DWR-1564

Y-1732

Metals, Ceramics,
and Materials

AEC RESEARCH AND DEVELOPMENT REPORT

MASTER

THE ANOMALOUS MECHANICAL PROPERTIES OF
URANIUM-7.5 WEIGHT PERCENT NIOBIUM-2.5
WEIGHT PERCENT ZIRCONIUM ALLOY

R. E. Oakes, Jr

THIS DOCUMENT CONFIRMED AS
UNCLASSIFIED
DIVISION OF CLASSIFICATION
BY 9.H.Kahn/lamb
DATE 10/9/70

UNION CARBIDE CORPORATION
NUCLEAR DIVISION
OAK RIDGE Y-12 PLANT

operated for the ATOMIC ENERGY COMMISSION under U. S. GOVERNMENT Contract W-7405 eng 26

UNION
CARBIDE

OAK RIDGE Y-12 PLANT
P. O. Box Y
OAK RIDGE, TENNESSEE 37830

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

P6944

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

Printed in the United States of America. Available from Clearinghouse for Federal
Scientific and Technical Information, National Bureau of Standards,
U.S. Department of Commerce, Springfield, Virginia 22151
Price: Printed Copy \$3.00; Microfiche \$0.65

LEGAL NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

- A. Makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or
- B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission, or employee of such contractor, to the extent that such employee or contractor of the Commission, or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with the Commission, or his employment with such contractor.

Date Issued: September 22, 1970

Document Y-1732

TID-4500

UNION CARBIDE CORPORATION
Nuclear Division

OAK RIDGE Y-12 PLANT

Operated under Contract W-7405-eng-26
With the US Atomic Energy Commission

THE ANOMALOUS MECHANICAL PROPERTIES OF
URANIUM-7.5 WEIGHT PERCENT NIOBIUM-2.5
WEIGHT PERCENT ZIRCONIUM ALLOY

R. E. Oakes, Jr

LEGAL NOTICE

This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Atomic Energy Commission, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

Oak Ridge, Tennessee

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

THIS PAGE
WAS INTENTIONALLY
LEFT BLANK

Document Y-1732

TID-4500

DISTRIBUTION

Atomic Energy Commission

Keller, C. A.

Zachry, D. S., Jr

Battelle-Northwest

Hays, D. D.

Dow-Rocky Flats

Seed, J. R.

Oak Ridge Gaseous Diffusion Plant

Jordan, R. G.

Nolan, T. A.

Wilcox, W. J., Jr

Oak Ridge National Laboratory

Vandermeer, R. A.

Yakel, H. L.

Oak Ridge Y-12 Plant

Alvey, H. E.

Ammons, A. M.

Beeson, H. C.

Bernander, N. K.

Blake, H. W.

Burkhart, L. E.

Cadden, J. L.

Denny, A. (2)

Ellingson, R. D.

Evans, G. W.

Gritzner, V. B.

Haeusler, K. R.

Hemphill, L. F.

Hulsey, W. J.

Jackson, V. C.

Johnson, D. H.

Kahl, K. G.

Keith, Alvin

Kite, H. T.

Ludwig, R. L.

Mason, D. L.

McLendon, J. D.

Mitchel, G. W.

Oakes, R. E., Jr (10)

Phillips, L. R.

Reeves, C. A.

Smith, H. F., Jr

Smith, R. D.

Stoner, H. H.

Tilson, F. V.

Townsend, A. B.

Warner, T. D.

Waters, J. L.

Wesley, R. L.

Winslow, F. R.

Yaggi, W. J.

Y-12 Central Files (5)

Y-12 Central Files (route)

Y-12 Central Files (master copy)

Y-12 Central Files (Y-12RC)

Paducah Gaseous Diffusion Plant

Winkel, R. A.

Sandia-Livermore

Mote, M. W.

In addition, this report is distributed in accordance with the category Metals, Ceramics, and Materials, as given in the "USAEC Standard Distribution Lists for Unclassified Scientific and Technical Reports", TID-4500.

ABSTRACT

Abnormal mechanical properties in the uranium-7.5 weight percent niobium-2.5 weight percent zirconium alloy (such as nonlinear "elasticity", rubber-like amounts of "elastic" spring back, and large amounts of mechanical hysteresis) have been examined in uniaxial tension and compression with respect to stress, strain, and aging. The results of these mechanical tests indicate the occurrence of a stress-induced phase transformation in both the as-quenched and aged alloy. Volumetric, resistance, and microstructural changes which occur under stress in the alloy further substantiate the occurrence of such transformation.

CONTENTS

SUMMARY	7
INTRODUCTION	8
DETERMINING THE ANOMALOUS MECHANICAL PROPERTIES OF THE URANIUM-NIOBIUM-ZIRCONIUM ALLOY	9
Material	9
Tensile and Compressive Properties	9
Test Equipment	9
Gamma-Quenched Alloy Properties	13
Gamma-Quenched and Aged Alloy Properties	16
Volumetric, Resistance, and Microstructural Properties	16
Volume Changes under Stress	16
Resistivity Changes under Stress	23
Micrographic Changes under Tensile Stress	24
Conclusions	31
REFERENCES	35
ACKNOWLEDGEMENTS	36

SUMMARY

The uranium-7.5 weight percent niobium-2.5 weight percent zirconium alloy has been shown to have abnormal mechanical properties both in the as-quenched and aged conditions. Among these abnormal properties are: (1) pronounced nonlinear "elasticity", (2) an unusual amount of "elastic" spring back, and (3) large amounts of mechanical hysteresis. These mechanical properties along with nonlinear volumetric changes under stress, nonlinear resistivity changes under stress, and microstructural changes under stress indicate the occurrence of a stress-induced phase transformation. The nonlinear "elasticity" observed in the alloy was found to coincide with the transformation threshold stress, and the large amount of "elastic" spring back was found to result from a reversibility of the transformation. Permanent strain was found to coincide with the formation of transformation products which were shown to be reversible through aging. Permanent strain was also found to lower the external transformation threshold stress, most likely by creating internal stresses produced by the change in volume which accompanies the transformation. It is hypothesized that the effect of aging on unstrained material is to relieve internal stresses created during quenching, thus raising the transformation threshold.

INTRODUCTION

The uranium-based alloy containing 7.5 weight percent niobium and 2.5 weight percent zirconium is the best uranium alloy yet produced for satisfying the requirements of corrosion resistance, fabricability, and strength. These properties are obtained by maintaining a form of the high-temperature, body-centered cubic gamma phase at room temperature.

The mechanical properties of this alloy are dependent upon the extent of the short-time, low-temperature aging given the material after the quenching process. According to Yakel,⁽¹⁾ who performed X-ray analyses on single crystals of the alloy, the high-temperature, body-centered cubic gamma phase is modified upon quenching to a transition gamma structure (γ^S) which he describes as being a modified body-centered structure in which the body-centered atoms are displaced from their normal position. Short-time aging at 150 or 350° C, according to his analyses, transforms the γ^S structure to yet another transition structure (γ^O) which is body-centered tetragonal. This same aging process changes the mechanical properties of the alloy and thus provides a control over these properties through heat treatment. The unaged gamma-quenched material typically yields in tension at about 60 ksi (if the 0.2 percent offset method is applied), has an ultimate tensile strength of about 125 ksi, and is ductile enough for good fabricability (greater than 20 percent elongation). By aging, the yield stress can be elevated to as high as 120 ksi (0.2 percent offset) and the tensile strength to more than 150 ksi while maintaining adequate ductility (still greater than 10 percent elongation). By choosing the proper heat treatment, these mechanical properties can be enhanced between these extremes to meet material application requirements.

During routine testing to establish the mechanical properties of this alloy, several anomalous phenomena have been observed, namely: (1) a significant change in the "elastic modulus" with permanent strain, (2) an uncommonly large amount of nonlinear "elasticity", (3) a large amount of hysteresis between loading and unloading, and (4) a near-rubber-like amount of "elastic" spring back. Hays,⁽²⁾ while determining the yield surface of this uranium alloy, reported a "moderate amount" of mechanical hysteresis in the alloy and also observed that a second loading cycle results in a lower "yield stress" than is observed during the initial loading cycle. Observations such as these prompted this investigation first into the mechanical properties of the alloy and, subsequently, into some other properties which help explain the observed mechanical behavior.

DETERMINING THE ANOMALOUS MECHANICAL PROPERTIES OF THE URANIUM-NIOBIUM-ZIRCONIUM ALLOY

MATERIAL

The material used for these tests was skull cast in ingot form, homogenized for 20 hours at 900° C, rolled into plate form, and cut into blanks of approximate specimen size. These blanks were then air annealed at 800° C for one hour and water quenched to room temperature. This procedure of quenching the small specimen blanks instead of the full-size plate was followed in an attempt to obtain a uniform quench throughout the specimen. The aging treatment, if any, was applied after the specimen machining was completed. All aging was done at 150° C for a period of 100 hours. The normal aging period for this material at 150° C is one hour, but the extended aging period was chosen to best demonstrate the differences in material properties obtained by aging.

It should be pointed out that the tensile, compressive, electrical resistivity, and photomicrographic specimens each came from different ingots, and some differences in the mechanical properties result from this fact.

TENSILE AND COMPRESSIVE PROPERTIES

Test Equipment

An Instron universal testing machine provided loading for both the uniaxial tension and compression tests. A four-arm strain gage, bending-beam-type extensometer with less than 0.25 percent combined nonlinearity and hysteresis, provided the longitudinal strain analog during the tensile testing. An instrumented tensile specimen is shown in the testing machine in Figure 1. Early in the work, the results of this type of extensometer were compared with the results from an instrumented specimen strain gaged so as to cancel bending, if present, to verify that the reported anomalous behavior is not a result of nonsymmetrical loading of the specimens. These duplicate measurements were nearly identical, verifying that the anomalous behavior is indeed a true material property.

An LVDT-type compressometer, shown in Figure 2 mounted on a specimen in the spherical-seated alignment fixture, was used for measuring the longitudinal compressive strain. Figures 3 and 4 show, in block diagram form, the instrumentation used during the tensile and compressive tests, respectively.

134469

Figure 1. TENSILE SPECIMEN IN THE TEST MACHINE. (Longitudinal and Transverse Strain Sensors Attached)

The conventional technique of measuring the slope of the stress-strain plot from an X-Y recording was used to determine the modulus values in tension and compression. Modulus values are shown on the respective stress-strain curves.

134471

Figure 2. COMPRESSION SPECIMEN IN THE ALIGNMENT FIXTURE. (Compressometer is Installed)

Figure 3. INSTRUMENTATION USED FOR THE TENSILE TESTS.

Figure 4. INSTRUMENTATION USED FOR THE COMPRESSION TESTS.

Gamma-Quenched Alloy Properties

This material was tested in the as-quenched condition with no artificial aging. The engineering stress-strain relationship for the unaged alloy in tension is seen in Figure 5.

Specimens were strained to predetermined strain values and the stress was then removed, with unloading occurring along the path indicated by the downward arrows. The strain path observed on reapplication of stress often differs from the unloading path, resulting in large hysteresis loops (labeled A through F, Figure 5). These hysteresis loops can be retraced many times by application and removal of stress without observable changes in shape as long as no additional permanent strain is added. The first such loop (A), with no permanent strain, has the appearance of a normal linear elastic material with only a small amount of hysteresis. In the second loop, where the material is stressed to a higher value than the first, deviation from linearity can be seen to occur at about 0.35 percent strain (Loop B), but no residual strain is observed upon unloading even after the specimen has been strained 1.4 percent and released. Further straining to 2.7 percent results in only 0.35 percent residual strain after unloading and illustrates the rubber-like properties of the material. A distinct change in slope or "knee" is observed as the stress is increased and decreased during Loops B, C, and D. Permanent strain lowers the stress at which this knee is observed until, in Loop E, the knee is no longer observable. For this specimen, additional permanent strain beyond two or three percent has little effect on either the modulus or shape of the subsequent hysteresis loops. The amount of permanent strain required to stabilize the characteristics of the hysteresis loop was found to vary from two percent in some as-quenched material to as much as ten percent in other material. Quenching conditions, chemical composition, and gaseous content all appear to have some effect on this characteristic of the alloy although these factors are difficult to control for a thorough analysis.

To provide convenient reference to these properties of the gamma-quenched alloy, the stress-strain diagram has been divided into three regions—I, II, and III. In Region I, the material behaves as a normal elastic material; in Region II, the properties are highly dependent upon the amount of permanent strain; in Region III, the characteristics have stabilized with respect to additional permanent strain.

In compression (Figure 6), the as-quenched alloy behaves basically the same as in tension. The normal elastic-type behavior is seen in Loop A, while Loops B through E have the same characteristics as the loops of Region II in the tensile tests of the as-quenched alloy. Machine-force limitations and specimen-stability problems prevented further straining in compression and, as a result, Region III was not reached.

Figure 5. TENSILE STRESS AS A FUNCTION OF THE STRAIN FOR AN UNAGED URANIUM-NIOBIUM-ZIRCONIUM ALLOY. (At Room Temperature; Strain Rate $= 0.00033 \text{ sec}^{-1}$)

Figure a. COMPRESSIVE STRESS AS A FUNCTION OF THE STRAIN FOR AN UNAGED URANIUM-NIOBIUM-ZIRCONIUM ALLOY. (At Room Temperature; Strain Rate = 0.00033 sec^{-1})

Gamma-Quenched and Aged Alloy Properties

Tensile stress-strain data taken from the alloy which had been aged for 100 hours at 150° C are seen in the graph of Figure 7. Loop A is a Region I-type elastic cycle and extends to a higher stress level than in the as-quenched material. The overall strength has been increased by aging as well as the stress required to reach nonlinearity in Loops B through E. The modulus likewise for the aged material is higher than for unaged material. Permanent strain decreases the modulus and lowers both the loading and unloading knee stress much as in Region II of the as-quenched material. No Region III-type properties are observed over the strain range investigated.

In compression, Figure 8, the same basic results were obtained as in tension.

Abnormal mechanical properties, such as changes in slope with application and removal of stress, large amounts of "elastic" spring back, and large hysteresis loops, have been reported in copper-zinc and titanium-nickel alloy systems and are results of stress-induced phase transformations.⁽³⁻⁶⁾ In Figure 9, taken from Pops,⁽³⁾ a loading-unloading stress-strain curve is shown for a copper-zinc-silicon alloy single crystal in which a stress-induced, diffusionless, martensitic-type transformation occurs. The similarities between this hysteresis loop and a hysteresis loop from the uranium-niobium-zirconium alloy are quite noticeable.

To further investigate the possibility of such a stress-induced phase transformation in the uranium alloy, tests were performed to determine if abnormal volumetric, resistance, or microstructural changes occur under stress. If so, such changes would further indicate the existence of a stress-induced transformation.

VOLUMETRIC, RESISTANCE, AND MICROSTRUCTURAL PROPERTIES

Volume Changes under Stress

To investigate volume changes in the alloy associated with the nonlinear behavior under stress, simultaneous longitudinal and transverse strain measurements were made as a function of the tensile stress. The transverse strain sensor was shown in Figure 1. The volume of unstressed material within the gage length of the longitudinal and transverse strain sensors is:

$$V_o = \ell_o w_o t_o,$$

where ℓ_o , w_o , and t_o represent the length, width, and thickness, respectively, of the unstressed material. When stress is applied, the new volume of this

Figure 7. TENSILE STRESS AS A FUNCTION OF THE STRAIN FOR THE URANIUM-NIOBIUM-ZIRCONIUM ALLOY THAT HAS BEEN AGED FOR 100 HOURS AT 150° C. (At Room Temperature; Strain Rate = 0.00033 sec⁻¹)

Figure 8. COMPRESSIVE STRESS AS A FUNCTION OF THE STRAIN FOR THE URANIUM-NIOBIUM-ZIRCONIUM ALLOY THAT HAS BEEN AGED FOR 100 HOURS AT 150°C. (At Room Temperature; Strain Rate = 0.00033 sec^{-1})

Figure 9. STRESS-STRAIN CURVE AT 27° C FOR A SINGLE CRYSTAL OF COPPER-ZINC-SILICON ALLOY "C". (Taken from H. Pops)

same material becomes (assuming isotropic behavior in the thickness and width directions which are both normal to the direction of applied stress):

$$V = (\ell_o + \epsilon_\ell \ell_o)(w_o + \epsilon_t w_o)(t_o + \epsilon_t t_o),$$

where ϵ_ℓ and ϵ_t are the measured longitudinal and transverse strains, respectively. The volume ratio of the stressed to unstressed material is then:

$$\frac{V}{V_o} = \frac{(\ell_o + \epsilon_\ell \ell_o)(w_o + \epsilon_t w_o)(t_o + \epsilon_t t_o)}{\ell_o w_o t_o}, \text{ which reduces to:}$$

$$\frac{V}{V_o} = (1 + \epsilon_\ell)(1 + \epsilon_t)^2. \quad (1)$$

This volume ratio, calculated from Equation 1 and using measured values of ϵ_ℓ and ϵ_t , was plotted as a function of tensile stress for a number of loading-unloading cycles. Figure 10 shows the strain and corresponding volumetric

(a) Strain vs Tensile Stress

(b) Volume Ratio vs Tensile Stress

Figure 10. STRAIN AND VOLUME RATIO AS FUNCTIONS OF THE TENSILE STRESS DURING THE "ELASTIC" LOADING-UNLOADING CYCLE. (Unaged Alloy; No Permanent Strain).

relationships as a function of tensile stress for an as-quenched alloy specimen with no previous strain during a loading-unloading cycle into Region II. Slope changes in the volumetric expansion and contraction occur at the same approximate stresses as slope changes in the stress-strain curve, and the process is reversible. Figure 11 shows similar relationships for as-quenched

(a) Strain vs Tensile Stress

(b) Volume Ratio vs Tensile Stress

Figure 11. STRAIN AND VOLUME RATIO AS FUNCTIONS OF THE TENSILE STRESS DURING THE "ELASTIC" LOADING-UNLOADING CYCLE. (Unaged Alloy; 1.2% Permanent Strain)

alloy with 1.2 percent permanent strain in which the knees have been nearly suppressed by permanent strain. The slope of this volumetric expansion curve is constant and is the same as the slope of the upper portion of the curve in which the knee was present, Figure 10(b).

Anomalous volumetric changes were also found to occur in the uranium alloy which had been aged for 100 hours at 150° C. Changes in the slope of the volumetric expansion and contraction curve were observed to occur at the same stress as the changes in slope of the stress-strain curve (Figure 12). The volumetric changes of this aged alloy are also fully reversible.

Figure 12. STRAIN AND VOLUME RATIO AS FUNCTIONS OF THE TENSILE STRESS DURING THE "ELASTIC" LOADING-UNLOADING CYCLE. (Alloy was Aged for 100 Hours at 150° C; 0.3% Permanent Strain)

Resistivity Changes under Stress

Electrical resistivity data as a function of the applied tensile stress were also obtained. A small-diameter tensile specimen was electrically insulated from the testing machine and extensometer arms in order to eliminate extraneous parallel resistance paths. A Keithley Model 503 milliohmmeter was used to measure the specimen resistance. Figure 13 presents a view of the

134857

Figure 13. INSTRUMENTED ELECTRICAL RESISTIVITY SPECIMEN IN THE TENSILE MACHINE.

instrumented resistivity specimen in the testing machine with an attached extensometer, current leads, and potential leads. A block diagram of the instrumentation used for these resistivity tests is given in Figure 14.

Figure 14. INSTRUMENTATION USED FOR THE RESISTIVITY VERSUS STRESS/STRAIN TESTS.

Figure 15 is a graph of the strain and electrical resistivity versus tensile stress for an as-quenched alloy specimen with a 1.08 percent permanent strain (corrected for the simultaneous specimen dimensional changes). Changes of slope in this curve occur simultaneously with changes in slope in the stress-strain relationship. Similar results are shown in Figure 16 for the uranium alloy that was aged for 100 hours at 150° C and strained to 2.17 percent. Changes in resistivity which occur under stress were observed to be fully reversible in both the as-quenched and aged alloy, provided that permanent strain was not added during the cycle.

Micrographic Changes under Tensile Stress

Photomicrographs were taken of the as-quenched alloy to determine the effects of stress and strain on its microstructure. A simple force-reversal fixture was designed which, when clamped in a bench vise and placed on the optical

(a) Strain vs Tensile Stress

(b) Electrical Resistivity vs Tensile Stress

Figure 15. STRAIN AND ELECTRICAL RESISTIVITY AS FUNCTIONS OF THE TENSILE STRESS. (Unaged Alloy; 1.08% Permanent Strain)

microscope, enabled specimens to be observed under tensile stress. This fixture with a specimen in place is seen in Figure 17. A strain gage was attached to the specimen for the purpose of determining the stress that was applied by tightening the fixture.

Prior to testing, the micrographic specimens were electropolished with six percent perchloric acid in methanol at -40°C and then etched with five percent oxalic acid to reveal the grain boundaries and structure. The microstructure of an area of a specimen after this treatment is presented in Figure 18. No

(a) Strain vs Tensile Stress

(b) Electrical Resistivity vs Tensile Stress

Figure 16. STRAIN AND ELECTRICAL RESISTIVITY AS FUNCTIONS OF THE TENSILE STRESS.
(Alloy was Aged for 100 Hours at 150° C; 2.17% Permanent Strain)

stress has yet been applied. This same area is shown in Figure 19 with a 39.5-ksi tensile stress applied (slightly beyond Region I). The appearance of the dark bands coincides with the departure from linearity in the stress-strain curve and are a result of surface upheavals. These upheavals are somewhat difficult to visualize from this photograph, although they are apparent by direct observation. Upon removal of the stress (Figure 20), the bands (assumed to be caused by reflections from the upheavals) recede, leaving slight surface marks where the dark bands previously existed. No residual strain is observed. The formation of these crack-like marks explains why this uranium alloy, which has been stressed past the Region I elastic range

134858

Figure 17. FIXTURE FOR THE APPLICATION OF TENSILE STRESS TO MICROGRAPHIC SPECIMENS.

OM-257

Figure 18. ELECTROPOLISHED AND ETCHED ALLOY SPECIMEN, (No Stress, No Strain; 450X)

OM-293

Figure 19. ALLOY SPECIMEN WITH A 39.5-KSI APPLIED STRESS, SHOWING FORMATION OF SURFACE UPHEAVALS. (450X)

OM-298

Figure 20. ALLOY SPECIMEN AFTER REMOVAL OF THE 39.5-KSI STRESS, SHOWING A RECES- SION OF THE SURFACE UPHEAVALS, LEAVING SURFACE MARKS. (450X)

and released, requires less stress to reach the knee on reapplication of the stress, even when no permanent strain is observed. These crack-like formations appear to act as nucleation centers for transformation when stress is reapplied. Figure 21 shows the same grain after the 39.5-ksi tensile stress has been reapplied. Reformation of the surface irregularities indicates a reversibility of the transformation. Increasing the applied stress to 57.5-ksi (Figure 22) causes growth of the existing surface upheavals and formation of a number of new ones. A further increase in stress to 65 ksi (Figure 23) continues to cause growth of the existing upheavals as well as formation of more new ones. Removal of all stress (Figure 24) causes the larger upheavals which formed first to diminish in size while the smaller upheavals recede leaving only the crack-like surface marks. The process has become partially irreversible as far as external stress is concerned, as evidenced by permanent upheavals. Permanent strain is now observed (about 2.7%). Similar observations of reversibility and growth of bands were reported by Pops⁽³⁾ in a copper-zinc-tin alloy. If a specimen of the uranium alloy is electropolished and etched after permanent strain is applied, a banded surface is observed and these bands have a different response to polarized light from the parent portion of the grain. Dean,⁽⁷⁾ while studying the time-temperature transformation characteristics of the uranium alloy, showed that a high-temperature age (325° C for one minute) removes these bands,

OM-300

Figure 21. ALLOY SPECIMEN WITH THE 39.5-KSI STRESS REAPPLIED, SHOWING THE REVERSIBILITY OF THE SURFACE UPHEAVAL FORMATION. (450X)

OM-302

Figure 22. ALLOY SPECIMEN WITH THE STRESS INCREASED TO 57.5 KSI, SHOWING GROWTH OF THE EXISTING UPHEAVALS AND FORMATION OF NEW ONES. (450X)

OM-318

Figure 23. ALLOY SPECIMEN WITH THE STRESS INCREASED TO 65 KSI, SHOWING FURTHER GROWTH OF THE EXISTING UPHEAVALS AND FORMATION OF NEW ONES. (450X)

OM-321

Figure 24. ALLOY SPECIMEN WITH THE STRESS REMOVED, LEAVING A 2.7 PERCENT PERMANENT STRAIN, SHOWING PARTIAL RECESSION OF THE SURFACE UPHEAVALS. (450X)

which he interpreted as being twins. However, after this aging, the small crack-like surface marks (similar to those observed upon removal of stress in this study) remained. Figure 25, taken from Dean's work, shows these surface marks which remain after one minute of the high-temperature aging process. This photograph indicates that aging reverses the effects of permanent strain.

CONCLUSIONS

Nonlinear volumetric expansion, nonlinear resistivity changes, and microstructural changes during application and removal of stress indicate that the anomalous mechanical properties of this uranium-niobium-zirconium alloy result from a stress-induced phase transformation. The nonlinear elasticity observed occurs when the applied stress exceeds a transformation threshold (knee) stress. Above this transformation threshold, greater dimensional changes occur for a given stress increment than occur in the truly elastic region below the transformation threshold, resulting in the observed decrease in slope above the knee. The rubber-like "elastic" behavior is a consequence of this transformation being reversible, under application and removal of stress, until a true yield stress is reached. Permanent strain in the alloy appears to be a result of the irreversibility of the transformation, or at least irreversible transformation products are observed simultaneously with ir-

Figure 25. MICROSTRUCTURE OF THE URANIUM ALLOY AFTER HAVING BEEN GIVEN A ONE PERCENT PLASTIC DEFORMATION AND SUBSEQUENT AGE FOR ONE MINUTE AT 325° C. (Etchant: 5% Oxalic Acid Solution; 250X; Taken from Dean)

reversible strain. No evidence was observed which would explain why the transformation is reversible below the yield stress and irreversible above the yield stress.

Decreases in the transformation threshold stress with increasing permanent strain appears to be a result of the creation of stresses around the transformation product in the parent and adjacent grains. These localized stresses are created by the volumetric change which accompanies the irreversible transformation during permanent straining. Application of external forces increases the magnitude of these stresses around the transformation products and results in an apparent lowering of the threshold stress, as observed

externally. The lenticulated shape of the transformation products seen in the photomicrographs is evidence of the existence of these stresses around the transformed material. Such stresses may account for the decrease in knee stress with permanent strain observed in Region II and the disappearance of the knee from Region III. Permanent strain and the resulting volume changes in Region II reduce the observed threshold stress by creating more and larger transformation products with more internal stress fields. The growth of these products as stress is applied along with the normal elastic deformation of the parent material results in a lowered "modulus" as more transformation products form by straining. If enough permanent strain is applied, the internal stresses become large enough to reach and then exceed the transformation threshold. When the threshold stress has been reached or exceeded by these localized stress fields alone, no knee is observed and the slope of the loading curve for any stress increment is similar to the slope of the upper portion of the loading curves in Region II, resulting in the observed properties of Region III.

Since similar behavior is observed in both tension and compression, the effective stress in bringing about the transformation must be shear, which would be present in polycrystalline material during both tension and compression testing.

The same explanation of the observed mechanical properties holds true for both the as-quenched and aged materials. The aged material with its higher modulus (10×10^6 psi aged, 8×10^6 psi as quenched) and higher threshold stress may indicate the presence of fewer and/or smaller internal stress fields. Relief of these stresses could occur during aging and would result if the $\gamma^s \rightarrow \gamma^o$ transformation upon aging is toward a smaller volume, which assumes that both γ^s and γ^o form during quenching. This latter change is highly likely since only a few minutes aging at 150°C is necessary to bring about observable differences in the mechanical properties. X-ray diffraction patterns of polycrystalline uranium alloy in the as-quenched condition generally show either no distinct diffraction pattern or extremely broad lines, a fact which supports the hypothesis of a stressed structure caused by the two different phases being present. Since aging a specimen with permanent strain removes optical evidence of transformation products, this fact suggests that the strain transformation may be the reverse of the aging transformation, giving for polycrystalline materials:

where the $\gamma^S + \gamma^O$ combination is a stressed state. Of course the relative proportions of $\gamma^S + \gamma^O$ would vary according to the quenching conditions or the amount of aging or strain. This hypothesis is the simplest explanation of the observed data, but the possibility remains of a more complex structure, such as that reported for the titanium-nickel alloy.⁽⁸⁾ It is apparent that crystallographic studies, especially under stress, are needed to identify these phases, and it is hoped that the information contained herein will be of some assistance to others studying the various properties of this alloy system.

REFERENCES

- (1) Yakel, H. L.; Crystal Structures of Transition Phases Formed in U-16.60 At % Nb-5.64 At % Zr Alloys, Not Published; Metals and Ceramics Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee.
- (2) Hays, D. D.; Yield Surface of the Uranium-7.5 Wt % Nb-2.5 Wt % Zr Alloy, BNWL-1112; Battelle Memorial Institute, Pacific Northwest Laboratory, Richland, Washington; August 1969.
- (3) Pops, H.; "Stress-Induced Pseudoelasticity in Ternary Cu-Zn Based Beta Prime Phase Alloys", Metallurgical Transactions, 1, pp 251-258 (1970).
- (4) de Lange, R. G. and Zijderveld, J. A.; "Shape-Memory Effect and the Martensitic Transformation of TiNi", Journal of Applied Physics, 39, pp 2195-2200 (1968).
- (5) Rozner, A. G. and Wasilewski, R. J.; "Tensile Properties of NiAl, and NiTi", Journal of the Institute of Metals, 94, pp 169-175 (1966).
- (6) Buehler, W. J., Gilfrich, J. V., and Wiley, R. C.; "Effect of Low-Temperature Phase Changes on the Mechanical Properties of Alloys Near Composition TiNi", Journal of Applied Physics, 34, pp 1475-1477 (1963).
- (7) Dean, C. W.; A Study of the Time-Temperature Transformation Behavior of a Uranium-7.5 Weight Percent Niobium-2.5 Weight Percent Zirconium Alloy, Y-1694; Union Carbide Corporation-Nuclear Division, Oak Ridge Y-12 Plant, Oak Ridge, Tennessee; October 24, 1969.
- (8) Wang, F. E., de Savage, B. F., and Buehler, W. J.; "The Irreversible Critical Range in the TiNi Transition", Journal of Applied Physics, 39, pp 2166-2175 (1968).

ACKNOWLEDGEMENTS

The author wishes to thank T. D. Warner of the Y-12 Metallurgical Development Department for suggesting this work, for supplying material for the tests, and for the many helpful discussions concerning this project. The author is also indebted to D. H. Johnson of the Y-12 Laboratory Development Department for his assistance with the metallographic investigations.