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REVERSIBLE MARTENSITIC TRANSFORMATIONS BETWEEN TRANSITION
PHASES OF URANIUM-BASE NIOBIUM ALLOYS

Ross J. Jackson

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CONTENTS

Abstract	1
Introduction	1
Experimental	1
Transition Phases and Composition	2
Transition Phases and Temperature	2
U-15 at. % Nb: $\alpha'' \leftrightarrow \gamma$ Thermal Reversibility	3
U-15 at. % Nb: Thermally Reversible Parameter Changes	3
U-19 at. % Nb: Thermally Reversible Parameter Changes	3
U-11 at. % Nb: Thermally Reversible Parameter Changes	3
Transition Phase Diagram	4
Modelistic Explanation	4
Summary	5
References	6
Illustrations	7-11

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REVERSIBLE MARTENSITIC TRANSFORMATIONS BETWEEN TRANSITION PHASES OF URANIUM-BASE NIOBIUM ALLOYS

Ross J. Jackson

Abstract. The occurrence and generation of transition phases in uranium-niobium alloys is discussed. The transition phases and unit cell parameters as a function of composition and temperature are presented. Also presented is an approximated transition phase diagram. The highly anisotropic lattice parameter changes of orthorhombic α' , monoclinic α'' and tetragonal γ^0 with temperature and composition are discussed from a crystallographic viewpoint. Inherent in this discussion is the consecutiveness and thermal reversibility of the α' , α'' and γ^0 structures; and the thermal reversibility of change in monoclinic cell angle of the α'' phase.

INTRODUCTION

Two uranium-niobium equilibrium diagrams have been reported.^{1,2} Other equilibrium aspects of the system such as phase stability and decomposition^{3,4} have been investigated. The occurrence and generation of transition phases in uranium-base niobium alloys have been studied by Anagnostidis, *et al.*,⁵ and Tangri and Chaudhuri.⁶ A temperature-induced shape memory in polycrystalline uranium-base niobium alloys has been characterized by Jackson and Johns.⁷ The occurrence, generation, and stability of transition phases in the U-Mo,^{8,9,10,11} U-Re,¹² U-Ru,^{13,14} U-Ti,¹⁵ and U-Zr^{16,17,18} systems have also been investigated.

The transition phases and their composition range of stability observed after water quenching from the bcc γ solid solution in the U-Nb system are summarized below:

- α'_a -A supersaturated α -uranium transition phase containing 1 to 5 at. % Nb. Alpha prime has the same crystal symmetry as α (orthorhombic) but differing unit cell parameters. There is a notable contraction of the b_0 parameter and expansion of a_0 and c_0 parameters with increasing niobium content. The subscript refers to an acicular microstructure. Alpha prime sub "a" forms by a martensitic $\beta \rightarrow \alpha$ transformation.
- α'_t -Similar to α'_a except for a higher niobium content (5 to 9 at. % Nb) and a highly twinned microstructure. Alpha prime sub "t" forms by a direct $\gamma \rightarrow \alpha'$ martensitic transformation, i.e., there is no intermediate beta.

α'' -A supersaturated α -like transition phase containing 9 to 16 at. % Nb. Alpha double prime has similar parameter changes as α' , and, in addition, shows an increase in cell angle between the a and b axes thus resulting in a monoclinic crystal symmetry with "c" axis unique. Alpha double prime sub "t" forms directly from γ in a martensitic reaction.

γ^0 -A tetragonal transition phase containing 16 to 21 at. % Nb. Gamma naught is similar to the bcc γ phase except two of the cell axes have expanded while the third has contracted. The structure is based on a block of 2 by 2 by 1 γ -like bct subcells with the super cell having $c/a < 0.5$.

The work reported to date on transition phases in uranium alloys has mainly concerned itself with: (a) the effect of composition, and to some extent cooling rate, on the transition phase obtained,⁸⁻¹⁷ (b) the crystallography of the transition phases,^{8-11,18} and (c) the shears involved to generate the transition phases, as well as their orientation relationships.^{9,11,18}

This report presents and discusses three experimental observations concerning the transition phases. These observations are: (a) a significant, continuous, diffusionless, athermal, reversible, and highly anisotropic change in unit cell parameters occurs on heating or cooling; (b) a continuous and highly anisotropic change in unit cell parameters occurs on increasing the solute content; and (c) at certain combinations of temperature and composition, the martensitic-like changes in the unit cell parameters manifest themselves as symmetry changes, which are likewise athermal, reversible, and highly anisotropic. These observations have resulted in an extension and confirmation of an earlier modelistic explanation,⁹ as well as in an approximated constitutional diagram for the transition phases.

EXPERIMENTAL

The purity and description of the uranium-niobium alloys are described elsewhere.⁷

Two variable-temperature X-ray diffractometers were used. The variable-temperature stage, used from -125 to $+250^\circ\text{C}$, consisted of a jet of precooled helium or preheated argon gas impinging on the sample. The precooled helium was obtained by passing it through a

coiled copper tube immersed in liquid nitrogen. The preheated argon was obtained by passing it through a fused quartz tube which was surrounded by a tube furnace. The sample temperature was varied by adjusting the flow of gas impinging on the sample. A thermocouple embedded in the sample was used to record temperature.

The other hot-stage x-ray diffractometer was of commercial design (MRC Model X-86-G). In this unit the sample was heated with a tantalum element. Vacuums of 5×10^{-7} torr were maintained during the heating and soaking cycles. The X-ray data at -196°C were obtained by immersing five sides of the sample in liquid nitrogen. Filtered CuK_α radiation was used. Lattice parameters were determined by least squares extrapolation against the Nelson-Riley function using an IBM Model 40-G, System 360 computer.

TRANSITION PHASES AND COMPOSITION

The effect of composition on transition phase structure in the U-Nb system has been determined by Anagnostidis *et al.*,⁵ and by Tangri and Chaudhuri.⁶ Both studies are in good agreement as to the compositional range of stability of the transition phases at room temperature. However, there are significant differences as to the value of unit cell parameters as a function of composition. For this reason their basic experiment was repeated. The results of the three independent studies are shown in Figure 1. The differences in reported unit cell parameters for a' and a'' were resolved in favor of Anagnostidis. The differences in reported unit cell parameters for γ^0 were also resolved. The values were found to be intermediate to those reported by Anagnostidis *et al.*,⁵ and Tangri and Chaudhuri.⁶

Figure 2 is a schematic representation of differences between equilibrium phases (α and γ) and transition phases (a' , a'' , and γ^0). Figure 2a shows schematic x-ray diffraction patterns and a unit cell for α , a' , and a'' . The packing and postulated shape of atoms in the $(001)_\alpha$ close-pack plane is also shown in this figure. Figure 2b shows schematic γ and γ^0 X-ray diffraction patterns and a γ^0 unit cell.

In this investigation, the lattice parameters a_0 , b_0 , c_0 , and angle γ of the a' and a'' structures were determined using specimens water quenched from 850°C . These results are represented by triangles in Figure 1. The main features are:

1. For a' and a'' , the values of a_0 and c_0 increase with increasing niobium content.

2. For a' and a'' , the value of b_0 decreases markedly with increasing niobium content.
3. The cell angle γ in a'' gradually increases from 90.0° at 9 at. % Nb to 92.8° at 15 at. % Nb.
4. For γ^0 , the value of a_0 increases and c_0 decreases with increasing niobium content. At 20.5 at. % Nb the c/a axial ratio for the subcell equals one.

The effect of introducing niobium atoms into the uranium lattice may be explained in the following manner. The unequal interatomic distances in α uranium give an ellipsoidal shape to each uranium atom rather than a spherical shape (Figure 2a). Assuming that each atom behaves as a hard particle, the niobium atoms may be represented as hard spheres of radius 1.472 Å when in a coordination of twelve, and uranium atoms may be represented as hard ellipsoids having center-to-edge distances along principle directions of closest approach of 1.381 Å (0, .21, .50), 1.426 Å (100), and 1.630 Å (110). The substitution of niobium spheres into the lattice of ellipsoids results in the theoretical lattice parameters shown in Figure 1. The theoretical values of a' are based on $a_0 = 0.098 \text{ at. \% Nb} + 2.582$, $b_0 = -0.568 \text{ at. \% Nb}$, and $c_0 = 0.328 \text{ at. \% Nb} + 4.945$ which are, at best, rough approximations. The agreement between experimental and theoretical lattice parameters is good considering that size was the only consideration in the latter calculation.

The lattice parameters for the retained γ alloys are shown to decrease with increasing niobium content. Chiotti *et al.*,¹⁹ report a value of $a_0 = 3.538 \text{ Å}$ for pure uranium at 800°C . Correcting this value to room temperature using the linear coefficient of thermal expansion determined by Chiotti *et al.*,¹⁹ of $22.5 \times 10^{-6}/^\circ\text{C}$ yielded an a_0 at 23°C of 3.476 Å. Hence, the addition of niobium atoms causes a contraction of the bcc γ cells. This contraction would be expected on the basis of size considerations. Theoretical lattice parameters for γ are shown in Figure 1, and agree well with experimental values. Size was the only consideration in calculating the theoretical parameters. Using a value of $a_0 = 3.301 \text{ Å}$ for bcc Nb yielded $a_0 = -0.175 \text{ at. \% Nb} + 3.475$ for a coordination number of eight.

TRANSITION PHASES AND TEMPERATURE

Representative results of low and high temperature x-ray diffraction studies are shown in Figure 3. This figure shows the reversible parameter and symmetry changes for three compositions. The parameter chosen to represent the a'' structure is the monoclinic cell angle ($L\gamma$), whereas, for tetragonal γ^0 the chosen parameter

is the c/a axial ratio. Parameter and symmetry changes for the three compositions are discussed individually in the following paragraphs.

U-15 at. % Nb: $a'' \leftrightarrow \gamma^0$ Thermal Reversibility:

The U-15 at. % Nb alloy water quenched from 850°C is single-phase, monoclinic a'' at 23°C with $\angle\gamma = 92.6^\circ$. This composition on heating undergoes a change to a single-phase tetragonal γ^0 structure between 60° and 90°C. That is, 60°C is the A_S temperature and 90°C is the A_F temperature. The γ^0 and a'' structures coexist between A_S and A_F . However, the individual phases could not be identified metallographically and it was assumed the two structures were near coherent. The $a'' \rightarrow \gamma^0$ transition is athermal in character, i.e., holding between A_S and A_F (at least for 4 hours) did not alter the relative amounts of the two phases present. That is to say, the x-ray diffraction peak intensities did not change.

The $a'' \leftrightarrow \gamma^0$ symmetry change is thermally reversible. The reverse transformation occurs in approximately the same temperature interval as it does on heating. The differences in M_S and A_F , and M_F and A_S were in both cases less than 10° Celcius. The thermal reversibility of the $a'' \leftrightarrow \gamma^0$ transition was illustrated by cycling a sample six times through the transformation in a hot stage x-ray diffractometer. The transformation temperatures remained approximately constant throughout the thermal cycling.

The M_S , M_F , A_S , and A_F temperatures are strongly dependent on niobium composition. Increasing the niobium content lowers the transformation temperatures. This effect is discussed later.

U-15 at. % Nb: Thermally Reversible Parameter Changes:

As mentioned earlier the U-15 at. % Nb alloy water quenched from 850°C is single-phase, monoclinic a'' at 23°C with $\angle\gamma = 92.6^\circ$. On cooling the alloy to -196°C, the monoclinic cell angle decreases to a value of 91.6°. On reheating to 23°C, the sample returns to its initial monoclinic angle of 92.6°. On heating to higher temperatures, the monoclinic cell angle increases reaching a limiting value of 92.8 at +60°C. Between +60 and +90°C there is change in symmetry to a tetragonal γ^0 structure having a 0.477 c/a axial ratio. On heating to higher temperatures the c/a axial ratio continually increases (i.e., the structure is approaching bcc symmetry). On cooling back to 23°C the parameter and symmetry changes are reversible and the structure is again monoclinic a'' with $\angle\gamma = 92.6^\circ$. The parameter and symmetry changes on heating

and cooling are completely reversible and are also athermal in character, i.e., not dependent on time at temperature.

The thermally reversible change of c/a axial ratio for γ^0 and monoclinic cell angle for a'' in a U-15 at. % Nb alloy is shown in Figure 3. Each datum point corresponds to a parameter calculated from a single x-ray diffractometer pattern. A single sample was used for the 25 data points. The sample was cooled to -196°C, heated to +250°C, cooled to 23°C, and reheated to 400°C. If the apex of the datum triangle points towards lower temperature then the value was obtained on cooling. The equilateral triangles represent values obtained on initial cycles, whereas the elongated triangles represent values obtained on subsequent thermal cycles.

U-19 at. % Nb: Thermally Reversible Parameter Changes:

The U-19 at. % Nb alloy water quenched from 850°C is single-phase, tetragonal γ^0 at 23°C with $c/a = 0.487$. This alloy on heating or cooling undergoes a thermally reversible change in c/a axial ratio. An increase in temperature causes an increase in c/a axial ratio, i.e., the structure is approaching cubic symmetry.

The thermally reversible change of c/a axial ratio for the tetragonal γ^0 phase in a U-19 at. % Nb alloy is shown in Figure 3a. On cooling the alloy to -130°C the c/a axial ratio decreases to a value of 0.482. On reheating to 23°C, the sample returns to its initial value of $c/a = 0.487$. Heating to 200°C results in an increase in c/a axial ratio to 0.495. On cooling back to 23°C the parameter changes are reversible and the c/a ratio is again 0.487. If these data are extrapolated, one could predict that a symmetry change from tetragonal γ^0 to bcc γ would occur at approximately 300°C. Likewise, a symmetry change from tetragonal γ^0 to monoclinic a'' would occur at approximately -210°C, provided a c/a axial ratio of 0.477 is the limiting value.

U-11 at. % Nb: Thermally Reversible Parameter Changes:

The U-11 at. % Nb alloy water quenched from 850°C is single-phase, monoclinic a'' at 23°C with $\angle\gamma = 91.0^\circ$. This alloy on heating or cooling undergoes a thermally reversible change in monoclinic cell angle. An increase in temperature causes an increase in monoclinic cell angle.

The thermally reversible change of monoclinic cell angle for the a'' phase in a U-11 at. % Nb alloy is shown in Figure 3b. On cooling to -120°C, the

$\angle\gamma$ decreases to a value of 90.2. At -196°C , the monoclinic cell angle was 90.0, hence the structure was a' . Interpolation of these data indicate the $a'' \rightarrow a'$ symmetry change occurred at approximately -150°C . On reheating the sample to 23°C , the sample returns to its initial value of $\angle\gamma = 91.0^\circ$. Heating to 300°C results in an increase in $\angle\gamma$ to a value of 92.4° . On cooling back to room temperature, the parameter changes are reversible and the $\angle\gamma$ is again 91.0° . If the high temperature data are extrapolated, one could predict that a symmetry change from monoclinic a'' to tetragonal γ^0 would occur at approximately 400°C provided a monoclinic cell angle of 92.8 is a limiting value.

TRANSITION PHASE DIAGRAM

The hot and cold X-ray diffraction studies discussed earlier, plus studies on similar and different compositions, have resulted in an approximated transition phase diagram for uranium-base niobium alloys. This diagram is shown in Figure 4 and defines the temperature-composition regions in which a given transition phase exists in quasi-equilibrium. The symmetry changes in the transition phase diagram, as in an equilibrium phase diagram, are all thermally reversible.

In interpreting the transition phase diagram, certain interesting features not shown on the diagram should be kept in mind, these are:

1. In the a' region the b_0 cell axis decreases as the niobium content and/or temperature increases. The a'/a'' boundary corresponds to a value of $b_0 \approx 5.83 \text{ \AA}$.
2. In the a'' region the monoclinic cell angle ($\angle\gamma$) increases as the niobium content and/or temperature increases. The a''/γ^0 phase boundary corresponds to a value of $\angle\gamma \approx 92.8^\circ$.
3. In the γ^0 region the c/a axial ratio increases as the niobium content and/or temperature increases. The γ^0/a'' phase boundary corresponds to a value of $c/a \approx 0.477$. The γ^0/γ phase boundary corresponds to a value of $c/a = 0.500$.
4. Above 350°C , for the times required to obtain suitable X-ray diffraction patterns, there is an interfering diffusion-controlled precipitation reaction. The amount of decomposition (e.g., $a'' \rightarrow a + \gamma_{1-2}$) increases as the temperature and time at temperature increase.

5. In moving from one T-X point to another in the single phase regions, the transition phases undergo continuous martensitic-like cell parameter changes.
6. The T-X areas separating single phase regions are areas representing "coherent mixtures" of two transition phases having differing crystallographic symmetries.

MODELISTIC EXPLANATION

A sketch of the γ^0 unit cell appears in Figure 2b. The γ^0 unit cell is a supercell based on a block of 2 by 2 by 1 γ -like subcells. The subcells are body-centered tetragonal with $c/a < 1.0$, and the supercell has $c/a < 0.5$. The supercell is necessary to account for weak superlattice reflections which are due to slight atom displacements and/or to restricting the niobium atoms to edge center sites. The subcell is closely related to the bcc γ cell, but there is a contraction in one of the bcc cell axes with an expansion along the other two orthogonal cell axes. This behavior can be pictured as the spherical bcc atom becoming squashed in one direction with attendant expansion in the other two principal directions. (an oblate spheroid).

A sketch of the a uranium unit cell appears in Figure 2a. The a uranium unit cell is orthorhombic and can be interpreted as an ABAB stacking of the (002) close-packed plane. The structure is like hexagonal metals except successive layers of atoms in the basal plane are skewed back and forth in the $\langle 010 \rangle$ directions. Thus, the packing and stacking in a uranium is similar to that in a hcp metal. For example, $c/a = 1.63$ and $b/a = 1.73$ for hcp metals indexed on an orthohexagonal cell, whereas for a uranium $c/a = 1.73$ and $b/a = 2.06$. Thus the a -uranium atom is a slightly extended spheroid with the long dimension in the b_0 direction, i.e., it approximates a prolate spheroid.

The effect of temperature and niobium composition on the consecutiveness and cell parameters of a' , a'' , γ^0 , and γ can be rationalized by assuming that the uranium atom becomes more spherical as the niobium content is increased and as the temperature is increased.

The effect of increasing niobium content is readily pictured as the substitution of spherical niobium atoms for ellipsoidal uranium atoms. The net effect of this is to decrease the ellipsoidal nature of the uranium atoms as the niobium content is increased. Temperature has a similar effect, in that the ellipsoidal nature of the

uranium atom decreases as the temperature increases. Chiotti *et al.*,¹⁹ have shown that there is an expansion of a_0 and c_0 and a contraction of b_0 as the temperature of α uranium increases. This change can be attributed to a delocalization of bonding electrons, i.e., the bonding is becoming more metallic and less covalent in character.

The "spheroidizing" of uranium atoms on increasing the temperature or niobium content results in a notable contraction of the b_0 parameter of U-Nb alloys with little effect on a_0 and c_0 parameters. Thus the c/a axial ratio between α and α' remains roughly the same, whereas there is a notable decrease in the b/a axial ratio. Therefore, α' is a more "hexagonal-like" metal than is α uranium.

When the b_0 parameter contracts to about 5.83 Å, there is a restriction toward closer approach in the b_0 direction and the lattice accommodates more niobium atoms by an increase in $\angle\gamma$ (first appearance of α''). This has the net effect of allowing a relative contraction in the b_0 direction, but at the same time maintaining the restriction toward closer approach in this direction. At a higher temperature or niobium content there is an increase in $\angle\gamma$ up to a value of 92.8° at which time there is a change to tetragonal γ^0 symmetry having a c/a axial ratio of 0.477. A further increase in temperature or niobium content results in an increase in the c/a ratio until cubic symmetry is achieved.

The temperature and composition dependency of the cell parameters and consecutiveness of α' , α'' , γ^0 , and γ can be explained by a shear mechanism. Figure 5 illustrates this model.

Figure 5a is the close pack plane (110) of the bcc γ structure. This drawing is also the projection of atoms in the bcc structure onto a $(110)_\gamma$ bcc plane showing the sequence of $\{112\}$ bcc planes. The $\{112\}_\gamma$ planes are perpendicular to the plane of the drawing and lie in the directions in which the circles touch, i.e., the $[111]_\gamma$ directions.

Figure 5b is the close pack plane (001) of the hcp structure indexed on the orthohexagonal cell. This drawing is used to represent the (001) plane in the α , α' , and α'' structures, and the (110) plane in the γ^0 structure.

If it is assumed that the close-pack plane in the parent γ phase becomes a close-pack plane in the γ^0 , α'' , and α' product phases during a shear transformation, then certain orientation relations become obvious. Figure 5a shows the resulting orientation relationships between

the γ , γ^0 , α'' , and α' phases. Some of the more obvious orientation relationships are:

$$[001]_\gamma \parallel [001]_{\gamma^0}; [100]_\gamma \parallel [100]_{\gamma^0}$$

$$[100]_{\alpha', \alpha''} \parallel [111]_{\gamma, \gamma^0}; [001]_{\alpha', \alpha''} \parallel [101]_{\gamma, \gamma^0}$$

These orientation relationships are also visualized in Figure 5b.

The γ^0 structure can be generated from the γ phase by shear of two sets of $(110)_\gamma$ planes in $\langle 111 \rangle_\gamma$ directions. This double shear maintains an orthogonal relationship in the $(110)_{\gamma^0}$ plane (see Figure 5b). This shear also has the effect of reducing the $\angle\gamma$ shown in Figure 5a from a value of $\sim 96^\circ$ to a value of possibly 93° in a continuous manner.

When the c/a axial ratio of γ^0 decreases to a value of 0.477 there is a symmetry change. The symmetry change can be interpreted as a preference to shear on one set of $(112)_\gamma$ planes in one $\langle 111 \rangle_\gamma$ direction. This has the effect of destroying the orthogonal relationship in the $(110)_{\gamma^0}$ plane and monoclinic symmetry becomes apparent. This shear is illustrated in Figure 5b and corresponds to a continuous change in $\angle\gamma$ of 93° to 90° (Figure 5a). When the shear has progressed to a value of $\angle\gamma = 90.0$, then the structure is orthorhombic α' .

Thus by considering a double shear of $(110)_\gamma$ planes in $\langle 111 \rangle_\gamma$ directions followed by a single $(112)_\gamma$ plane shearing in $\langle 111 \rangle_\gamma$ directions plus minor adjustments (not discussed); then, it is possible to generate the varying cell parameters and symmetries, and to show the consecutiveness of the α' , α'' , γ^0 , γ phases.

SUMMARY

Three distinct transition phases: α'_t (orthorhombic), α''_t (monoclinic), and γ^0_t (tetragonal) occur in uranium-niobium alloys as intermediates in the direct $\gamma \rightarrow \alpha$ transformation. The transition phases form via a diffusionless mechanism and the sequence of generation is $\gamma \rightarrow \gamma^0_t \rightarrow \alpha''_t \rightarrow \alpha'_t$. When the transition phases occur in an alloy, they are present as a rather-stable single phase and do not coexist as noncoherent phases. The temperature-induced transformations from one transition form to another are athermal, reversible, continuous, and diffusionless, and the transformation temperatures decrease as a direct function of solute composition. For a given crystallographic symmetry, there is a significant

athermal, reversible and continuous temperature-induced change in unit cell parameters. The cell parameter changes are interpreted as a continuous shear transformation operating in the composition interval of 5 to 21 at. % niobium and in the temperature range of -200° to $+400^{\circ}\text{C}$. This continuous shear transformation results in dimensional unit cell changes that are highly anisotropic. At certain combinations of temperature and composition, the continuous shear results in the unit cell changes that manifest themselves as symmetry changes. This interpretation has resulted in an approximated constitutional diagram for the transition phases and in a model for the consecutiveness of the transformations.

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ILLUSTRATIONS

Figure 1. Unit Cell Parameters of the α' , α'' , and γ^0 Transition Phases and the Metastable γ Phase as a Function of Composition at Room Temperature.

Figure 2. Schematic Representation of Differences in Stable Phases and Transition Phases: (a) Unit Cells and X-Ray Diffraction Patterns of Orthorhombic a and a' , and Monoclinic a'' ; and (b) Unit Cell and X-Ray Diffraction Patterns of Tetragonal γ^0 and Metastable γ .

Figure 3. Some Unit Cell Parameter of Transition Phases as a Function of Temperature: (a) the c/a Axial Ratio of the Tetragonal γ^0 Structure in the U-15 and U-19 at. % Nb Alloys; and (b) the Monoclinic Cell Angle of the a'' Structure in the U-11 and U-15 at. % Nb Alloys.

Figure 4. Approximated transition phase diagram for uranium-base niobium alloys. The dotted lines represent equilibrium phase boundaries. The solid lines represent non-equilibrium phase boundaries.

Figure 5. Modelistic Explanation for the Consecutiveness and Thermal Reversibility of the $\gamma \leftrightarrow \gamma^0 \leftrightarrow \alpha'' \leftrightarrow \alpha'$ Transformation: (a) the Close-Pack Plane of the bcc Structure; and (b) the Close-Pack Plane of the hcp Structure.