 92-256 kpsi, to bend strengths from 247-500 kpsi, and to compressive yield strengths from 160-320 kpsi. At lower hardness levels, the alloy appeared to develop a tough structure, at high hardness levels, significant ductility was present after oil quench treatment (45% compressive deformation at 55 Rc); since the work-hardening rate is low, relatively low tensile elongations were obtained.


U-2w/o Mo alloy was subjected to a series of aging heat treatments to select the microstructure that provided optimum mechanical properties for high-density projectile applications. Results showed that the lamellar structure (32Rc) obtained by aging at ~1000°F possessed the highest toughness and ductility. Aging in the α + γ region (1050°F) resulted in a basketweave structure which was accompanied by an increase in hardness and a decrease in toughness near the eutectoid line but at higher temperatures the properties were recovered.


Summary of data found on U-alloys containing <1w/o Mo, Nb, Zr, and Cr and U-7.5w/o Mo-2.5w/o Zr alloy in the unclassified literature from 1947 to 1969, interpreted and evaluated to present the most accurate property information available. Phase relationships, corrosion, dimensional stability, mechanical and physical property data are included for each alloy.

Effect of both heat treatments and the small addition of a third element on the mechanical properties of U-0.5Ti alloys was studied. Aging for 1 hr at 400° increased tensile strength from 144,600 to 173,000 psi and reduced elongation from 22.8% to 18.8%. Aging at 200° for 1 hr did not significantly change properties but aging at 600° for 1 hr overaged the alloy thereby decreasing strength and ductility. Quenching was detrimental to both strength and elongation.


Mechanical and metallurgical properties of several U-alloys were investigated. U-3.5w/oMo exhibited increased thermal stability and creep strength at 900°F. These properties were further increased in U-3.5w/oMo-0.1w/oAl alloy. U-2.0w/oZr-0.1w/oAl alloy exhibited poor dimensional and geometric stability.


Evaluation of the effects of various alloying element additions on the dimensional stability - creep, hardness, tensile properties, and microstructure - were studied. U-3.5Mo, U-3.5Mo-0.5Si, U-3.5Mo-0.1Si, U-3.5Mo-0.1Al, U-3.5Mo-0.1Al and U-2Zr-0.1Al were the alloys studied and unalloyed U was used for reference.


The first and fourth quadrant yield surfaces of a U-0.75%Ti alloy were determined. The specimens were solution treated at 800°C for 20min, followed by a H2O quench, and then electron beam welded. The electron beam welding process was used to weld two identical halves together to make one complete specimen and the effects of the process were tested. Results indicated that the weld metal is stronger than that of off-weld metal and fracture did not start or propagate through the weld. The tentative biaxial yield surface is described by the maximum strain criterion.


A polynary uranium alloy system containing equal amounts of Nb, Zr, and Mo up to 2w/o and 0.5w/oTi is described. Mechanical properties in both extruded and solutionized and aged conditions indicated UTS ~ 300 ksi, 0.1%YS > 200 ksi are possible.

Mechanical properties were correlated with constitution diagrams and structure of forged binary U-alloys containing 1-15% Zr, Nb, and Mo and U-60-99% Zr alloys. Results indicated: (1) effect of alloying elements on heat and creep resistance were irregular, (2) Zr lowered the creep rate more effectively than Nb or Mo, (3) Mo increased heat resistance at 550°-580°C more than Zr or Nb.


The yield surface of γ stabilized U-7.5w/o Nb-2.5w/o Zr alloy under biaxial stress conditions was determined and is best described by Von Mises theory of yielding.


Compilation of mechanical property data on binary and ternary U-base alloys containing a maximum of 5%Hf, Ir, Mo, Nb, W, Ta, Th, Ti, V, and/or Zr prepared and tested at Oak Ridge, Y-12 Plant and compiled in their technical reports issued though October 1969 as well as unpublished data (designated as ABT) is presented. Where applicable the report number follows the data presented.


Tensile tests were performed on oil-quenched and Hg-quenched U-1.5-30a/o Mo alloy specimens. Relationships of tensile results vs nominal composition and hardness vs composition were plotted. Circumferential cracking due to SCC by air occurred in alloys containing < 20a/o Mo while intergranular cracking occurred with > 20a/o Mo. Mechanical properties and the results of metallographic and x-ray analyses are presented in tabular form.


Effects of dynamic tension loading on the mechanical properties of γ-phase U-7.5w/o Nb-2.5w/o Zr alloy are presented. Test data were taken at 23°, 200°, 300°, 400°, and 500° at strain rates varying from 10⁻³ to 160in/in/sec. Dynamic loading caused only a slight decrease in ductility and tensile strength increased moderately with increasing strain rates. TTT curves indicated that the γ-phase begins to transform to a cellular structure in a few minutes at temperatures slightly > 300° and to a pearlitic structure (α + γ 2) at temperatures > 500°C.

As-cast and wrought U-10w/oMo alloys with varying C content (71-8800ppm) and having varied heat treatments were tensile tested on a universal machine at strain rates of 4.8x10^-8 and 0.057in/in/sec and on a modified Dynapak at strain rates up to 100in/in/sec at 75°-600°F. Relatively low strain rates were used to determine the susceptibility to SCC and higher strain rates to determine the sensitivity of the mechanical properties to strain rate effects.


Thermomechanical properties of U-6-10w/oMo alloys, in which the γ-phase can be retained to room temperature and below, were determined. The temperature dependence of the mechanical properties of U-6w/oMo alloy from 77°-800°K indicated large dependence at < 300°K and small dependence above that temperature. Fractography studies revealed only ductile-type fracture at > 150°K. Plastic deformation and shear dimple size increased as the temperature increased. The percent elongation and toughness decreased just above 400°K.


Notched and unnotched strengths for several alloys including U-8Mo-0.5Ti were determined at load duration of 10 msec-5 min at -320° to 500°F. With Kt=9.5, the notch strength of all the alloys was less than the unnotched strength at low temperature under both dynamic and static conditions but exceeded the unnotched strength in the higher temperature range.


The mechanical properties of U-2Mo and U-1Mo-.7Nb-.7Zr alloys heat treated in various ways were studied and compared. Embrittlement occurred in both alloys at 371° and just above the eutectoid line (565°C). The quaternary alloy generally had superior mechanical properties - hardness, flow stress, toughness, and ductility - but further tensile tests are required.


Tensile, compressive, and impact properties of γ-quenched and aged U-4.5,6.8w/oNb alloys and U-7.5w/oNb-2.5w/oZr alloys are provided in graphic form. Tensile property data were obtained from 527 tests using 264 different combinations of composition, time, and temperature and impact property data from 100 tests using 36 combinations of composition, time and temperature.

Tensile and impact test data correlated with metallographic and x-ray studies for U-7.5w/oNb-2.5w/oZr alloy are presented. Strength is increased with no loss in supersaturation by aging at < 800°C for up to 80 hrs. Data are interpreted in terms of plastic deformation occurring by the motion of twin interfaces and in the pinning of twin interfaces by ordering on aging.


Tensile test data for U-11,15,17 and 19w/oNb alloys in both the solution-quenched and aged conditions are presented and correlated with concurrent metallographic and x-ray studies. Results show a wide range of mechanical properties are available from these heat-treatable, corrosion-resistant high-density alloys.


The rare earths Tb, Lu, and Y (0.1-0.3w/o) were added to U and U-alloys with Ni, Co, and Rh being the noncarbide carrier metals and their mechanical and physical properties were determined. Material containing Rh and Y additions exhibited the greatest increase in tensile strength.


Additions of up to 1.5%Ti improved the mechanical properties of U especially when cooled rapidly from the γ-phase. Rupture and yield strength increased uniformly with the Ti content and maximum elongation occurred with 0.4-0.6Ti content. U-5.5Ti alloy was studied extensively and results showed it to be relatively insensitive to quenching conditions.


The temperature dependence for Young's Modulus for U-1/2a/oNb and U-1/2a/oCr were measured dynamically up to 500°C. E decreased from - 19x10^11 dyne/cm² at 20°C to - 1.2x10^10 dyne/cm² at 500°C. Alloying elements did not significantly alter the behavior.
Theoretical results obtained from a model developed as a result of a strain energy-to-failure investigation of properties of U-7.5\%\text{Nb}-2.5\%\text{Zr} alloy agreed well with experimental results of a previous investigation. A relationship was established between strain rate and temperature over wide ranges, including a phase change for the alloy. A softening-point correlation was also established.

Data are presented on creep strength at 400°-550°, the effect of thermal cycling, tensile properties between 600°-700°C, thermal expansion, and thermal conductivity for a large range of U-alloys.

Addition of 0.1-1.5\%Ti to U improved its mechanical properties and corrosion resistance. Mechanical properties of the U-Ti alloy were very dependent upon the rate of cooling applied after heat treatment in the γ-temperature region. Increasing the amount of Ti increased strength, hardness, and corrosion resistance but decreased ductility and impact strength.

Study of the U-2\%\text{Mo}-2\%\text{Zr}-2\%\text{Nb}-0.5\%\text{Ti} alloy indicated that in as-forged and as-rolled conditions it possessed little or no ductility, but in the solution-treated and aged conditions possessed a measurable amount of ductility (1-3\% elongation).

High temperature tensile tests (up to 600°) and prolonged hot hardness tests (400°C) of U alloys were conducted for correlation of results with in-reactor behavior. The mechanism of flow and fracture is discussed in terms of temperature, structure, strain, and strain rate. Activation energy values for plastic flow are given. Resistance to identification appeared to be a function of solid solution strengthening and not of total alloy content.

Abnormal mechanical properties - nonlinear "elasticity", rubberlike amounts of "elastic" spring back, and large amounts of mechanical hysteresis - of U-7.5w/oNb-2.5w/oZr alloy were examined in uniaxial tension and compression with respect to stress, strain, and aging. Results indicated a stress-induced phase transformation in both the as-quenched and aged alloy. This is substantiated by volumetric, resistance, and microstructural changes in the alloy under stress.


The physical and mechanical properties of U-7.5w/oNb-2.5w/oZr alloy in the temperature range 150°-350°C are described. Results indicated mechanical strength increases with aging temperature and time within this range. Tensile yield strength after heat treatment for 1 hr at 150° was 92,000 psi, and after 4 hrs at 350°, 215,000 psi, with elongation for these strength levels 20% and 3.5% respectively. Electron diffraction data showed that an ordered structure is developed during aging at 150°C for 10⁴ min and it is believed this ordering is partly the cause for the increase in strength.


Summary of the mechanical properties of high-strength, γ-phase U-base alloys containing Nb, Nb and/or Zr. Data for room temperature and elevated temperature tensile properties, DFN hardness, Charpy impact strength, and tensile properties of TIG welds are included.


The U-7.5w/oNb-2.5w/oZr alloy was studied and the relationship of transformation characteristics to physical and mechanical properties was determined. Tensile yield strengths of 100,000-200,000 psi were obtained, with elongations of 12% to 4%, respectively, depending on heat treatment.

The influence of the γ grain size on the mechanical properties of quenched and tempered U-2Mo alloys is described. Experimental results indicate that refining the grain size from 500 to 600 μm when tempering at < 400°C slightly improved the alloy strength. Ductility was decreased at all aging temperatures and strength generally increased. Reducing the C content from 200 to 85 ppm led to a systematic increase of the reduction of area and a 15% increase of bend strength.


Wrought U-<1.5Ti alloys prepared from castings by hot rolling or hot extrusion were heat treated by quenching in H2O from the γ-region. The as-quenched material was ductile and strong. Aging at 400°C for 2 hrs produced a 30-50% increase in yield strength and increased ductility. The alloys did not appear to be susceptible to SCC in 100% relative humidity environments and appeared to have superior corrosion resistance to that of unalloyed U.


Procedure for melting, alloying, and casting of U-Mo, U-Mo-Nb, U-Mo-V, U-Mo-Zr, and U-Mo-Ti alloys on a laboratory scale was established. Maximum heat treatment hardness (463-639 VHN) was obtained by heating to 800°C for 1/2 hr, H2O quenching, and aging from 1/2 min to 2 hrs at 400°C-500°C for the U-1Mo and U-1Mo-2Ti alloys. Maximum as-cast tensile strength was 176 kpsi for the U-1Mo-2Nb and heat-treated tensile strength was 219 kpsi for U-1Mo-0.5Ti. Densities ranged from 17.7 g/cc (U-1Mo-2Ti) to 18.9 g/cc (U-1Mo).


General characteristics of U and some possible nonnuclear applications of U-alloys are surveyed. Metallurgy, general principles of alloying, and heat treatment of U are described. Response to heat treatment of various U-Mo, Nb, Fe, Cr, Si, Ti, Zr, V, Ni, Mn, Co, W, Ta alloys was determined by hardness measurements. Compression, tensile, bend, and impact properties as well as corrosion properties of the more promising alloys were determined. Results indicated that high-density, high-strength U-alloys can be developed successfully.

The effects of adding, together or separately, 1% W and 1% Ta to U-Mo-Nb alloys were determined. Addition of W and Ta did not appear to affect strength and ductility of heat treated U-alloys but did seem to have an important effect on corrosion, especially in the presence of Nb. Alloys containing small amounts of Cr and Ni were tested and results confirmed an increase in corrosion resistance.


Ternary, quaternary, or more complex U-base-Mo, Zr, Nb, V alloys formulated by varying the amount of and by removing one or two of the alloying elements in turn from the base alloy were studied. Each alloy was studied under three different heat treatments and the mechanical and corrosion properties of each were determined. Most exhibited the desired hardness and strength but were extremely brittle and notch-sensitive under tensile stresses.


The conditions under which soft U-Nb-Mo alloys with varying amounts of alloy addition can be obtained and also retained in the soft and ductile state and in the soft α- and γ-phases at room temperature are described. Mechanical properties were determined and the changes in properties of the alloy phases after decomposition or transformation by heat treatment or cold work were measured during and after treatment. Results indicated that the α-phase alloys are unstable and transform rapidly during deformation but γ-phase alloys do not decompose as readily but can be deformed more extensively.


The principal physical, chemical, and mechanical properties of both natural and depleted U are presented in a brief summary. The various techniques for processing U are described. Heat-treatable U-alloys with high mechanical properties are considered.


Tensile properties and hardness of U-O-14%Nb alloys were studied at room temperature and elevated temperature. With Nb additions < 5.5% the breakdown of the γ solid solution to unstable microstructure led to pronounced aging effects but with > 5.5% additions the γ-solid solution was retained to room temperature and showed enhanced properties up to 800°C.
U-Mo alloys are susceptible to a wide variety of heat treatments. Three groups of alloys exist in which hardening is due to: (1) solid solution of Mo in α-U, (2) formation of α + γ dispersions, (3) solid solutions of Mo in γ-U. Effects of irradiation are not clear.

Mechanical and physical properties of Mulberry, a γ-phase metastable U-7.5w/oNb-2.5w/oZr alloy, are described in detail. Tabular data figures are provided.

Mechanical properties, tensile properties, and fabrication of binary and ternary U-alloys were studied. Effects of quenching and aging on hardness of U-Mo alloys were investigated. Additions of 1-5% Al, Mo, Nb, Si, or Sn had little effect on the hardness. Effects of time and temperature on the tensile properties of solution treated and aged U-Mo alloys were also determined. Hot working properties of the U-Mo alloys previously reported are summarized.

Small additions of Mn, Ni, Ta, W, V, Hf or Cr to the polynary U-1-1/2Nb-1-1/2Zr-1-1/2Mo-1/2Ti alloy were found to have little influence on mechanical properties. Corrosion in boiling water is reported. Three alloys with higher Nb content (up to 7.4w/o) exhibited adequate ductility, impact energy, and corrosion resistance at the 200 ksi yield strength level.

Metallurgical properties and structures of 12 U-base alloys containing 0.13-4.5w/oMo, Cb, V, and Ti were determined for various heat treatments. In general, tensile ductility and impact energy were increased at constant tensile strength levels for extruded, solutionized, quenched and aged material compared to as-extruded material. Grain size was enlarged and chemical homogeneity improved by the heat treatment. Replacing Zr with V in the U-2Mo-2Cb-2Zr-0.5Ti alloy improved corrosion resistance in boiling H2O at atmospheric pressure.
The mechanical properties, deformation characteristics, and fracture behavior of γ-phase U-6w/oMa alloy were studied. Environment controlled elevated temperature tensile testing system and a cryogenic temperature tensile testing apparatus were used to determine the mechanical properties from 77°-798°K. Activation enthalpy and volume, and other strain rate parameters were calculated from the data obtained by strain rate cycling and stress relaxation measurements for the analysis of the deformation mechanisms. Metallographic and fractographic methods were used to characterize plastic deformation and fracture behavior. It was established that fracture mechanism is a function of testing temperature.

Preliminary experiments with two-phase heat treatments of a U-2.3w/oNb alloy indicated that improved mechanical properties can be achieved by age hardening and/or strain hardening. Young's modulus 20x10^6 psi, yield strength (0.2% offset) 120,000 psi and elongation 5% can be obtained.

Mechanical properties obtained by five different heat treatments of U-2%Mo alloy for use at temperatures from -198° to 100° are reported. Tensile strengths reached a maximum of > 200,000 psi at -30° but failed in a brittle manner. Impact strengths were generally low and decreased at temperatures < -30°C. Homogenization followed by furnace cooling increased ductility but decreased tensile and yield strengths.

The U-8w/oMo alloy was one of many materials considered in a program to develop a high strength, high density, low-temperature ductility alloy. Recommended areas for further study include fiber reinforcing the U-Mo alloy with W or W-alloy wire and alloying additions to the U-8w/oMo alloy.

A U-base alloy containing 1.5-6.0% Mo-0.1-1.0% OAl-0.2-1.0% OAl and Si in which either Al or Si is at least 0.1% with improved high temperature deformation was developed.


Method of producing high strength U alloys containing 93.5-96.5% U, 0.75-2.0% each of Mo, Zr, and Nb, and 0.25-0.50% Ti. In the "as solutionized" condition these alloys exhibit maximum ductility and impact resistance whereas maximum strength and hardness are obtained in the "as extruded" condition.
The influence of isothermal heat treatment at temperatures between 600°C and the eutectoid transformation temperature on grain size was investigated. Formation of columnar grains in U-45a/oCr alloy after quenching at 720°C and transforming isothermally between 635°C and 600°C was due to the difference between the surface and bulk nucleation rates.

Results of a brief program using metallographic and electron microscopic techniques in detecting microstructural differences in cast U of varied chemistry and casting history are described and evaluated. Evidence of differences in substructure and in fine-particle distribution and form between the various materials are reported. Micrographs illustrating the effectiveness of the techniques are included.

Calorimetric and x-ray spectrometer measurements were used to verify the presence of an order-disorder reaction in the U-Mo system. Results were in good agreement with order-disorder theory but a slight adjustment of previously accepted phase boundaries was necessary.

Experimental results showed that the addition of small amounts of C can lead to considerable refinement of the y-grain size of U-1.1Mo alloys. The mechanism for the C effect is discussed and methods of introducing C to the U-Mo alloy are proposed.

Grain refinement of the γ-phase of U and U alloys by addition of small amounts of low atomic number elements is described. γ-grain refinement by C was maximum at 500-800 ppm C for fast cooling rates; however, the maximum disappeared at slow cooling rates. Mechanism for grain refinement is discussed.


The metallographic characteristics of microstructures of U-0.2-2.0w/oMo, U-1.0-8.0w/oZr and up to 2w/oC additions to these alloys which were slowly cooled to room temperature with intervening stop-annealing treatments at <950°C are described. Distinction between the constituent phases in these alloys was made by replica techniques. Possible effects of the microstructural condition of the alloys during irradiation are discussed.


U-0.2-0.5Mo-0.5-0.7Nb and U-0.2-0.4Mo-1.0-2.0Zr alloys were investigated in order to improve the irradiation resistance of metallic U. Microstructures were characterized according to heat treatment, with optimum grain size obtained by continuous cooling. TTT diagrams, aging response, and isothermal sections of the U-Mo-Nb system are reported.


Jominy end-quench data for binary, ternary, and quaternary U-alloys with Mo, Cr, and Nb indicated grain size of the alloy specimens was comparable to predictions of equations based on additive and multiplicative models. Better correlations for the additive models were obtained.


Dilute U-alloys (up to 5a/oCr, Si, and Zr) can be used to reduce as-cast, fabricated, and θ-treated grain size (<60μm). The neutron absorption cross section and concentration required for each of alloying elements are sufficiently low to permit the use of natural U in thermal neutron reactors.

Study of microstructures in samples β-phase quenched and annealed at high α-phase temperatures supported a recrystallization mechanism for grain refinement. The effect of C content on the magnitude of grain refinement was small in the surface layers but large on the interior. Within the range 10-1000 ppm, grain refinement increased with increasing C content.


Transformation temperatures and grain sizes of dilute U-Cr alloys were studied as a function of cooling rate. Results indicated that an alloy which would transform from the β-phase to fine equiaxed α-grains could be developed by a non-orientation dependent mechanism, when cooled to rates comparable to those observed on air-cooling large bars.


An attempt to develop ternary alloys, with an element added to the U-3.6a/oMo alloy, having the same reactivity loss as the binary alloy and containing a high γ/α ratio when heat treated to give lamellar or "divorced" (α+γ) microstructures is reported. The U-Mo-Nb, U-Mo-Pc, U-Mo-AI, U-Mo-Ru, and U-Mo-Cr systems were investigated and the summary of the compositions, heat treatments, and phases detected are presented in tabular form. The U-3.6a/oMo-6.9a/oNb alloy was partially successful but the higher γ/α ratio did not persist at 550°C.


Short-range order in U-Nb solid solutions of equiatomic composition was studied by an x-ray diffuse scattering method. Short-range order parameters for three shells were measured and the static distortion parameters of the crystal lattice due to differences in the atomic volumes of the components were determined.


Optimum composition and heat treatment of U-0.3, 0.45, 0.6a/oCr alloys to yield maximum grain refinement were investigated. Optimum isothermal transformation temperature in the 0.45Cr alloy was 525°C and for the 0.6Cr alloy, 500°C. Increasing the Cr content from 0.3 to 0.6 decreased the temperature at which maximum grain refinement was achieved. Prior thermal history had no effect on grain size obtained by isothermal transformation.
Method of producing U-Mo-Nb alloy with grain structure refined to 50-100μm from 175-225μm by heat treating cast alloys (0.8-1.2% each of Mo and Nb) to 700°-900° and continuously cooling the alloy from this temperature at a rate of 100°-250°C/min.

Method of producing ternary U-alloys-U-0.1-0.3%Mo-0.6-0.8%Nb and U-0.2-0.4%Mo-1.5-2.2%Zr. The γ-phase of the alloy is heat treated at 900° and then continuously cooled at a rate of 5-60°C/min. The grain size after heat treatment is 20-100μm, depending on the cooling rate.

 Isothermal annealing characteristics of the eutectoidal decomposition of β-U-base dilute Al alloys were studied by optical and electron microscopy. The effects of Fe and C on the decomposition are described.

U-Ti alloy system differs from U-Nb and U-Mo alloys with regard to the nature and processes of formation of metastable phases in the following points: (1) the existence of a biphase region of Nb = γ, (2) the variation of the parameter does not follow Vegard's law, (3) the boundaries of the different phases do not correspond to the same contractions of the parameter, b. These differences could be due to the relative positions of Ti, Mo, and Nb in the periodic table or to different bonds between the U and Ti.

Characteristic needles of Cr were found following the β→α transformation in U-Cr alloys made from electrolytic pure U (EPU) but not in alloys made from nuclear pure U (NPU). Growth rate (G) and its dependence on undercooling and Cr content of the eutectoid decomposition product in EPU alloys varied from those established for the NPU alloys and was attributed to the difference in the mode of nucleation of Cr. It is suggested that the growth of α or another process not dependent on the interspacing between the needles is the growth governing factor.

The rates of nucleation and both lateral and longitudinal growth of martensite plates (needles) in α-α transformation in U-0.1, 0.16, and 0.21 w/o Cr alloys were determined as a function of temperature and activation energies were calculated. The lateral and longitudinal growth rates had identical values suggesting they are both controlled by the same process, the rate of single-atom movement.


Growth rates of pearlite, bainite, and martensite in U-Cr alloys were determined. For bainite, experimental results showed good agreement with the predictions of a model based on growth control by interface atom jumps. The bainite measurements, extrapolated into the pearlite range, showed good agreement with measured pearlite growth rates, and hence a similar growth mechanism was indicated. The growth-rate measurements in martensite were consistent with interface movement which is controlled by individual atom jumps. Martensite growth is believed to occur in two steps: a rate controlling step and a step in which some atoms cross the interface by a cooperative process.


Rates of nucleation and growth rates of α-phase U-0.45, 1.1, and 1.35 w/o Cr alloys were determined at various temperatures and proof that the α-α transformation is controlled by nucleation and diffusional growth was obtained. The experimental results were in agreement with the classical theories that nucleation is controlled by the law of nuclei formation and the growth is controlled by bidimensional nuclei formation. Cr retarded both the rates of nucleation and growth.


Nucleation and growth rates, measured for several U-Cr alloys undergoing transformation, were found to be in agreement with the Volmer-Turnbull theories for nucleation and growth. Kinetics of γ decomposition in a U-10.8 w/o Mo alloy were determined by x-ray measurements. Two C curves were detected; at 550° γ-α+γ' proceeded directly, while at 500°C and below α+γ' is preceded by the appearance of α+γ.


Metastable phases of U-4.8 and 9.5 w/o Ti alloys were studied by micrography, x-ray diffraction, and both hardness and microhardness measurements and compared with the behavior of U-Mo and U-Nb alloys. The metastable phases in the U-Ti alloy appeared to behave quite differently from those in U-Mo and U-Nb alloys. This may be due to the presence of the intermetallic U₂Ti.

Transformation characteristics of U-1w/oMo-1w/oNb-1w/oZr and U-2w/oMo-2w/oNb-2w/oZr alloys were determined by dilatometry and thermal analysis. The alloy richer in U decomposed rapidly during cooling: the eutectoid temperature was 661° and the Ms temperature 495°. The more highly alloyed material had a eutectoid temperature of 642°, displayed a shear transformation, γ→γ*, at γ200, and the Ms for γ→α was 315°C. Possibilities of thermomechanical treatment for improved toughness and strength of these alloys are discussed.


High temperature x-ray diffraction of U-10-20a/oNb alloys showed a cubic γ-phase was present with no evidence pointing to a stable γ'-tetragonal phase.


Martensite and bainite transformations in U and U-base alloys are reviewed and results of recent kinetic and metallographic studies presented. In U-alloys the martensite change occurred isothermally by slow growth and had C-curve kinetics. Addition of 1.0w/oCr to U decreased Ms from 600° to 200°C. Driving force above Ms was found to be ~500cal/mole and the reaction above Ms was similar to the bainite reaction in steels and is thought to involve both lattice shear and diffusion of Cr in the β-phase.


Isothermal β→α phase transformation of U-1%Cr alloy after quenching from 700°-740°C were studied by resistivity measurements, and by metallographic and SEM observations. Results confirmed earlier conclusions that the TTT diagram comprises three C-curves and the activation energies are ~70, 45, and 22 kcal/mole respectively. α-U with a dispersion of Cr particles was produced by reaction in C1 and heavily dislocated α-U in C2.


The experimental work on the β→α and γ→α phase changes in U and dilute U-alloys is reviewed. The β→α phase change may take place by normal diffusion controlled nucleation and growth rate or by a low temperature mode in which the growth of α-phase takes place slowly and isothermally. The γ→α phase change may take place by a diffusion-controlled nucleation and growth mechanism, and there appears to be at least three other modes. In dilute Mo alloys an acicular product forms but with increasing Mo content and other alloys a banded structure occurs. Another product in some ways similar to both a Widmanstätten pattern and a banded structure occurs and some work suggests a phase change in the β temperature range.

TTT curves for U-1.0%Mo, U-1.0%Nb, U-0.5%Mo-0.5%Nb, and U-0.2%M0-0.7%Nb alloys are presented and discussed. Transformations that occurred at various temperatures are summarized and the morphology of the precipitated phases are discussed.


TTT curves for U-2w/oMo and U-2w/oNb alloys are presented and discussed together with different transformations which occur at various temperatures.


Results of the metallographic and x-ray diffraction studies on metastable phases produced in γ-quenched U alloys containing up to 10a/oRu are reported. The effect of alloy content on the distortion of the orthorhombic structure, microstructure, and hardness was investigated.


Theories of martensite crystallography are analyzed using the 8→a and γ→a transformations in U and U-5a/oMo. Insufficient experimental data exist for any conclusions to be drawn about 8→a transformations. For some observed cases of the γ→a transformations, no real solutions of the theories are possible, demonstrating inadequacies of the theories. Preliminary account of a new theory of transformation shears is given.


TTT behavior of γ-U-7.5w/o Nb-2.5w/oZr alloy was determined for the temperature range 200° - 800° and a TTT diagram consisting of two overlapping C-curves was determined by metallography, microhardness, and x-ray diffraction techniques. The critical temperature was determined to be 650°C, above which the bcc γ-phase was stable. Upon H2O quenching to room temperature, a transition γ→γ' phase was formed in which the body-centered atoms were slightly shifted. Aging transformed this phase to γ" (tetragonal) and further aging produced γ" which is a monoclinic crystal structure with hardness of 250 to >6000PH.

Resistivity, metallographic, and microhardness techniques were used to study the 8-α transformation of U-0.5α/oCr alloy at sub-critical temperature. Results indicated that the TTT diagram consists of three C-curves: (1) highest-temperatures (650°-530°C) associated with diffusion-controlled transformation, (2) intermediate temperature (530°-400°C) involving simultaneous lattice shear and short-range diffusion, (3) low-temperature as applied to martensitic transformation by a shear mode.


X-ray diffraction and metallography were used to study the isothermal decomposition of the γ solid solution of U-9.5w/oNb alloy at 450°-600°C. The decomposition proceeded in two steps: (1) a discontinuous precipitation of a lamellar two-phase aggregate compound of a solid solution and a metastable γ-phase, having a constant composition at given temperature, (2) an equilibrium (α+γ) phase mixture after longer annealing. A modification of the original lamellar microstructure took place during the process.


Up to ~2α/oTi additions were very effective in increasing the strength of martensitic U. Lattice parameter changes with composition were continuous with a₀ increasing and c₀ decreasing with increasing solute. X-ray diffraction line broadening indicated non-uniform microstresses were present. Volume strain during γ-α transformation at Mₐ decreased at ~2α/oTi; it is suggested hydrostatic pressure during transformation decreased to below the yield stress at this composition. Both tangential stress and hydrostatic stress were sufficient to plastically deform the specimens.


Results of γ-phase isothermal transformation of U-Zr-Nb and U-Nb-Mo alloys (containing <1α/o components other than U) at temperatures >500°C indicated that in the upper range of the S-curves transformation was governed in the early stages by supercooling, and in the later stages by the difference in composition between the parent and forming phases. In the U-Zr-Nb alloy disintegration proceeded homogeneously, but in the U-Nb-Mo alloy, heterogeneously. Attainment of the equilibrium composition of the forming γ and δ-phases does not generally mean the cessation of volume changes (to within 5-10⁻⁸%/hr).

Dilatometry, metallography, hardness, and x-ray diffraction were used to study the $\gamma$-$\alpha$ transformation in U-0.25$\alpha$/Cr-0.5$\alpha$/Mo alloy. A TTT diagram consisting of two C-curves was developed. The upper curve was predominantly associated with a diffusion-controlled transformation but testing results suggest a subdivision of this upper region at 480°-510°. The intermediate mechanism of transformation was of a bainitic type and extended down to -425°C. The low-temperature C-curve appeared to involve a shear-type transformation.


The $\gamma$-phase of U-10$\%$Ti, Nb, Zr or Mo alloys is metastable under critical cooling conditions and may transform by a shear mechanism to form a characteristic banded structure or by a nucleation and growth process to equiaxed grains. Metallographic appearance of these structures differ, but x-ray patterns are similar, termed "distorted" because of a contraction of the $b$ parameter. Formation of the metastable structures depends on composition, cooling rate, the effect of the solute on the incubation period, and decomposition rate of the $\gamma$-phase.


Three metastable phases are encountered when quenching U-Ti from the $\gamma$-phase: (1) $\alpha_3$ is formed by shear transformation at 10$\%$Ti, (2) $\gamma_7$ exhibiting some degree of anisotropy at 15$\%$Ti, (3) $\alpha_4$, a distorted $\alpha$ structure formed by nucleation and growth on slower cooling. $\gamma$-phase can be retained in 20$\%$Ti alloy. Hardness changes along end quenched rods are attributed to precipitation of U$_2$Ti from the supersaturated metastable phases.


The orientation relationship between the $\gamma$-phase of U and $\alpha$-like phase resulting from its direct transformation was determined for U-50$\%$Zr alloys. Results showed it to be similar to that reported for the transformation of the bcc structure to the hexagonal close-packed structure in Zr. Relationships of the metastable $\alpha'$ structure to $\alpha$-U is discussed. It is shown how a banded microstructure can develop by the transformation of $\gamma$ along all crystallographically equivalent paths so that each band is twin-related to adjacent bands.

U-alloys containing \( 0.9-4.17 \text{a/oMo} \) were cooled at various rates from the \( \gamma \)-phase field and studied by an end-quenching technique. The alloy structure was examined by metallography, x-ray diffraction, and hardness measurements. Structure was found to be dependent on the cooling rate and the original \( \gamma \)-grain size.


Rods of U-2.5-50a/oZr alloys were end-quenched and the phases formed, their distribution and hardness were studied as a function of composition and cooling rate. It was found that in alloys containing 10-50a/oZr, the \( \alpha \)-phase played a large part in the decomposition but in those alloys containing 10-30a/oZr the \( \alpha \)-phase was unstable at room temperature, and in the more dilute alloys there was no evidence of its appearance.


Metallographic, x-ray, and hardness measurements were used to study transformation of U-Mo (up to 25a/oMo) alloys at 450°-550°. In 5, 10 and 15a/o Mo alloys, transformation to equilibrium \( \alpha + \gamma' \) increased hardness whereas transformation of the 25 and 30a/o Mo alloys produced only minor hardness changes. Two metallographic forms of \( \gamma' \) were observed in U-25a/oMo, a banded appearance at 450° and an acicular form at 550°C.


U-1.0-10a/oRu alloys were cooled from the \( \gamma \)-phase and the transformations studied by an end-quenching technique. At the quenched ends a monoclinic \( \alpha \)-type structure, \( \alpha'' \), was found and beyond the quenched ends the decomposition is dominated by the rapid diffusion of Ru-U,Ru. Appendix on the grain refinement of the \( \alpha \)-phase produced from isothermal transformation of U-0.0.25-1.0a/oRu alloys is included.


Study of the shear transformations in the \( \gamma \)-phase U-6.0-12.5a/oMo alloys showed two shear transformations occurred successfully over most of the composition range; enthalpies were measured as a function of composition and cooling rate. The dominant factor appeared to be the changes in the degree of order produced by the change in cooling rates.

The kinetics of the isothermal decomposition of $\gamma$-U-2.5w/oMo alloy at 390°-670°C was investigated using dilatometry, metallography, hardness measurements, and x-ray diffraction.


X-ray patterns were analyzed and results suggest that the decomposition of the $\gamma$ solid solution of U-Mo alloys takes place by the formation of ordered $U_2Mo$ regions, coherently bound to the initial $\gamma$ solid solution on the (001) planes. In the U-Nb and U-Zr-Nb alloys, the $\gamma$ solid solution decomposes homogeneously at low temperature and low Nb concentrations due to the formation of highly dispersed $\alpha$-U precipitates, and heterogeneously at high temperatures and high Nb concentrations due to the limited number of centers and proceeds as diffusion continues.


The isothermal transformation behavior of $\gamma$-U-13a/oNb alloy was determined for the temperature range 23°-640°C. A TTT diagram was constructed from hardness, x-ray diffraction, and both optical and electron micrographic data. Two overlapping C-curves were disclosed in the diagram, the upper one corresponding to $\alpha$-plus a Nb-deficient $\gamma$-phase which gradually enriches in Nb to form the equilibrium $\gamma_2$ structure and the lower one being a general precipitation of $\alpha$-phase on dislocations.


The occurrence and generation of transition phases in U-Nb alloys are discussed and the transition phases and unit cell parameters as a function of composition and temperature are presented. Three distinct phases, $\alpha$'(orthorhombic), $\alpha$' (monoclinic) and $\gamma$' (tetragonal), occurred as an intermediate in the direct $\gamma$-$\alpha$ transformation. An approximated transition phase diagram and a model for the consecutiveness of the transformation are also presented.


Time-temperature-composition dependence of crystal structure and microstructure under nonequilibrium cooling conditions for U-rich U-Re alloys was studied. Sluggish reactions of the stable phases led to nonequilibrium quantities of the equilibrium $\alpha$, $\delta$, $\gamma$, and $\delta$ phases. The $\gamma$-$\alpha$ transformation was found to proceed by an isothermal martensitic reaction or by sluggish nucleation and growth. Under certain conditions, $\delta$-$\alpha$ transformation was direct, passing through the intermediate $\delta$ structure, but was accompanied by a number of metastable transition states. Compositional limits within which stable and metastable phases form and the effect of cooling rate and temperature on producing alternative structures were determined.

An unusual, internal, athermal, and reversible strain behavior was observed in U-6-21a/oNb alloys (maximum at 15a/oNb) in the temperature range -200° to +400°. Dimensional change per temperature increment varies as a function of temperature, composition, and cooling rate from the γ-region. Conditions necessary for subsequent temperature-induced shape change are: (1) sample must be cooled rapidly from the γ-region, (2) as-quenched shape must be subjected to a plastic deformation at <425°C.


General properties and characteristic behavior of heat treated U-Mo alloys were investigated. Specimens were made by vacuum induction melting and rolled at 600° and 900°. Phase changes by quenching from various high temperature phases and the effect of quenching rate from the γ-phase were studied. Mₐ was determined for U-1.3a/oMo. Thermal cycling tests on U-0.5-9.0%Mo rolled rods indicated that greater improvement was obtained by γ-quenching followed by α-annealing. Oxidation in air at 300°, 400°, and 500°C showed the rate constant decreased with increasing Mo content in the lower Mo range but increased as Mo content became higher.


Kinetics of the decomposition of γ-solid solution in ternary U-Zr-Nb alloys and in binary U-20a/oNb alloys were studied by dilatometry, and TTT diagrams were constructed. Resulting C-shaped curves consisting of two branches indicated that the transformation occurred by different mechanisms at high and low temperatures.


8-α transformation in U-1a/oPt alloy, at 0°-600°, measured by electrical resistivity suggested three modes of transformation: (1) at high temperatures nucleation-and-growth process occurs and is consistent with a diffusion-controlled process, (2) at intermediate temperatures the reaction is consistent with a bainite process, (3) at low temperatures, <100°C, the reaction is characterized by a shear mechanism. Measurements were unable to separate the high temperature 8-α transformation in dilute U-Pt alloys.
Kinetics of the $\gamma'\gamma$ transformation in U-16 wt% Mo alloys were studied by metallography and electrical resistance measurements. A nonequilibrium $\alpha$-phase appeared in the transformation anneal, reached a maximum volume fraction when $\gamma'$ emerged, and in time disappeared. At 550°C transformation to $\gamma'$ first appeared in 48-72 hrs, but at 500°C it took 120 hrs.

The kinetics of formation and disappearance of the transient $\alpha$-phase during the $\gamma$-to ($\alpha'$, ordered bct) $\gamma'$ transformation in U-16 wt% Mo alloys at 400-500°C were studied using quantitative metallography and electrical resistivity measurements. It was shown that the $\alpha'$-phase is not an equilibrium phase at any temperature but that it is easy to nucleate from the $\gamma$-phase and it contributes to a reduction in the free energy of the system. A final decrease in free energy occurs when $\gamma'$ forms and occupies the whole sample.

A nomenclature system devised for U-Mo alloys is described. It is believed to be sufficiently general to cover equilibrium and metastable phases existing in all U-systems showing a solubility in the bcc $\gamma$-phase. The proposed terminology is presented in tabular form and illustrated by micrographs.

Binary, ternary, and quaternary alloys of U with Mo, Cr, Nb, Fe, and Al were end-quenched from 720°-800°C using a Jominy rig. The grain refinement produced depended on the arithmetic sum of the effect on grain size which the alloying elements separately produced. Binary U-Cr alloys transformed by a shear process but U-Nb and U-Nb-Cr alloys gave a fine grain size when air cooled.

Kinetics of isothermal $\beta$-$\alpha'$ martensitic transformation in a U-0.5 wt% Mo alloy was investigated. Specimens were quenched from the $\beta$-region at 690°C and held isothermally at temperatures from 120°-196°C. The percentage of the $\alpha$-phase formed was determined and the half-thickness, $c''$, and radius "r", of plates were calculated by quantitative metallographic methods. It was found that the activation energy for nucleation depends linearly on temperature and that the nucleation rate is a parabolic function of temperature.

The kinetics of transformation in U-0.45w/oMo alloy at different cooling rates, 2°C/sec-80°C/sec, from the β and β+γ regions were studied. The changes in microstructure with the changes in temperature in the β and β+γ regions and with cooling rate are presented. A change in the mechanism of transformation as a function of the cooling rate was observed in all specimens. Contrary to previous investigations, lowering the overheating temperature did not accelerate transformation.


The effect of the degree of superheating in the γ and β temperature regions on the kinetics of isothermal β-α' transformation in U-0.45% and 0.6%Mo alloys was investigated. At room temperature the β-α' transformation depended on time which indicated that thermal activation affects the kinetics of the process. A modified Arrhenius equation, \( x=1-\exp\left(-k\cdot t^n\right) \), where \( t \) is time and \( k \) and \( n \) are constants, was used to analyze the kinetics of transformation. The calculated value of parameter, \( n=2+3 \), concurred with the mode of growth of the martensitic phase experimentally established. There are indications that the kinetics of β-α' transformation changes at the transition from the upper to the lower β-solid solution region.


A selected area electron diffraction technique was used to study the isothermal transformation of γ in U-1.6w/oMo alloy. Results showed this technique to be more sensitive to detection of γ'-phase, formed as discrete particles in the γ-matrix, than the x-ray method previously used.


The isothermal transformation of γ in U-1.6w/oMo alloy at 500°-640°C was studied using ordinary light, electron microscopy, and x-ray diffraction. The TTT diagram for the transformation of γ conformed to normal C curves representing beginning and end of the formation of α and γ'.


A simple automatic, high-temperature high-vacuum thermal analysis apparatus based on the differential method is described. The phase transformations of a series of U-Mo, U-Nb and U-Zr alloys were determined using samples of a few grams heated and cooled over a range of 1000°C in a vacuum of 10⁻⁵ torr. The effects of the alloying additions on the γ-phase stability of the U under low rates of thermal changes were recorded, Mo>Nb>Zr.

Isothermal TTT characteristics for γ-phase binary and ternary U-alloys containing Mo, Nb, and/or Zr are presented. Hardness and microstructural changes are described and correlated with time as determined by resistivity measurements, to initiate transformation at a given temperature.


Electrical resistivity of U-4.9w/oCr eutectic was determined as a solid from room temperature to its melting point, 870°±3° and as a liquid from its melting point to 1000°C. Measurements were obtained in vacuum by use of a standard four-probe method with current reversal. Comparison of the eutectic with pure U showed a reduction in the temperature of the phase transformations of the alloys and in the magnitude of the drop in resistance.


Part I-TTT diagrams using metallographic, dilatometric, hardness, and x-ray techniques are presented and transformation kinetics are described for U-10%-Mo, U-8%-Mo-1/4%Ti, U-8%Mo-1%Ti, and U-24%Mo-2%Nb-2%Zr-1/2%Ti. Part II-Metallographic techniques suitable for the identification of phases, intermetallic compounds, and nonmetallic constituents in these alloys were developed. Techniques for U-base alloys were perfected for nearly all conditions of heat treatment. Certain low temperature transformation structures and the identification of nonmetallic inclusions were not completely resolved.


The high and low temperature characteristics of binary U-8Mo and U-10Mo and ternary U-8Mo-0.5Ti and U-8Mo-1Ti alloys are described. TTT diagrams based on metallographic, dilatometric, microhardness, and x-ray diffraction data are presented. Decomposition of the bcc γ-phase at high temperature was a result of either a cellular mechanism or a combination of cellular Widmanstätten mechanisms at low temperature by a different but unrevealed mechanism and was initiated by precipitation of γ" homogeneously throughout the γ-matrix.


Comments on the work reported of Ivanov and Virgiliev (See-Entry No.287) on the x-ray diffraction measurements of the γ-transformation mechanism in U-Mo alloys. It is proposed that the transformation occurs by the formation of γ"-nuclei with some small degree of ordering. These grow as plates and continue to order simultaneously.

Type II deformation twins having irrational twin planes, but rational twin directions predominant in metals. Crystallographic restrictions imposed by Type II twinning on the martensite theories are considered and extended to the case of U-5%Mo alloys to include the possibility of Type II twins arising from two-fold rotation axes of the parent γ structure.


The properties of U-alloys containing <0.1w/oCr, Mo, and Nb were investigated to study structural refinements and to find a satisfactory combination of random structure, fine grain size, and high temperature properties which are essential for reactor fuel elements. TTT curves for U and the U-alloys are presented. Additions of Cr and Mo in the ternary alloys, isothermally treated at 500°C, refined the grain size and were thermally stable but the Nb additions were so small (0.04-0.07w/o) no changes were observed.


Effects of ternary and quaternary alloying on the stability and properties of U-Zr, U-Nb, and U-Mo γ-phase alloys are reviewed. The effects of ternary and quaternary additions of Cr, Mo,Cb,Ru,V, and Zr on transformation kinetics, transformation temperature, hot hardness, and corrosion resistance were also investigated.


Mode of formation of metastable phases in the U-Nb system (1.0-18.15a/oNb), were studied. Composition limits of α′, α″, and γ phases and changes in parameters a, b, c, and γ(ab) were determined. A study of the effect of cooling rate showed its effect on the mode of formation is similar to that observed in the U-Mo system. Reasonably accurate values of the α′/α″ boundary composition for various binary U-alloys based on atomic size may be calculated.


Composition limits of metastable α′- and α″-phases, changes in parameters a, b, c, angle γ(ab) of α-U lattice with increasing Mo additions (0.07-11.39%), and effect of cooling rates in producing alternative structures were determined. Increasing Mo additions stiffened the U lattice, making it more resistant to shear but increasing the cooling rate favored shear and carried the transformation toward completion.

The effects of various heat treatments on the kinetics of the 8-α phase transformation in U and U-0.5-0.85w/oCr alloys were examined using an electrical resistivity technique. Transformation at temperatures near the equilibrium transition temperature was reduced by increasing the 8-quenching temperature. This retardation was due to increased 8 grain size in the alloys, but in the pure U other factors are involved. At temperatures in the martensitic and bainitic region transformation was accelerated with increasing temperature because of the removal of the grain boundaries.


X-ray diffraction study of the decomposition of U-7.5w/oNb-2.5w/oZr indicated atom displacement ordering in as-quenched tetragonal phase (γ₀). An intermediate stage in γ to γ₀ transition was found; this γs state is reflective of the high temperature γ-phase and is spoken of as the short range atom displacement ordering counterpart to the γ₀ phase.


Two metastable transition phases were revealed when γ-quenched and aged U-7.5w/oNb-2.5w/oZr alloys were studied by single crystal x-ray diffraction. The first transition structure, γs, is cubic with space grouping of 143m and may be described as eight unit cells of the γ-structure in which the body-centered atoms have moved in correlated <100> directions. Aging at 150° or 350°C produced the second transition structure, γ₀, which is tetragonal with space grouping of P4/mnm. No evidence was found to suggest significant chemical order in either transition structure.


Metastable phases occurring in the decomposition of bcc γ-phase in U-90, U-Nb and U-Nb-Zr systems to equilibrium phases are described. Degree of distortion decreased with increasing solute concentration in alloys quenched from the γ field. γs appearing in high solute as-quenched crystals, had atoms shifted by displacement waves over short distances only; for decreased solute or prolonged aging, monoclinic a' was obtained. Finally, orthorhombic a is described which is most closely related to equilibrium a.
319. Zukas, E. G., "Transformation Kinetics and Mechanical Properties of Uranium-0.5\% Cottium-0.5\% Molybdenum Alloy," Trans. ASM, 52 (1960), 797-811.

Isothermal transformation studies were made on the U-0.5\%Cb-0.5\%Mo alloy at 950°C, the high temperature γ-phase field and at 675°C, the (B+γ) phase field to increase the amount of retained δ-phase. Transformation boundaries were determined by hardness, density, metallography, and x-ray diffraction. Tensile and impact properties were determined from -196°C to 100°C after four heat treatments which were chosen on the basis of isothermal kinetics studies. The isothermal transformation diagrams are presented.


Process of achieving an unusual, thermally recoverable and reversible pseudo-plastic strain behavior in U-Nb alloys (5.0-25.0\%Nb) which comprises heating the alloy to ~850°C, H2O quenching from the γ-region at room temperature, and plastically deforming it at a temperature <450°C.


Alloy specimens as U-4-6\%Mo, U-4-10\%Nb, U-2-7\%Re, and other alloys, useful as thermal sensors, change their shape reversibly when deformed or strained at low temperature and heated above a transition temperature to induce a metastable stressed high-temperature structure. A stress transformation to a martensitic structure occurred on cooling, causing the change in shape. Shape memory on cycling through the temperature -196°C and +100°C is described using U-5\%Mo as an example.


Binary U-alloys containing 2-7\%Mo, 3-10\%Nb, or 2-7\%Re and other alloys were provided with good heat recoverable properties by preheating, cooling rapidly to prevent α-phase or eutectoid precipitation, and deforming at the M₁ transformation temperature. Original shape was recovered by reheating above a critical temperature. Shape memory is described for U-5\%Mo deformed at room temperature, heated to 150°-200°C, and subsequently cooled to room temperature again.

Precipitation


Strengthening mechanisms in U-<1.0\%Ti alloys were determined and studied. Results showed some gain in mechanical properties in the as-cast alloy, higher strength and elongation in the γ-quinched alloy, and increase in hardness and yield strength and decrease in ductility with subsequent aging of γ-quinched alloy. Maximum hardness (S110PH) and maximum strength (145.2 ksi) were produced after aging for 72hrs at 400°C. Continued aging produced growth of precipitates at grain boundaries.
Hardness and microstructure of U-8.5-50a/oTi alloys were correlated with heat treatment. A direct quench of γ-U-Ti solid solution resulted in the formation of a hard martensitic phase in low-Ti alloys. Subsequent tempering first increased the hardness by the precipitation of U₂Ti but further precipitation with tempering time resulted in softening. Isothermal transformation of U-8.5a/oTi at 400°-500°C gave high-hardness levels. The overall response to heat treatment was dependent upon the phases present during solution treatment. Results are interpreted in terms of the phase diagram.

The mechanism of transformation of the quenched metastable phase (α') in a U-2Mo alloy was studied. Isothermal and anisothermal tempering processes were used, including metallography, x-ray diffraction, hardness and dilatometry on quenched samples either during or after tempering. Results indicated the α'-phase decomposed in two stages: (1) formation of G. P. zones, (2) formation of peralitic structure (α+γ') the matrix impoverished in Mo and hardness reduced. Final stage is analogous to that of normalized or annealed steels.

Tensile test data correlated with metallographic and x-ray studies for U-11, 15, 17, 19a/oNb alloys in the γ-quenched and aged conditions are presented. Aging for 1hr at temperatures <300°C increased the strength of the alloys with no loss in ductility. The alloys are heat treatable and corrosion resistant, have high density, and show promise as an economic engineering material.

TEM of thin foils was used to investigate precipitates formed when dilute alloys of U-Al and U-Fe-Al were γ-quenched and α-annealed. In both alloys intergranular precipitates were identified as UA1₂ and in the ternary alloy large precipitates of U₆Fe formed rapidly at grain boundaries. Evidence for the orientation relationships between these phases is presented and the atomic rearrangements involved in their formation are discussed.

Three stages were observed in the annealing of γ-quenched u-5a/oMo alloy: (1) age-hardening in the supersaturated α'-phase, (2) over-aging with recrystallization and discontinuous precipitation of nonequilibrium γ, (3) a second recrystallization with re-precipitation to form a pearlite structure. Proposed explanations to account for these reactions are speculative and further work is necessary.

A compound wheel composed of a Be hub and a U-8%Mo alloy rim increased both the angular momentum and the gyro sensitivity of the wheel. Aging heat treatment provided the U-Mo alloy with the physical properties desired in a gyro wheel rim material. A wheel design for operation at 36,000 rpm was defined.


Isothermal decomposition of the γ-phase in U-12, 20, 25a/oTi alloys was studied by single-crystal and polycrystalline x-ray methods, metallographic observations, microhardness measurements, and limited electrical-resistivity measurements. Single-crystal x-ray studies showed the U-2STi alloy decomposed during quenching from 1475° to room temperature into a coherent hexagonal phase. In polycrystalline specimens there was continuous precipitation within the grains and a grain-boundary-nucleated discontinuous reaction to α+U2Ti.


Optical microscopy, quantitative metallography, and hardness measurements were used to study the effect of fabrication and heat treatment variables on the structure of U-3.96w/oSi alloy. Results showed an optimum temperature below the peritectoid temperature where maximum transformation to U3Si occurred in a given period of time. The time required is affected by the size of the primary U3Si2 particles; at <12μm complete transformation can be achieved in four hours.

Impurity Effects


The effect of H, particularly at low concentrations (0-200 ppm) and in different environments (vacuum, air, H2O), on both the dynamic and static tensile properties of U was studied. At all concentrations the H embrittled the U and caused intergranular fracture. The ductility and tensile strength of the hydrided U decreased in the order vacuum > air > H2O. Adverse mechanical effects of H were eliminated at temperatures >100°C. Electrolytic etch treatment revealed no preferential hydriding at the grain boundaries.
A series of U-1.8wt%Nb and U-0.75wt%Ti alloys with varying amounts of C, O, and N were prepared. The final chemical compositions of the alloys were related to their heat-treated mechanical properties. It was found that C was detrimental to ductility in both alloys but did not affect the yield or ultimate strengths in the U-Ti alloy which exhibited a "gettering" action in which some C was removed by Ti. Variations in the Ti content affected the yield and ultimate strengths.

H can be associated with U either in the form of a hydride or dissolved in the metal. Solubility of H in U depends on the allotropic forms of the metal and is greater in molten than solid U. Two types of hydrides, both UH₃, have been identified:
(1) α-type, stable at low temperature but completely transformed to β at temperatures >140°C, (2) β-type. Effects of H on the mechanical properties of U are included.

Metallographic study of the nucleation and propagation of tensile fracture in α-range of commercially pure U was made. Crack nucleus appeared to be associated with the inclusions present. The effect of prior annealing in the γ-range on the size-distribution of these inclusions was studied semi-quantitatively.

A literature review of H embrittlement of U was made. The probable causes of the severe impairment of the ductility at room temperature are threshold concentrations of 0.2-0.5 ppm H present as β-UH₃. The morphology of UH₃ and the microcracks associated with this phase also influence the degree of embrittlement. The reaction of U with H₂O vapor in the annealing media is the source of H in heat-treated U.

Occurrence and identity of inclusions in U due to the presence of C, N, H, Fe, Si, and Al are discussed. A complex of O, C, and N can exist in normal-purity U because of the isomorphous nature of the individual compounds UO₂, UC, and UN. The metallic impurity elements Fe, Si, and Al all form U compounds that precipitate on cooling.
Diffusion


The intermetallic diffusion characteristics of U with pure metals as Zr, Mo, Ti, and Nb and with the alloys U-30a/oNb and U-30a/oMo were measured. Study of the concentration-penetration curves made it possible to calculate the diffusion coefficients and to draw up the equilibrium diagram in the polyphase field in the U-Zr system. Results were compared by micrographic examination, microhardness measurements, and autoradiography. Diffusion mechanisms were studied by the Kirkendall effect and by calculating the Darken coefficients in the U-Zr and U-Mo systems.


The existence of a Kirkendall effect in bcc γ-phase U-Zr alloys is in agreement with recent results and supports an argument in favor of a mechanism invoking lattice vacancies. It has been shown that small concentrations of C can totally inhibit diffusion which leads to a weak bond, at the time of sheathing, between the metal and its sheath. Additions of Zr can fix the C present, and "normal" diffusion does not occur.


A diffusion couple technique to obtain meaningful data regarding the diffusion kinetics in the Al-Ni-U system was developed and standardized. The technique involved hot pressing under a dynamic vacuum and subsequent isothermal soaking treatments within evacuated quartz tubes for 10-7000hrs at 650°-950°F. The Ni barrier bond formed was metallographically examined and results are presented.


Intermetallic diffusion in U-Al alloys was investigated in the temperature range 300°-600°C using "sandwich"-type couples and employing metallographic and autoradiographic techniques. The growth kinetics of various phases and their relative distribution in the diffusion zone were found to vary with different temperatures. Kinetics of layer growth are discussed in relation to Kidson's analysis. Structural instability of the diffusion couples was attributed to volume changes occurring in the zone as the relative distribution of various phases changed during diffusion.
Diffusion measurements were made in the bcc solid solution of U-Nb alloys at all compositions using incremental diffusion couples at composition intervals of 10 a/o. Diffusion coefficients were determined by the Matano method from concentration gradients obtained with the electron-probe microanalyzer. The activation energy for interdiffusion showed three distinct areas that seemed to correspond to regions of different atomic order. The frequency factor (D₀) and the activation energy (Q) for interdiffusion obey the relationship \( \log D₀ = aQ + b \).

Interdiffusion and intrinsic diffusion coefficients using pure element couples were determined with an electron microbeam probe. Due to the large variance of the diffusion coefficient with composition, reliable data were obtained only in the range of 0-10 a/o Nb. A large Kirkendall effect and considerable porosity in the U-rich areas of the specimen are reported, suggesting that the true diffusion coefficients are somewhat larger.

Diffusion coefficients for Ce in molten U were measured for the temperature range 1170°-1480°. The equation relating the diffusion coefficient and temperature is \( D = 0.02e^{-15\text{Kcal}/RT} \) and \( D \) at 1200° is \( 10^{-4}\text{cm}^2/\text{sec} \). Solubility of Ce varied from 1-2% by wt at 1140°-1345°C. Experimental error involved in the measurements at high temperature is discussed.

Solution potentials for U and U-2%Mo alloys measured in 0.1N NaCl solution with a Ag:AgCl reference cell were 0.748 and 0.660V respectively. MIL-P-8585 ZrCrO3-lacquer and ZrCrO3-epoxy primers did not retard galvanic corrosion when used as coatings for galvanic couplings of U and U-alloys with steel and W alloys.


Galvanic corrosion behavior, in ocean H2O and HCl, was determined by measuring the gravimetric corrosion rates of galvanically coupled specimens and by constructing a galvanic series for each environment from electrode potential measurements. Gravimetric-galvanic test results showed the following order of increasing nobility in 0.1N HCl at 70°: 7178AI, depleted U, U-4.5Nb, 4540 steel, U-6Nb, U-8Nb, U-7.5Nb-2.5Zr, U-10Mo, Ti-6Al-4V, 304 stainless steel. In 0.1N HCl and ocean H2O at 25°C the galvanic series, based on electrode potential, are the same as those based on gravimetric-galvanic corrosion tests except for the Ti-6Al-4V alloy.


Th, U, U-10w/oMo alloy, bare and organic - or metal coated coupons, and composite assemblies, joined by soldering, welding, or machine screws were evaluated for corrosion behavior during storage for 1 yr. Material was uncovered or in loosely covered metal containers under ambient temperatures in a waterfront building, a rooftop, and an unheated warehouse. In no case was severe corrosion encountered and the U-10w/oMo alloy was the most durable, displaying a relatively uniform tarnish film.


Corrosion of U by gases, moisture, aqueous media, and nonaqueous media is described. U-alloys are grouped according to type: compound-free, U-nonmetal, low miscibility with intermetallic compounds, and miscibles.


Method of making U-alloy containing 0-9w/o of an addition of Zr and Nb in the proportions by wt of 10:3 with improved corrosion resistance and dimensionally stability is described. The alloy is cold rolled, heated to two different temperatures, air-cooled, heated to a third temperature, and quenched in H2O.

Method of improving corrosion resistance of metallic U by adding 0.2-10.0wt/oMo (preferably 1.0-6.0wt/o). The alloy is formed by melting U and Mo together out of contact with air between 1150°-1400°C and subsequently annealing by heat treatment.


Method of producing a ternary alloy with increased corrosion resistance of which major proportion is U containing 0.2-1.0wt/o each of Mo and Si. U-metal of customary purity is heated with Mo and Si out of contact with air at 1150°-1400°C and subsequently subjected to stress relief heat treatment.


Method of optimizing the corrosion resistance of U-alloys containing 3.5-3.7wt/oSi, 0.1-1.5wt/oAl, balance U and consisting of U₃Si, a minor amount of UA₂, and isolated small particles of U₃Si₂. The cast alloy is heat treated at 800°C for ~72-144 hrs until the U₃Si is in the 6-phase.

Aqueous Corrosion


After exposure to high temperature H₂O, γ-phase U-base alloys containing 7-12wt/oMo or Nb, failed either by uniform oxidation or by sudden cracking and disintegration of the metal. The disintegration resulted from the oxidation of a second phase, metastable UH₃, which is precipitated during the corrosion process.


Corrosion behavior of U, U-alloys, Al-alloys, Zr-alloys, and stainless steel in H₂O is described. U-5%Zr and U-5%Zr-1.5%Nb, U-3-6%Nb, U-10%Mo, U-Ti and U-Ti-Nb alloys were investigated.


U-5%Zr-1.5%Nb alloy γ-quenched in H₂O to ~315° showed good corrosion resistance. Optimum heat treatment resulted in a rate of ~6mg/cm²/day at 290°. Moderate aging at 400°C for 2 hrs resulted in reduced corrosion rates in the early stages of corrosion but corrosion resistance could be destroyed by overaging. Samples failed after extended exposure to H₂O and steam and this is believed to be due to absorbed corrosion product hydrogen.

Various methods of increasing corrosion resistance of U in H₂O and steam are discussed with emphasis on alloying and heat treatment. U alloys containing Hf, Ni, Nb, Ti, Zr, Mo, and Si exhibited the highest corrosion resistance and effects of different combinations of these alloys on corrosion resistance are described in relation to heat treatment and exposure to aqueous media. Data for corrosion rates in boiling distilled H₂O, corrosion resistance in H₂O and in H₂O and steam are presented in tabular form. Corrosion rates after various heat treatments are also tabulated.


A windowed autoclave system for study of corrosion by pressurized hot H₂O is described. A method by which corrosion rates were calculated from pressure and temperature measurements is given. Corrosion rates for U-5, 10, 15, and 20w/oZr alloys subjected to 600°F H₂O are reported.


Corrosion of U and U-7.5Nb-2.5Zr alloy in aqueous solution of 0.1M₃SO₄ at pH5-10 and 0.1NH₄SO₄ at 25°C was studied in order to outline the electrochemistry of these materials in the absence of aggressive ions. Reviews and analyses of literature information pertaining to the electrochemistry of U corrosion in aqueous environments at < 100°C and to certain aspects of pitting, crevice corrosion, and SCC are presented in the Appendixes.


In the U-Nb-Zr system, the alloys U-20-30a/0Nb-20a/0Zr proved to be the most corrosion resistant in H₂O at 100° after quenching. After tempering, the alloys U-10a/0Zr-10a/0Nb, U-30a/0Nb, and U-15a/0Zr-15a/0Nb were the most resistant. Corrosion resistance of quenched U-Nb-Zr alloys in H₂O at 100°C is lower than that of the corresponding binary alloys, but quenched U-15a/0Nb-5a/0Mo and U-22.5a/0Mo-7.5a/o% had higher resistance.

Measurements of electrical capacity and conductivity of the surface oxide layers on U-12%Mo alloys indicated that H changed the conductivity without altering the thickness of the layer. The relationship between the conductivity and the rate of corrosion is discussed.


Corrosion of U, Zr, and U-Zr alloy were studied as a function of potential in some alkaline solutions. Corrosion differed significantly from that in a pH5.4 solution. Difference is ascribed to the formation of oxides other than UO₂ in solutions of high pH.


Corrosion of U and U-5-10%Zr was studied in both acidic and basic environments. Activation energy for aqueous corrosion was found to be considerably lower than that for atmospheric oxidation or diffusion of U and O and was influenced by formation potential of the oxide, alloying with Zr, and pH of the environment. It is suggested that electronic conductivity influences corrosion behavior.


The corrosion rates for several U-alloys were measured in three environments: (1) ASTM ocean H₂O at 26°, showed increased corrosion resistance in the order depleted U (Tuballoy), U-4.5Nb, U-6Nb, U-8Nb, U-7.5Nb-2.5Zr (Mulberry), and U-10Mo, (2) 1N HCl at 26° showed increased corrosion resistance in the order Tuballoy, U-4.5Nb, U-6Nb, U-10Mo, Mulberry, and U-8Nb, (3) 0.1N HCl at 70° showed the positions of U-8Nb and Mulberry are reversed from those given for 1N HCl.


The effect of the concentrated citric acid solution temperature on the kinetics of anodic dissolution of α-U in the homogenized state and U-0.45w/oMo alloy in the homogenized and in the β-quenched states was investigated. The anodic behavior is represented by means of the Tafel constants, the exchange currents, the dissolution potentials, and by the activation energies.

The electrochemical behavior of U-3.0w/oNb and U-4.0w/oNb alloys with banded martensitic structure in concentrated citric acid was investigated using the potentiostatic method to determine the polarization curves, Tafel constants α and i0. Fine microstructural details such as dislocations and etch-pits were revealed using low current densities.


A collection of 52 references with abstracts on the aqueous corrosion of U and U-alloys covering the period 1952 to early 1963. Nuclear Science Abstracts, The Review of Metal Literature and current periodicals were used as the sources of information.


Experimental data for the aqueous corrosion of U and various γ-phase U alloys are provided. A "hydroxide" mechanism theory which incorporates the features of both the hydride and oxide mechanisms is suggested.

Oxidation


U-1w/oCu alloy was oxidized in a CO2 atmosphere at 500°C to determine the effect of Cu on the rate of oxidation. The presence of UCu₅ at the metal/oxide interface increased oxidation resistance of the alloy.


Weight gain-time curves for U-up to 7.3%Si, U-10%Si, U-10%Ti, U-5%V, U-10%Zr, U-15%Mo, U-10%Nb and U-1%Cu alloys in CO2 at 500°-1000° and in air at 500°C were obtained. Additions of Ti, Mo, Nb, and Cu reduced attack by CO2 at 680°-1000° by factors up to 500. None of the elements was found to be markedly beneficial in CO2 at 500° and Si had a high rate of attack at most temperatures. In air at 500°C, additions of Mo, Ti, and Nb reduced attack by factors up to 200.

Isothermal oxidation studies of U, U-1a/oCu, and U-1a/oAl alloys were carried out at 300°-625° and showed substantially identical self-accelerating reaction rates up to 400°. The effect of oxidation kinetics of the two alloy additives studied were useful in understanding the differences in ignition behavior for binary U-alloys. Empirical equations were formulated for the results of isothermal oxidation studies for pure U above 300°C.


Oxidation of U-0.6w/oMo-0.6w/oZr-0.5w/oNb-0.5w/oTi alloy at 300°-400°C in O at pressures of 1-10 torr was studied. Parabolic reactions were found for all conditions except for the aged material at 400°C where reaction curves were "S" shaped and exhibited "breakaway" rates. Oxidation of both the quenched and aged alloys was faster than that of the binary U-6.0-8.0w/oNb alloys.


Surface stresses between 105-106 psi are frequently generated during the oxidation of metals and several stress generating mechanisms are discussed. The influence of such stresses on scale rupture, structure and morphology, diffusion rates within the scales, and on shape change experienced by oxidation specimens is pointed out as is the evidence for extensive high-temperature plasticity in the oxides. The oxidation of U-16.6a/oNb-5.6a/oZr and U-21a/oNb alloys is described to emphasize the importance of the mechanical properties of scales in gas-metal reactions.


Tensile and creep properties of U-21a/oNb, U-16.6a/oNb-5.6a/oZr alloys were studied over a temperature range 750°-900°C and creep data were fitted to an equation, \( \varepsilon = k\varepsilon^\alpha \exp(-Q/RT) \), which was used to calculate stresses in oxidizing alloy specimens based on elongation measurements. Estimates were made of the average stress in the oxide scale as a function of time. A comparison of the relative stress levels indicated that the major difference in the oxidation properties could be attributed to changes in the mechanical properties of scales.


The oxidation rate data for U-9.4w/oNb and U-7.5w/oNb-2.5w/oZr alloys at 100°-300°C in pure O are presented. Both alloys formed compact, generally protective, oxide films. The reaction appeared to be diffusion controlled and the activation energy, \( \sim 13 \text{ kcal/mole} \), suggests the probable importance of short-circuit diffusion modes in the process. Gaseous impurities - H2O, SO2, CC14 - slightly increased the oxidation rate of the ternary alloy.

Oxidation characteristics at 700°-1100°C of U-16.6 at%Nb-5.6 at%Zr, U-21 at%Nb, U-15 at%Zr, and U-18 at%Mo alloys are discussed. In all cases oxide formation involved the generation of large stresses, ~10^6 psi, in the oxides that lead to large increases in the lateral dimensions of the specimens. Stress relief processes, including plastic deformation of both the oxide and metal, are discussed.


Oxidation properties of U-base alloys, U-16.6 at%Nb-5.6 at%Zr and U-21 at%Nb at 500°-1000°C in O at 0.05 torr are described. At temperatures >650°C both alloys undergo extraordinarily large dimensional changes. Oxidation rate curves were determined at 100°C intervals between 500°C-1000°C. Activation energy for the process was ~15 kcal/mole. Morphology of the oxide scale formed on both alloys is described in full detail. Stresses in the oxide were estimated to be ~106 psi. A mechanism for stress generation in the oxide is proposed.


The oxidation resistance and mechanical properties of 25 U alloys including U-Ti alloys, U-0.5Ni, U-0.5Cr, U-0.5Ni-0.5Cr, U-1.5Mo-0.5Ni-0.5Cr, U-1.5Mo, and U-2Nb-2Ti were evaluated. U-1w/oTi and U-2w/oMo alloys had good yield strength (>100,000 psi) and good ductility (>9% elongation). Additions of Nb were required to achieve corrosion resistance and only U-7.5w/oNb-2.5w/oZr (Mulberry) alloy could withstand exposure at 260°C but U-4.5w/oNb-2.5w/oV alloys had excellent corrosion resistance at 120°C when hot-rolled. Mechanical properties of U-Ti alloys can be controlled by aging and optimum properties were obtained by aging at 400°C for 4 hrs. Increased homogeneity by thermal processing and mechanical working also increased the corrosion resistance.


Mechanisms of atmospheric corrosion of pure U and U-alloys in relation to the effects of temperature and humidity on the corrosion rates are described. Data for the resistance of various U-alloys under different test conditions are given. The corrosion rates of U in H2O saturated steam, and Ar, and the free energy of formation of U compounds are presented in tabular form.

Oxidation behavior of U-alloys containing 0.9, 3.6, 8.1 and 18.8/oTi were studied in pure CO₂, using weight gain, metallographic and x-ray techniques. Only corrosion product identified was U₂O₅. U₁₇Ti resisted oxidation by CO₂, and in U-Ti alloys containing < 4/oTi there was little effect on the oxidation rate but in those containing > 4/oTi the oxidation rate was increased.


The results of a series of qualitative U-H corrosion tests are presented. The effects of surface treatment and inhibitive agents on the corrosion reaction are discussed.


Using an ellipsometric high vacuum technique, oxidation kinetics (surface oxide thickness up to 2000Å) of U-7.5w/oNb-2.5w/oZr alloy (Mulberry) in air containing H₂O vapor was investigated. The alloy samples were oxidized in dry air, < 5 ppm H₂O, and in air containing H₂O vapor, 15,000 ppm H₂O, at 630 torr in the temperature range 22°-200°. The oxidation followed a parabolic-rate law, indicating a diffusion-controlled mechanism and the temperature dependence of the reaction was examined by the Arrhenius equation. A difference in the activation energy of the low temperature (22-100°) and the high temperature (100°-200°C) was revealed.


Rate of formation of oxide films and the diffusion rates in these films on U, Zr, and U-Zr alloys are described. The influence of H₂O and H on the protective properties of oxide films was studied.


The presence of a kinetic barrier, usually a surface layer of a reaction product through which the reactants and sometimes the products diffuse, causes some metals to oxidize slowly. In order to understand the factors influencing the diffusion rate the oxide layer was considered not only an inert obstacle to ionic diffusion, but as an electrolyte and semiconductor. Electronic conductivity and H pick up of the metals in the case of oxidation by H₂O or steam as well as oxide thickness and structure were found to be important. The effect of alloying U with Zr on oxidation is used as an example.
Experimental studies concerned with the measurement of metal-H2O reaction rates and determining emissivity in order to measure the chemical reaction of steam with U and U-Nb-Zr alloys when contacted at high temperatures in nuclear reactors are reported. Spectral and total emissivities were measured for all materials in the as-rolled condition with O added at 0°C-1075°C.

Cold-rolled, γ-phase U-7w/oMo and U-10w/oNb alloys were oxidized in H2O vapor forming highly oriented oxide films. Dodecahedral planes of UO2 were formed parallel to the specimen surface. An epitaxial relationship was found between the UO2 and the Nb alloy.

Flexure technique was used to observe the stress buildup during oxidation of a U-7.5w/oNb-2.5w/oZr alloy. Measurements indicated that the effective stress in the oxide film approached 108 psi and were used to establish a mechanism accounting for the stress generation and relief behavior.

Reaction of U-7.5w/oNb-2.5w/oZr alloy with CO2, H2O, N and O gases was surveyed. With CO2 a linear reaction was found at 600°C but with the other gases parabolic reactions were generally found and deviations were attributed to spalling of the reaction product. At 600°C catastrophic reactions were found with H2O and O. The reactivity of the gases can be ordered as CO2 > H2O > N > CO.

The sputter cleaning and oxidation of U and U-7.5w/oNb-2.5w/oZr were studied by an ellipsometric method. The reactive index of both these materials and of their oxides was measured. Corrosion curves were drawn at several different temperatures for films up to 4500Å thickness and equations of the curves were determined. The effect of tensile stress on the ellipsometric data was studied.

X-ray diffraction of the oxidation of U-7.5\%Nb-2.5\%Zr alloy was studied at 150°, 200°, 400°, 500°, and 600° in both dry and wet air and with different surface preparations. At 150°-250°, \( \text{UO}_2 \) was formed, at 400°, \( \text{U}_2\text{O}_3 \) was also formed, and at 500°-600°C, \( \text{U}_2\text{O}_3 \) was formed immediately. Results indicated that \( \text{U}_2\text{O}_3 \) does not form under normal storage conditions (< 200°C) and therefore played no part in the mechanism of SCC of Mulberry alloy.

Stress Corrosion Cracking (SCC)


Characteristics of slow cracking, in air and in \( \text{H}_2\text{O} \) of U-10\%Mo alloy were studied. The propagation was discontinuous, and the cracking occurred with divergent or non-divergent branching. The fracture faces also exhibited these characteristics. A hypothesis of H embrittlement is advanced.


Electrochemistry and SCC properties of U-7.5\%Nb-2.5\%Zr alloy were studied. Potential variations were detected on the surface and anodic currents were introduced in air equilibrated moisture films on the alloy. Electrochemistry of the stress-corrosion attack was similar to that of the metal having no oxide film. The SCC was accelerated by Cl ions penetrating the oxide film. Cold-worked alloy was resistant to SCC and the mechanism was unrelated to a pitting-reaction mechanism.


Effects of heat treatment and ambient temperatures on the SCC behavior of U-10\%Mo alloys were studied using a field-emission microscope. Cathodic protection, the effect of static electric DC fields, and a special gas shield were investigated as possible protective techniques. It was found that heat treatments influenced the SCC behavior. A tentative critical stress for initiation of SCC was set at 36,500 psi and was strongly dependent upon heat treatments and critical structures. Cathodic protection and electric fields had no effect on the SCC behavior but pure dry N was found to be most effective as a protective shield during cyclic loading. Also reported in Applications of Field-Ion Microscopy in Physical Metallurgy and Corrosion, R. F. Hochman, E. W. Mueller, and B. Ralph, eds.

Two types of smooth SCC tests - constant deflection 4-point bend tests and static tensile tests - were used to determine the SCC behavior of U-4.2w/oNb alloy aged 80 hrs at 260°C. SCC occurred when the alloy was stressed and exposed to H2O containing both 0 and Cl ions. Cracks which nucleated in the Cl ion environment proceeded by an intergranular fracture mode. No failures were observed in dry air containing < 200 ppm moisture or in 100% relative humidity air. Tensile stress required for SCC was < 20,000 psi in 0 saturated H2O containing 50 ppm Cl ions.


SCC behavior of U-4.5w/oNb alloy aged 80 hrs at 200°C was investigated using smooth four point bend and static tensile SCC tests. Results showed susceptibility to SCC when stressed and exposed to H2O containing 0 and Cl ions. In the aqueous Cl environment SCC proceeded by an intergranular fracture mode. In dry air (< 200 ppm moisture) and in 100% relative humidity no failures were observed. Surface tensile stress required for SCC in 0 saturated H2O containing 50 ppm Cl ions was < 20,000 psi.


SCC tests using smooth, dead-weight loaded, static tensile specimens which were exposed to 0 saturated with H2O containing 50 ppm Cl ions were conducted on a single phase underaged (230°-24 hrs) and on a two phase overaged (430°C-24 hrs) U-2.3w/oNb alloys. Results showed that the underaged alloy was more susceptible to SCC by a transgranular mode and the overaged alloy could fail by corrosion penetration that leads to mechanical failure.


Room temperature tests showed that vapor deposited Al coatings can protect aged U-4.5%Nb alloy from SCC in aqueous chloride environments. This degree of protection increased with increasing coating thickness and decreased with increasing size of uncoated flaws.


In thermal processing of γ-quenched U-5w/oNb alloy optimum resistance to the initiation of SCC was obtained by aging above 550°. A change in fracture mode from intergranular for low aging temperatures to transgranular, quasi-cleavage, for high aging temperatures was observed. A wire specimen aged for 8 hrs at 600°C had a UTS of ~ 160 ksi with 17% elongation.
SCC of U-7.5w/oNb-2.5w/oZr alloy was studied by failure mechanics and SEM. Two modes of cracking were observed. A transgranular mechanism occurred in dry and wet gaseous environments with O required for propagation and a $K_I_{SCC} \approx 18$ ksi $\sqrt{in}$. An intergranular mechanism occurred in high relative humidity conditions and aqueous environments with $H_2O$ required for propagation and a $K_I_{SCC} \approx 5$ ksi $\sqrt{in}$. Cold working inhibited the intergranular mode of failure.

The effect of Cl ions on the SCC behavior of U-7.5w/oNb-2.5w/oZr and U-4.5w/oNb alloys was investigated. Time-to-failure at a given stress intensity decreased in both alloys as the Cl content in aqueous solutions increased ($< 2$ ppm-50 ppm). Cracking without application of stress when the Cl level reached 500 ppm is attributed to corrosion product wedging. Data indicate that two cracking mechanisms - dissolution and mechanical - are operative.

Susceptibility to SCC in several environments was determined for U-7.5w/oNb-2.5w/oZr, U-4.5w/oNb, U-10w/oMo, and U-0.5w/oTi alloys. All the alloys listed failed in laboratory air. The humidity in the air did affect the time-to-failure but did not significantly affect the threshold intensities required for cracking. Test environments ranged from $< 10\% RH$ air to $100\% RH$ air, pure gases (O, N, H, and CO$_2$), and aqueous solutions containing $< 20$ to 500 ppm Cl.

SCC behavior of U-4.5w/oNb alloy was determined by dry ($< 10\% RH$) air using side-grooved tapered double-cantilevered-beam specimens. Crack propagation was monitored using crack opening displacement gages and acoustic emission detection techniques. The threshold stress intensity for SCC was determined to be $\approx 20$ ksi $\sqrt{in}$ and the plain strain fracture toughness $\approx 50$ ksi $\sqrt{in}$.

Effect of Cl ions on the SCC behavior of U-4.5w/oNb and U-7.5w/oNb-2.5w/oZr (Mulberry) alloys was investigated. Both the fracture mode and the critical stress intensity for SCC, $K_{I_{SCC}}$, were found to be strongly dependent on Cl ion concentration.

SCE behavior of U-4.5w/oNb reinforced with 0, 17, 38, and 51w/oW fibers was studied in 100%RH air. Results indicated that as the W content increases, the susceptibility to SCC decreases to the point that the 51% composite is only slightly susceptible. Fractographic analysis showed that not all the fibers were equally effective in reinforcing the matrix.


Two SCC modes of U-7.5w/oNb-2.5w/oZr (Mulberry) alloy were observed: (1) transgranular (quasi-cleavage) which required 0 for propagation and had a critical stress intensity of ~ 20 ksi√ft; (2) intergranular which occurred both in high relative humidity air and in aqueous environments.


The effects of loading rate, stress level, and notch factor on the resistance to stress corrosion of U-10w/oMo alloy in air and at ambient temperatures were studied. In general, the rough-cast material was found to be stronger than the normalized.


The stress-corrosion resistance of U-10w/oMo alloy in both the normalized and rough-cast states was studied in air and in H2O. Crack propagation occurred after an incubation period and the rate was constant. In air, transgranular cracking was observed, but in H2O both transgranular and intergranular cracking were found.


A simple rapid test to evaluate the effect of heat treatment on SCC susceptibility of several U-alloys: U-10w/oMo, U-7.5w/oMo, U-7.5w/0Nb-2.5w/oZr, and U-6w/oNb, is described. The test had the advantages of speed of assessment and the use of standard laboratory apparatus and results were in agreement with those of longer term tests.

Metastable γ-phase U-8w/oMo-0.50w/oTi alloy was studied during static and low strain-rate loading and exhibited stress cracking in air. The average critical stress observed (60,000 psi with 75 ppm C) was higher than that observed in the U-Mo binary alloy but too low to be acceptable for use under stress except in O free environments. Transformation from the γ to the α + γ condition eliminated stress-cracking brittleness.


Two U-7.5w/oNb-2.5w/oZr alloys - one solution treated by quenching from the γ field and the other solution treated and aged for 1 hr at 150°C - were tested to evaluate the delayed cracking propensity. Tests used were sustained loading near yield strength in dry and wet air and dynamic testing to failure at strain rates of 0.05-0.005in/in/min in dry and wet air and in vacuum. Delayed cracking was not observed in the alloys which were finish-machined after heat-treating.


U-10w/oMo alloy was found to be subject to delayed failure in air due to O. Critical or threshold stress was as low as 37,000 psi but increased with decreasing C content. The offset yield strength in the dynamic tensile test was typically 125,000 psi.


The cause of cracking in U-4w/oMo alloys was investigated. Studies showed that cracking could be eliminated by testing tensile specimens in a He atmosphere. Results of various experiments on both U-4w/oMo and U-5w/oMo alloys showed that cracking was transgranular, occurred only in tension, and was caused by SCC due to the presence of O in the atmosphere. Increasing the temperature above 25°C increased the rate of cracking and in the U-5w/oMo alloy, no cracking was observed below 10°C.

The effect of various metallurgical and electrochemical variables on the initiation of SCC in U-7.5Nb-2.5Zr, U-4.5Nb, U-6.5Nb, and U-8.5Nb alloys was determined. Passivation behavior of U-7.5Nb-2.5Zr alloy was determined in several different solutions using a potentiostat. Bend specimens, immersed in aqueous solutions and held at constant potentials, were used to investigate the SCC initiation of the various alloys. The effect of potential on crack initiation in U-7.5Nb-2.5Zr alloy in 10^{-4} M KCl was determined.


Field emission microscopy was used to study the effects of heat treatment and ambient temperatures on the SCC behavior of U-10w/oMo alloys. Cathodic protection, the effect of static DC fields, and a special "gas shield" protective technique were investigated. SCC behavior was influenced by heat treatments. Susceptibility was reduced by a γ-homogenization and quench followed by a tempering treatment. Tentative critical stress for initiation of SCC, strongly dependent upon heat treatment and intermicrostructure, was set at 36,500 psi. Cracking was found to be intergranular, transgranular, or both depending on the particular atmosphere.


Representative microstructures of U-7.5w/oNb-2.5w/oZr alloy specimens heat treated at selected temperatures (150°-600°C) were obtained from both SCC and mechanical property data. Results showed that, in general, mechanical strength and resistance to SCC increase as the aging temperature increases, particularly when a two-phase microstructure is produced. Polarization curves indicated that the general corrosion resistance was not significantly affected by heat treatment.


The effect of O, Cl and H_{2}O vapor on U-7.5w/oNb-2.5w/oZr alloy, in particular on susceptibility to initiation of SCC, was studied. Results indicated that when the Cl present was < 2 ppm, a smooth specimen with or without Ni couples did not exhibit crack initiation during a 3 year period. Also in a Cl environment that would otherwise be detrimental, crack initiation was prevented if the O is reduced to a very low but indeterminable amount.
SCC in U-10w/oMo and U-6w/oMo alloys and the effect of moisture on U-7.5w/oNb-2.5w/oZr alloy have been investigated. Initial results indicated that moisture not 0 affected the stress corrosion of the binary alloys and that cracking in the 6w/oMo alloy was intergranular not transgranular as reported by others. High humidity also caused intergranular cracking in the binary alloy.

Protective Coatings


A reliable method for providing an adherent and protective electroplate of Ni on U was developed. A method for evaluating specimens and the detailed results are presented. A plated specimen with an exposed U-Ni interface was subjected to a H2O saturated atmosphere at 200-250°F. Specimens can be soldered and give the full strength properties of other good soldered joints. The plate cannot be peeled or chipped with a chisel.


Inhibiting or limiting the oxidation of U by CO2 at high temperatures by means of protective metallic coatings was investigated. Surface films containing intermetallic compounds or solid solutions of U with Al, Zr, Cu, Nb, Ni, or Cr were formed. Processes recommended are direct diffusion starting from a thin sheet or tube, vacuum deposition, and immersion in a molten bath of suitable composition. Conditions were optimized as a function of the protective effect obtained in CO2 at 450° or 500°C. Only Zr and Al based coatings were satisfactory, and provided the U good protection against atmospheric corrosion.


U-10w/oMo alloy fast-burst reactor parts were ion plated with thin (< 2x10^-4in) Al coatings to protect the alloy from oxidation and as a means of containing the fission products. These coatings were compared with Ni coatings. At fissions > 10^17 and temperatures > 700°C the Ni plating spalled severely while the Al and the U-Al intermetallics formed during the process remained adherent to the alloy.

Ion-plated Al coatings provide excellent corrosion protection on unalloyed depleted U when exposed in an accelerated corrosion test (100°C - 1 atom H2O vapor pressure). Processing parameters which influence corrosion protection were determined to be substrate precleaning, amount of gaseous contamination in the glow discharge, sputter cleaning voltage and pressure, filament contamination, deposition rate, and amount of U-Al interdiffusion, either during plating or in subsequent vacuum heat treatment. A diffusion layer consisting of U-Al intermetallic compounds is believed to provide corrosion protection in this system.


Coating of depleted U with thin Al coatings (< 5 x 10^-4 in) using the ion plating process provided corrosion protection of U exposed to a condensed H2O vapor at 950°C for up to 95 hrs. Surface preparation and deposition parameters are discussed. Vacuum deposited coatings did not provide corrosion protection.


Methods of depositing metals, e.g. Zn, on U to inhibit corrosion were investigated. Anhydrous plating with zinc acetate-formamide gave good adhesion but an oxide film was formed between the U and the coating. A method of depositing Zn, having an appreciable vapor pressure, directly on U showed promise but additional experiments are necessary. A simple method for determining the porosity on the electroplated coatings on U was developed.


Improvements in the process for plating U with an adherent and protective Ni coating are reported (see entry No. 419). A superior pretreating solution using NiCl rather than SnCl and a new method for cleaning pieces to be plated are described.


A process for reliably protecting unalloyed depleted U with an adherent corrosion-resistant electroplated deposit was developed. The key to the process was treatment of U with a metal chloride/HNO3 solution (preferably SnCl2 or NiCl2) before plating. Plated parts, pretreated with any metal chloride solutions were capable of withstanding exposure to a H2O saturated atmosphere at 200°F for > 90 hrs without deterioration. Plated parts pretreated in the SnCl2 solution showed no signs of deterioration after five years of outdoor exposure.

Salt spray (fog) and SCC tests were conducted on an aged U-4.2 wt% Nb alloy having a sprayed paint coating consisting of an epoxy primer with a polyurethane cover coat. Results showed some protection from corrosion in a 3.5 wt% NaCl salt fog environment but when subjected to appropriate surface tensile stresses failure by SCC occurred at flaws or scratches in the point coating.


The incubation time for the initiation of SCC in γ-quenched U-5 wt% Nb alloy was extended by coating the alloy with Cd or Al. Coatings of 10-25 μm thickness were deposited by electroplating with Cd and by plasma spraying, PVD, CVD, and electroplating with Al. No attempt was made to develop continuous impermeable coatings. Coating type, aging times and temperatures, time-to-failure, test environment, and gap size data are presented in tabular form.


Procedures for depositing metals on U were investigated by the National Bureau of Standards. Plating Cr, Ni, Cu from aqueous baths was not satisfactory but plating of Al and Zn from anhydrous baths prevented the formation of a black oxide film between the base and plating metals. A simple method for determining porosity in the U-plated specimens stored 24 hrs in an evacuated vessel is to partially refill with H and heat to ~200°C. Any U exposed through a pore in the plate is attacked by the H.


The corrosion behavior of U electroplated with Ni was investigated. Due to loss of adhesion the protective quality of the plating decreased as exposure to aqueous environments increased due to the formation of UH₃ which resulted from H penetration at the Ni-U interface. Corrosion could be inhibited if the H generated were free to escape. A mechanism for the corrosion of U beneath the Ni plate is proposed.


Both Al and Cd coatings on U-5.0 wt% Nb alloy significantly reduced susceptibility to SCC. Unprotected samples under a static tensile stress of 44,000 psi failed in less than 30 min exposure to 10⁻⁴ M KCl solution but the coated samples lasted 1-4 mos under similar conditions. Results indicated that the Al and Cd coatings sacrificially protect the base alloy. Four methods of applying the coatings are described.

The change in corrosion potential as a function of the changing U-alloy area fraction in galvanically connected Ni-U alloy couples in KCl electrolytes was determined. The technique was calibrated so that the exposed base metal area fractions of U-alloy specimens plated with Ni could be estimated.


Method of preparing articles of U and U-alloys for the reception of a protective metal coating by immersing the article in an etching bath consisting of an aqueous solution of either FeCl₃ or NiCl₂ for 10-15 min at 30°-60°C, rinsing it in H₂O, and then immersing it in a solution containing an Fe solvent to remove the surface Fe.


Method for providing a U-metal article with a tenaciously adhering metal plating deposited from an aqueous solution. Oxides and other contaminants are removed from the surface and the article is then heated in vacuum under selected conditions to form thereon a very thin and highly protective oxide film which is non-wettable by H₂O but may be made wettable by H₂O by contact with a basic wetting solution. The resulting wettable, oxide-coated article is then electroplated with metal in an aqueous metal-plating bath.


Method of surface preparation of U and U-base alloys prior to electroplating parts. Preliminary procedures involve chemical degreasing, electrolytic degreasing, and if necessary chemical etching and an anodic electrolytic treatment in an aqueous solution of an organic salt or a Li and/or Mg sulphate.


Method of electrodepositing metallic coatings on parts fabricated of U and U-alloys is described. A first coating, containing at least one metal layer, is heated under vacuum at 350°-400°C for 1-3 hrs to degas the metal coating, then mechanically treated by cold-working to close the surface defects of the first coating. A second coating, containing at least one metal layer, is electrodeposited to provide an imperious composite coating. At least one of the coatings must be formed by successive deposition of layers of different metals.
Method of electroplating articles the surface of which consists of U-alloys and others. A first layer of metal is electrodeposited followed by a second layer (55 μm) of Sn, Zn, Pb, or In which is impervious to outside agents. Plated articles to be primarily used in plating rods or plates of alloyed U for nuclear fuels.
**ALLOY INDEX**

**Binary Alloys -**

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